### WOODHEAD PUBLISHING SERIES IN CIVIL AND STRUCTURAL ENGINEERING



# ECO-EFFICIENT PAVEMENT CONSTRUCTION MATERIALS



Edited by FERNANDO PACHECO-TORGAL, SERJI AMIRKHANIAN, HAO WANG, ERIK SCHLANGEN

### **Eco-efficient Pavement Construction Materials**

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Woodhead Publishing Series in Civil and Structural Engineering

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Edited by

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# Introduction to eco-efficient pavement materials

1

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### 1.1 The state of the Planet

Almost 50 years ago several scientists used a system dynamics computer model to simulate the interactions of population, food production, industrial production, pollution, and consumption of nonrenewable natural resources, having predicted that during the 21st century the Earth's capacity would be exhausted, resulting in the collapse of human civilization [1]. Two decades later, in an update of this study, the same authors showed that some limits had already been crossed [2]. Meadows et al. [3] conducted a 30-year update of the original study and concluded that period was nothing than a waste of time and that Humanity has done very little to avoid the collapse of the Planet's environment. Turner [4] also studied Meadows' projections with 30 years of real events and concluded that the global system is on an unsustainable trajectory unless there is a substantial and rapid reduction in consumptive behavior. One year later Rockström et al. [5] suggested an innovative approach for global sustainability defining nine interdependent planetary boundaries. They also stated that humanity has already transgressed three planetary boundaries for changes to the global nitrogen cycle, rate of biodiversity loss, and above all climate change. More details on the suggested planetary boundaries framework can be found in Ref. [6]. The UN Intergovernmental Panel on Climate Change released a special report warning that "Limiting global warning to 1.5°C would require rapid, far-reaching, and unprecedented changes in all aspects of society" [7]. The target of  $1.5^{\circ}$ C is being the threshold beyond which climate change would imperil species survival. Randers et al. [8] stated that the world will not reach all Sustainable Development Goals by 2030, nor by 2050, and that the global safety margin will continue to decline. And in truth, Distelkamp and Meyer [9] mentioned that although the global average  $CO_2$  intensity is projected to decline against 2015, it is expected that global CO<sub>2</sub> emissions will further increase by 44% up to 53 Gt in 2050. In order to keep economy running, several institutions such as UNEP, World Bank or the European Commission thus claim for green economy and green growth that is expected to do more with less while improving human wellbeing and social equity but some believe that that is not compatible with the ecologic limits of the Planet [10]. Some authors [11] stated that using a low-tech approach can not only contribute to an increase in resilience but can also create the

cultural conditions needed for politics and macroeconomics of degrowth to emerge. On the other hand, Holford [12] reminded us not only that technology has control over humans but also that it is driven by the neoliberal socioeconomic quest for profit maximization and economic growth. And others even claim that only a severe shut down of the main carbon polluters could have meaningful results still they seem to forget that such action would have a major impact on the increase of poverty. One of the solutions recommended by several authors including the 2018 Nobel Laureate of Economy is the carbon tax. Nordhaus [13] suggested carbon tax as an important way for households, firms, and governments to reduce emissions cost-effectively. He also claimed that carbon prices will strengthen incentives for research and development of technologies that will lower the cost of reducing emissions. Others, however, claim that the wellbeing of the rich must decline rapidly as the only solution to ensure that the environmental burden associated with the rise of the wellbeing of the poor does not override the sustainable carrying capacity of planet Earth [14]. Be there as it may and while no wonder solutions are found, which can green industries without shutting it down, incremental improvements are the only short-term solution for the problem. This was the rationale that led to the concept of eco-efficiency first coined in the book "Changing course" [15] in the context of the 1992 Earth Summit process. This concept includes "the development of products ... while progressively reducing their environmental impact and consumption of raw materials throughout their life cycle, to a level compatible with the capacity of the planet."

### 1.2 Scientific production on civil engineering and pavements

Civil engineering is the scientific area that investigates and forms professions to deal with the challenges posed by the built environment that is still associated with the world's highest consumption of raw materials, as well as very high energy consumption. Of course the fact that recent surveys show that students interested in civil engineering do not believe in anthropogenic climate change [16,17] is a problem that also requires urgent attention because it is impossible to enforce the ASCE Code of Ethics that states, "Engineers shall hold paramount the safety, health, and welfare of the public and shall strive to comply with the principles of sustainable development" with those that are skeptical on such principles. A shortcoming that is especially serious because climate change raises many questions with ethical dimensions rooted in the human condition [18,19]. In this context, perhaps the suggestion of [20] for civil engineers to play a role in helping developing countries can help overcome some of that skepticism. Concerning the subfield of pavements although being one of the major civil infrastructures represents a small subset in the larger field of civil engineering. It is worth noting that according to [21], the total mileage of roads has reached 70 million kilometers. Still, other authors [22] are more modest mentioning that asphalt roads span a distance of more than three times around the sun (around 13 million kilometers). But that value can hardly be an accurate one because the United States alone has 6.5 million kilometers of roads [23]. This author also mentioned that asphalt pavements cover an area, in the order of magnitude, of Cuba or Iceland (100,000 km<sup>2</sup>). A search on the Scopus database for all publications concerned with civil engineering shows that in the beginning of the 80s, when publications about roads and pavements start to appear, other sub-fields of civil engineering already represented more than 700 publications per year (Fig. 1.1). Only in 1988 the number of pavement-related publications crossed 100 per year, still representing 0.8% of the civil engineering output. And only by 2004 research investigations in the field of roads and pavements were able to reach an accumulated number of more than 1000 publications that push that area to 1.6% of the civil engineering universe.

Currently, the accumulated number of Scopus publications concerned with civil engineering exceeds half a million and the subfield of roads and pavements still represents less than 2%. Fig. 1.2 shows that in the subfield of publications concerning roads and pavements, the first publications concerned with sustainability appeared only in 2003. And only 7 years later the accumulated number of those publications reached 100. In 2018 the accumulated number of publications concerned with environmental aspects of roads and pavements has not yet reached 8% of the publications of that area. As shown in Fig. 1.3, the sustainability-related publications of all areas of civil engineering started to appear only by 1985 but around 2010 they were able to surpass all pavements and roads-related publications and since then these have increased in an exponential mode. Meaning that many of the advances carried out in the field of civil engineering were directly for certain



**Figure 1.1** Evolution of the accumulated total number of publications (articles/reviews/ chapters, books, and referenced conference papers) in Scopus affiliated with Civil Engineering Departments (gray line) and the keywords "Roads" or "Pavements" searched in the section title (black line).



**Figure 1.2** Evolution of the accumulated total number of publications (articles/reviews/ chapters, books, and referenced conference papers) in Scopus affiliated with Civil Engineering Departments having the keywords "Roads" or "Pavements" in the title (gray line) and the keywords "Sustainability," "Sustainable," "Eco-efficiency," and "Eco-efficient" searched in the section title, abstract and keywords (black dashed line).



**Figure 1.3** Evolution of the accumulated total number of publications (articles/reviews/ chapters, books, and referenced conference papers) in Scopus affiliated with Civil Engineering Departments having the keywords "Sustainability," "Sustainable," "Eco-efficiency," and "Eco-efficient" searched in the section title, abstract, and keywords (dotted line); the keywords "Roads" or "Pavements" in the title (gray line); and the keywords "Sustainability," "Sustainability," "Sustainable," "Eco-efficiency," and "Eco-efficiency," and "Eco-efficient" searched in the section title, abstract, and keywords (black dashed line).

subfields alone [24-27]. Only in 1988 has the number of pavement related publication cross one hundred per year still representing 0.8% of the civil engineering output. And only by 2004 were the researcher's investigation in the field of roads and pavements able to reach an accumulated number of more than 1000 publications

that push that area to 1.6% of the Civil Engineering universe. This fact by itself constitutes a strong justification for this book. Concerning the sustainability of pavements, several issues merit an especial concern.

A first and obvious one is related to the Circular Economy where the value of products, materials, and resources is maintained in the economy for as long as possible, and the generation of waste minimized [28,29]. And that is why several chapters in the first part of this book are concerned with waste recycling and also life-cycle assessment (LCA). The other part concerns the influence of climate change on the durability of pavements. This is because some studies reported that thermal cracking in the asphalt pavement will decrease over the next 50 years, while rutting will occur earlier than expected [30]. And this has a direct influence on traffic accidents. Tsubota et al. [31] studied age and pavement types and their ages on the accident risk on urban expressway routes. The results showed that the age of road pavement has a positive effect on the accident risk and that the effect becomes remarkable under the rain condition on curve and tight-curve sections. That is why is worrying that in a rich country such as United States, the share of roads in poor condition nationwide increased from 14% to 20% between 2009 and 2017 [32]. It is also predicted that climate change will be associated with increased temperatures. Data show in MCCarthy [32] that the summers of 2016 and 2017 had some of the highest average surface temperatures ever recorded. It is worth mentioning that in India the intensity of heat wave for cities uses increases from 40°C in reference time frame to 45°C in short-term projection to 49°C in far future [33]. And that helps to explain why some roads have had cases of melting bitumen [34]. The temperature increase will be further aggravated due to urban heat island (UHI) (higher temperature of metropolitan areas than that of their surrounding countryside). UHI is triggered by absorption radiation due to artificial urban materials, transpiration from buildings and infrastructure, release of anthropogenic heat from inhabitants and appliances, and airflow blocking effect of buildings [35,36]. Concerning the Urban Heat Island Intensity (the difference in temperature between an urban site and a rural site), some projections show that in the northwest of the United Kingdom, summer mean temperatures could increase by 5°C (50% probability, 7°C top of the range) by the 2080s [37]. Strangely Plati [38] cited [39] on the fact that permeable pavements could help to mitigate UHI effects. However, the work of those authors has nothing on the influence of permeable pavements on UHI. In fact pervious pavements help to reduce peak stormwater runoff, thereby preventing incidents of combined sewer overflows. It is important to remember that in 2017, more than 1000 people died and 45 million people lost homes, livelihoods, and services when severe floods hit Southeast Asian cities, including Dhaka in Bangladesh and Mumbai in India [40]. Pervious pavements are therefore an important feature for city resilience [41,42] and constitute another eco-efficient approach for pavement materials. Also very important is pavements with self-healing capacity that will allow for higher durability, reduced pavement maintenance, reduced resource consumption, reduced carbon emissions, and also reduced traffic congestion problems. In the United States alone, costs related to wasted fuel and time loss due to traffic congestion were estimated between 50 and 100 billion dollars [43,44]. That

importance explains why is issue is addressed by four chapters in this book. And also why on June 13, 2019, it was considered one of the 100 Radical Innovation Breakthroughs (RIBs) for the future by a report of the European Commission [45]. Jiang et al. [21] recently presented a review on the performance, applications, and challenges of six environmentally friendly functional pavement materials, namely the permeable asphalt concrete, noise-reducing pavement materials, low heat-absorbing pavement materials, exhaust gas-decomposing pavement materials, deicing pavement materials, and energy harvesting pavement materials. For energy harvesting, they have included piezoelectric pavements, photovoltaic powergenerating pavements and also thermoelectricity pavements. Wang et al. [46] reviewed energy harvesting technologies and also included two mores technologies, solar collectors and geothermal (heat pipes with geothermal water). However, the cutting edge case of self-charging roads in which roads deliver energy to electric vehicles also needs to be highlighted as an important multifunctional asset of ecoefficient pavements; this case will be addressed in the last part of this book that also deals with the important issue of energy harvesting. It is worth mentioning that energy harvesting is one of the few RIBs with an impact on more than 15 global value networks [45]. All this means that in the 21st century, much will be expected from pavement materials as high added value multifunctional materials, not only to have enough durability being capable of high performance despite an increase in temperature stress, to help to tackle stormwater events, to help to reduce UHI effects but also to be able to generate energy and deliver it to electric vehicles.

### 1.3 Outline of the book

This book provides an updated state-of-the-art review on eco-efficient pavement materials. Part 1 of the book concerns pavements with recycled waste (Chapters 2-6).

Chapter 2, Utilization of scrap plastics in asphalt binders, addresses a case study on the use of three different postindustrial polyethylenes as partial replacement of bitumen in asphalt-based pavements.

Chapter 3, Use of waste engine oil in materials containing asphaltic components, discusses the use of waste engine oil in pavement materials containing asphaltic components. The refinement of waste engine oil, its properties, and its use in bituminous binders are covered.

Chapter 4, Microstructure and performance characteristics of cold recycled asphalt mixtures, reviews the microstructure performance of cold recycled asphalt mixtures.

Chapter 5, Life-cycle assessment of asphalt pavement recycling, reviews the LCA studies focusing on asphalt pavement recycling, including hot, warm, and cold recycling methods, as well as in-plant and in-place recycling technologies.

Part 2 deals with pavements for climate change mitigation (Chapters 6-8).

Chapter 6, Cool pavements, presents the UHI and the urban heating phenomena and provides an overview of cool pavement technologies, detailing areas that require further scientific investigation.

Chapter 7, Reflective coatings for high albedo pavement, reviews the LCA studies focusing on asphalt pavement recycling, including hot, warm, and cold recycling methods, as well as in-plant and in-place recycling technologies. The aim is to provide constructive recommendations and pointing out the potential research gaps for future studies.

Chapter 8, Influence of aging on the performance of cool coatings, summarizes the key parameters contributing to the aging of cool coatings, along with the main methods for assessing their weathering and aging; in the second part, the effect of the aging on the thermal performance of cool coatings, with regard to both the urban microclimate and the buildings' energy performance, is discussed.

Part 3 (Chapters 9-12) deals with self-healing pavements.

Chapter 9, Self-healing property and road performance of asphalt binder and asphalt mixture containing urea formaldehyde microcapsule, discusses the self-healing ability and rheological property of asphalt binder containing urea formaldehyde resin self-healing microcapsule and other microcapsules.

Chapter 10, Self-healing biomimetic microvascular containing oily rejuvenator for prolonging life of bitumen, focuses on the case of hollow fibers containing oily rejuvenator fabricated by a one-step wet-spinning method using polyvinylidene fluoride resin.

Chapter 11, Self-healing pavements using microcapsules containing rejuvenator: from idea to real application, is concerned with the fabrication method of microcapsules containing oily rejuvenators, including controlling the mean size, shell thickness, mechanical property, and thermal stability.

Chapter 12, Novel magnetically induced healing in road pavements, addresses novel approaches to generate engineered healing capabilities through induction heating of ferrous material-asphalt composites. These include ferromagnetic fillers, ferrous fibers, mixed-metal alloy fibers, and recycled fibers from shredded vehicle tires, among others.

Finally, Part 4 concerns pavements with energy harvesting potential and vehicle power charging ability (Chapter 13-15).

Chapter 13, Thermoelectric technologies for harvesting energy from pavements, introduces the thermoelectric technologies for harvesting energy from pavements. It starts by presenting the basic principle of thermoelectric effect known as "Seebeck effect." It then explains the temperature characteristics of road surface, ambient air, and subgrade. Based on this, pavement-ambient thermoelectric system and pavement-subgrade thermoelectric system are introduced. At the end of the chapter, the problems and development of pavement thermoelectric technologies are analyzed and discussed.

Chapter 14, Piezoelectric energy harvesting from pavement, reviews piezoelectric energy harvesting from pavement. It includes piezoelectric materials, principle of energy harvesting, and piezoelectric transducer designs and types. It reviews previous studies on piezoelectric energy harvesting in pavement, as well as the use and storage of harvested energy and challenges of piezoelectric energy harvesting in pavement.

Chapter 15, Inductive power transfer technology for road transport electrification, closes Part 4 reviewing electrified road system feasibility studies conducted with the aim of bridging these emergent gaps, which serve as an opening-up work in this new research area.

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### Part 1

### Pavements with recycled waste

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### Utilization of scrap plastics in asphalt binders

2

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### 2.1 Introduction

In 1907 Bakelite, the first synthetic plastic was produced. The rapid growth in global plastic production, for many reasons, initiated in 1950s. The annual production of plastics increased by 200 folds over the next 65 years to over 400 million tons annually (Fig. 2.1). In general, plastic pollution has had a negative impact on the oceans and wildlife as higher income countries generate more plastic per person. However, most of the global ocean pollution is from the middle- and low-income countries because they do not have, in general, a very effective waste management program (Fig. 2.2). It is estimated that approximately 80% of ocean plastics come from land-based sources and the other 20% from marine (e.g., fishing nets, ropes and lines). However, the use of plastics in many parts of the world will not decrease because plastic is a unique material with many benefits including, but not limited to: (1) it is relatively inexpensive, (2) versatile, (3) lightweight, and (4) recyclable. Therefore, a complete ban or finding a substitute might be a very complex issue at this time. It is estimated that, by 2018, over 9 billion tons of plastic have been produced in the world. That is more than 1.2 tons of plastic for every person alive. Recycling, in many formats, of plastic was not very organized before 1980; however, it is estimated that by 2050, incineration rates would increase to 50%, recycling to 44% and discarded plastic waste to only 6% [1,2] (Fig. 2.3).

### 2.2 Background

In the U.S., Environmental Protection Agency (EPA) estimates that Americans produce over 260 million tons of waste and recycle or compost more than 89 million tons at a recycling rate of approximately 35%. Some of the recyclable materials (e.g., plastics, rubber, etc.) have been exported to many countries around the world (e.g., India, China, etc.). China has been taking about 40% of United States' paper, plastics, and other recyclables. However, recently, the Chinese government has stopped this practice by implementing the National Sword policy at the beginning of 2018 to protect their environment and develop their own domestic recycling capacity by restricting imports of waste. For example, recycled plastics from the



Figure 2.1 Plastic waste generation worldwide [1,2].



Figure 2.2 Share of plastic waste that is mismanaged [1,2].

United States to China dropped by 92% over the first 5 months of the year when the restrictions took effect. All types of exported scrap, from plastics and paper to aluminum, copper, and stainless steel, fell 36%. The Chinese market was greater than the next 15 markets combined, thus leaving the U.S. market, among others around the world, without any backup. Now, some of these materials that would have been recycled are now going into landfills instead. In some cases, municipalities have stopped collecting items that can be recycled and others have been storing them.



Figure 2.3 Plastic exports to China before the ban [3].

According to the American Chemistry Council, plastics generation is approximately 35 million tons [13% of municipal solid waste (MSW)] and more than 80% of Americans have access to different plastic recycling programs. It is estimated that it takes several hundred years to decompose plastic items in landfills. According to the EPA, plastic recycling results in significant energy savings compared with the production of new plastics using virgin material. It is estimated that 1000–2000 gallons of gasoline can be saved by recycling just 1 ton of plastics.

Nowadays, environmental sustainability has become the main issue in many projects around the USA and the world. Particularly, the problem of where and how to dispose a large amount of the daily waste is one of the concerns of many municipalities all over the world [4]. Waste plastic is a material composed of one or more types of high-molecular organic polymers, which is solid in general state and can flow under specific state [5]. There are many types of waste plastics, including polyethylene (PE), polypropylene, polystyrene, and so on. Meanwhile, the disposal of waste plastic is also an issue of great concern worldwide due to its considerable quantity and growth. In order to handle this problem, many recycling techniques and research studies about the incorporation of waste plastic into asphalt binders and mixtures have been conducted [6-20]. And among all types of waste plastic used in paving applications, PE has a prominent utilization.

Hinislioglua et al. [21] studied the feasibility of modified binders using various waste plastics containing high-density polyethylene (HDPE) in different content and used them in an asphalt mixture. Binders used in hot mix asphalt were made by mixing the HDPE in 4%, 6%, and 8% (by weight of optimum asphalt content) and base binder AC-20 at 145°C, 155°C, or 165°C and mixed for 5, 15, or 30 min. They concluded that the asphalt mixture with 4% HDPE, which was mixed at 165°C for 30 min, showed the highest stability and the smallest flow value. Moreover, the mixture was also greatly resistant to rutting. Therefore,

they concluded that these types of mixtures could be utilized to make an asphalt pavement that has more resistance to permanent deformation while solving a problem with the waste plastic disposal.

Attaelmanan et al. [22] investigated the possibility of using PE as a modifier for asphalt. They modified the 80/100 penetration grade asphalt by adding different contents of PEs (by weight of asphalt). The standard asphalt binder tests indicated that the softening point increased and the penetration and temperature susceptibility decreased as the PE content increased. The results also indicated that the performances of PE-modified asphalt mixtures were better than that of traditional mixtures, with greater stability, tensile strength ratios and resilient modulus values. Hence, they reported that flexible pavements with higher performance and durability and lower costs than conventional pavements could be obtained with an additional 5% PE.

Fuentes-Audén et al. [23] carried out a study about the impacts of polymer content on the recycled PE-modified asphalt. The base binder was a 150/200 penetration grade asphalt. To analyze the evolution mechanism of optical microscopy, modulated differential scanning calorimetry measurements, steady and oscillatory shear tests, and dynamic mechanical thermal analysis, respectively, were utilized. The results indicated that the addition of PE to asphalt produced a significant improvement to some of the mechanical properties, such as higher resistance to permanent deformation, thermal cracking, and fatigue cracking. They further concluded that the modified binder of low PE content (less than 5%) could be possibly applied to highway pavements.

Many researchers have indicated that the utilization of PE in asphalt reduces the volume of waste, conserve both material and energy and provides a comparatively simple way to make a substantial reduction in the overall volume of solid waste. On the other hand, PE could enhance some rheological characteristics of asphalt binders and better-performing field mixtures.

### 2.3 Materials and experimental design

Two base binders of different sources (PG 64-22 binder) were used to produce PEmodified binders, referred to as binders A and B, at a blending temperature of  $350^{\circ}F$  (177°C) and mixing for 2 h (700 rpm). Three types of PEs were utilized, and the designed three PE contents included 2%, 4%, and 6% by weight of base binder. The base binders were also blended for 2 h at  $350^{\circ}F$  to avoid the temperature impact. In addition, two Styrene-Butadiene-Styrene (SBS)-modified binders (PG 76-22) and two rubberized asphalt binders were used, for comparison purposes, in this study. The experimental design for this research study is shown in Fig. 2.4. For each test, at least two replicates were prepared and tested.

In this study, three aging states of all binders were investigated, including virgin state, rolling thin film oven (RTFO) aging state (short-term aging) and pressured aging oven (PAV) state (long-term aging). In addition, the following tests were performed according to American Association of State Highway and Transportation Officials (AASHTO) or American Society for Testing and Materials (ASTM) specifications.



Figure 2.4 Flowchart of the experimental design.

### 2.4 Brookfield rotational viscosity test

Brookfield rotational viscometer was used to test the viscosity of the virgin binders at three different temperatures (e.g.,  $135^{\circ}$ C,  $150^{\circ}$ C, and  $165^{\circ}$ C) in accordance with AASHTO T316. A number 27 spindle and a specimen weight of 8 - 11 g were used for this test.

### 2.5 Performance grade determination

Dynamic shear rheometer (DSR) was used to measure the intermediate and hightemperature rheological properties of binders based on AASHTO T315. The complex shear modulus ( $G^*$ ) and phase angle ( $\delta$ ) values were measured at the given temperature. According to AASHTO M320, a bending beam rheometer (BBR) was used to test the performance grade (PG) low-temperature grade by using the PAVaged binders.

### 2.6 Multiple stress creep recovery test

The multiple stress creep recovery (MSCR) test was conducted by the repeated loading for the duration of 1 s followed by 9 s of recovery period using the DSR system in accordance with ASTM D7405-15. All RTFO-aged binders were tested at 76°C and a 15-min temperature equilibrium period was used before the initiation of the test procedure. Two stress levels of 0.1 and 3.2 kPa were applied and 10 cycles were conducted for each stress level. The MSCR test uses two parameters, the percent recovery (*R*) and the nonrecoverable creep compliance ( $J_{nr}$ ), to characterize the viscoelasticity properties of an asphalt binder. Two parameters are calculated as the following two equations:

$$R = \text{SUM}\left(\frac{\frac{(\varepsilon_1 - \varepsilon_{10})}{\varepsilon_1}}{10}\right)$$
(2.1)

$$J_{nr} = \text{SUM}\left(\frac{\varepsilon_{10}}{\tau}\right) \tag{2.2}$$

where  $\varepsilon_1$  is the strain at the first second for each cycle,  $\varepsilon_{10}$  is the strain at the tenth second for each cycle, and  $\tau$  is the applied shear stress level.

### 2.7 Linear amplitude sweep test

According to AASHTO TP 101-12, the linear amplitude sweep (LAS) test was used to evaluate the fatigue resistance of binders by applying cyclic load with increasing amplitude. And it was conducted using DSR with a 2 mm working gap and an 8 mm diameter plate at 25 °C. Meanwhile, each binder sample was further aged using RTFO and PAV to simulate the short- and long-term aging procedures of asphalt pavements.

Based on the viscoelastic continuum damage theory, the test results, that is, the fatigue life  $(N_f)$  of binders, can be calculated by Eq. (2.3) below. The failure of binders is defined as 35% decrease in initial modulus.

$$N_f = A_{35} \left(\gamma_{\rm max}\right)^{-B} \tag{2.3}$$

where  $N_f$  is the failure life,  $\gamma_{\text{max}}$  is the maximum shear strain for the given pavement structure, and finally,  $A_{35}$  and B are the parameters that depend on the material characteristics.

### 2.8 Frequency sweep and amplitude sweep tests

The amplitude sweep and frequency sweep tests were carried out at  $64^{\circ}C$  for each base binder and at  $76^{\circ}C$  for modified binders. Amplitude sweep tests were measured at varying shear stresses and strains. For the frequency sweep tests, frequency ranged from 0.1 to 100 Hz was selected to run at 0.1 kPa.

### 2.9 Results and discussions

#### 2.9.1 Virgin binders

### 2.9.1.1 Rotational viscosity

The rotational viscosity is used to determine the flow characteristics of an asphalt binder and to make sure that it can be pumped and handled at the asphalt plant. In this paper, the viscosity values of each binder were obtained at 135°C, 150°C, and 165°C, and, as expected, the viscosity of all asphalt binders decreased as the test temperature increased. The viscosity values of various binders from two sources at 135°C are shown in Fig. 2.5.

Fig. 2.5 indicated that PE-modified asphalts generally showed higher viscosity than base binders, crumb rubber modified (CRM) and SBS-modified binders. The viscosity value increases with an increase in the amount of PE additives regardless of the binder type. On the other hand, it seemed that there was no significant relationship between viscosity and PE type.

#### 2.9.1.2 Failure temperature

The grade determination feature of the DSR was used to determine the failure temperatures of various binders. This procedure tested the sample at a starting temperature and increased the temperature to the next PG grade if the value of  $G^*/\sin\delta$  was greater than the value required by AASHTO M320 (1.0 kPa for virgin binders). Two replicates were tested for each binder, and the results are shown in Fig. 2.6.

As shown in Fig. 2.6, PE could effectively improve the failure temperature of base binders, and PE-modified binders even showed higher failure temperatures than those CRM and SBS binders. Obviously, the failure temperature of PE-modified asphalt increased with PE content from 2% to 6%. And for binder A,



Figure 2.5 Rotational viscosity results of two binder sources: (A) Binder A and (B) Binder B.

binders modified by PE3 tended to have the highest failure temperature, followed by PE1 and PE2. While for binder B, the rank was: PE2 > PE1 > PE3.

#### $G^*/\sin\delta$

Based on the values of complex modulus ( $G^*$ ) and phase angle ( $\delta$ ), the values of rutting resistance factor ( $G^*/\sin\delta$ ) at the performance test temperatures of each virgin-state binder are shown in Fig. 2.7.

As shown in Fig. 2.7, the  $G^*/\sin\delta$  value shows similar trends to failure temperature. The binders with higher PE content tended to have higher  $G^*/\sin\delta$  values. And which type of PE-modified binder performed the highest  $G^*/\sin\delta$  value depended on the base binder source and test temperature.



Figure 2.6 Failure temperature of virgin-state binders: (A) Binder A and (B) Binder B.

### 2.9.1.3 Frequency sweep

The frequency sweep tests were performed under stress proportional to frequency, and the used frequencies were from 0.1 to 100 Hz in this research study. The frequency sweep tests at various frequencies could identify the linear viscoelastic response of the binders. The test results are shown in Fig. 2.8.

As shown in Fig. 2.8, it could be found that the increased frequency generally resulted in an increase of complex modulus and a reduction in phase angle for all asphalt binders regardless of the binder source. Moreover, it could be observed that with the increased dosage of PE, the complex modulus increased regardless of binder type, while the phase angle values decreased in most frequencies. Therefore, the frequency sweep test indicated that the viscoelastic properties of various binders were based on the PE content and type.


**Figure 2.7**  $G^*/\sin\delta$  of virgin binders. (A) Binder A with varying PE contents (PE type: PE2), (B) Binder A with varying PE types (PE content: 4%), (C) Binder B with varying PE contents (PE type: PE2), and (D) Binder B with varying PE types (PE content: 4%)



**Figure 2.8** Frequency sweep results of virgin-state binders. (A) Complex modulus versus frequency (Binder A), (B) phase angle versus frequency (Binder A), (C) complex modulus versus frequency (Binder B), and (D) phase angle versus frequency (Binder B).

#### 2.9.1.4 Amplitude sweep

In this study, the amplitude sweeps were performed to determine the complex modulus and phase angle values in terms of the shear strain responses. The results of virgin-state binders are presented in Fig. 2.9.

As shown in Fig. 2.9, the increased shear strain significantly reduced the complex modulus and increased the phase angle of binders with high PE content (4% and 6%), regardless of the binder source. On the other hand, shear strain did not, as expected, influence these binders with PE content of 2%. In addition, with the increased amount of PE, the complex modulus increased and the phase angle decreased. Thus, additional PE not only reinforced the viscosity of binders but also enhanced the elasticity.

#### 2.9.1.5 Multiple stress creep recovery test

The MSCR tests were conducted at 76°C and the results are shown in Figs. 2.10 and 2.11. The values of nonrecoverable creep compliances  $(J_{nr})$  and percentage recoverable strains (*R*) under 0.1 kPa of various binders are presented in Fig. 2.10. In addition, the rebound curves of shear strain versus test time are shown in Fig. 2.11.

As shown in Fig. 2.10, the elastic performance of the base binder was improved by the PE modifier, as the PE-modified binders showed higher R and lower  $J_{nr}$ values than other references. In other words, these PE-modified binders had better recovery performances at high temperatures, as shown in Fig. 2.10. And an increased PE content resulted in a reduction in  $J_{nr}$  and an increase in R. With regard to PE types, for binder A, binders modified by PE3 showed the best recovery performances, followed by PE1 and PE2. For binder B, the best modifier was PE1, and then PE2 and PE3.

## 2.10 PAV-aged binders

#### 2.10.1 Low-temperature properties

This study used BBR to test asphalt beam's deformation as a function of loading time; thus, stiffness and *m*-value could be calculated at different temperatures  $(-6^{\circ}C, -12^{\circ}C, \text{ and } -18^{\circ}C)$ , as shown in Fig. 2.12. In order to prevent thermal cracking, the creep stiffness has a maximum limit of 300 MPa, and *m*-value is limited to a minimum value of 0.3 at 60 s of loading time according to Superpave specification. And the PG low-temperature grade was determined by these two criteria. The critical low temperatures of various binders are shown in Fig. 2.12.

- 1. Stiffness and *m*-value
- 2. Critical low temperatures



**Figure 2.9** Frequency sweep results of virgin-state binders. (A) Complex modulus versus shear strain (Binder A), (B) phase angle versus shear strain (Binder A), (C) complex modulus versus shear strain (Binder B), and (D) phase angle versus shear strain (Binder B).



**Figure 2.10** MSCR test results. (A) Percentage recoverable strain (*R*) at 0.1 kPa (Binder A), (B) nonrecoverable creep compliance ( $J_{nr}$ ) at 0.1 kPa (Binder A), (C) percentage recoverable strain (*R*) at 0.1 kPa (Binder B), and (D) nonrecoverable creep compliance ( $J_{nr}$ ) at 0.1 kPa (Binder B).



Figure 2.11 Shear strain versus test time: (A) Binder A and (B) Binder B.

Fig. 2.13 shows that PE-modified binders produced higher critical low temperature than base binders, CRM and SBS binders, which means that PE had a negative effect on the low-temperature property of binders. And when the PE content increased, the absolute value of critical low temperature decreased. In other words, binders with higher PE content tended to have higher stiffness and lower *m*-value, as shown in Fig. 2.12. With regard to PE type, PE3 was the worst produced modifier, followed by PE2 and PE1. The low-temperature grades of various binders are presented in Table 2.1.



Figure 2.12 Stiffness and *m*-value. (A) Binder A with varying PE contents (PE type: PE2), (B) Binder A with varying PE types (PE content: 4%), (C) Binder B with varying PE contents (PE type: PE2), and (D) Binder B with varying PE types (PE content: 4%).



Figure 2.13 Critical low temperature: (A) Binder A and (B) Binder B.

## 2.11 Conclusions

- 1. Virgin binders
  - **a.** With regard to high-temperature properties, PE modifier could effectively improve the rotational viscosity, failure temperature and the  $G^*/\sin\delta$  value of base binders. And a higher PE content had a greater effect on the high-temperature properties. In addition, PE type had the greatest effect depended on base binder source.
  - **b.** The frequency test indicated that the increased frequency generally resulted in an increase in complex modulus and a reduction in phase angle. With the increased dosage of PE, the complex modulus increased, while the phase angle values decreased in most frequencies.
  - **c.** The amplitude test showed that the increased shear strain significantly reduced complex modulus and increased phase angle of binders with high PE content (4% and 6%)

Binder type	Low-temperature grade (°C)	Binder type	Low-temperature grade (°C)
A A-CRM-15% A-SBS A-PE1-2% A-PE1-4% A-PE1-6% A-PE2-2% A-PE2-4% A-PE2-6% A-PE3-2%	$ \begin{array}{r} -16 \\ -16 \\ -22 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ \end{array} $	B B-CRM-15% B-SBS B-PE1-2% B-PE1-4% B-PE1-6% B-PE2-2% B-PE2-4% B-PE2-6% B-PE2-6% B-PE3-2%	$ \begin{array}{r} -16 \\ -16 \\ -16 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ -10 \\ \end{array} $
A-PE3-4% A-PE3-6%	-10 - 10 - 10	B-PE3-4% B-PE3-6%	-10 - 10

Table 2.1 Low-temperature grade of various binders.

regardless of the binder source, and additional PE not only reinforced the viscosity of binders but also enhanced the elasticity.

- 2. RTFO-aged binders
  - **a.** The PG determination, frequency sweep and amplitude sweep tests of RTFO-aged binders showed similar results to virgin binders.
  - **b.** The MSCR test indicated the elastic performance of base binder was improved by the PE modifier, as the PE-modified binders showed higher *R* and lower  $J_{nr}$  values than other references. Moreover, an increased PE content resulted in better recovery performances at high temperatures.
- 3. PAV-aged binders
  - **a.** In terms of low-temperature properties, PE-modified binders showed higher critical low temperature than base binders, CRM and SBS binders, which meant that PE had a negative effect on the low-temperature property of an asphalt binder. Those binders with higher PE content tended to have worse thermal cracking resistance.

With respect to fatigue properties, the  $G^*\sin\delta$  factor indicated that the PE modifier had a negative effect on the fatigue resistance performance, while the LAS test found that PE-modified binders showed longer fatigue life, that is, higher fatigue cracking resistance. The discrepancy might due to that the  $G^*\sin\delta$  factor did not consider the nonlinear viscoelastic behavior of the binder. Hence, the LAS results are hypothesized to be more reliable.

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# Use of waste engine oil in materials containing asphaltic components

3

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## 3.1 Introduction

It is a well-known fact that the use of combustion engine-based automobiles is increasing across the globe, especially over the last five decades. Petroleum-based oil is used for the smooth running of the engine. This engine oil is commonly referred to as lubricating oil, crankcase oil, cylinder oil, and motor oil [1,2]. During its service life within the engine, this engine oil undergoes (1) significant changes in its physical properties and (2) marginal changes in its chemical composition. These changes can be attributed to temperature variation within the engine, chemical reactions, and molecular structure changes within engine oil. After its beneficial use, this engine oil is replaced with the fresh one. The duration of its beneficial use depends on the driven distance by vehicle, type of oil, type of vehicle (or engine), the duration for which oil is within the engine, moisture, soot, and engine wear metal particles [3–5]. The oil that is removed from the engine is commonly referred to as waste engine oil (WEO).

Factories, aviation sector, the marine sector, and automobiles are primary sources of this WEO [6]. About 3.5 billion liters of engine oil is wasted in the US every year [7]. Zhang et al. [8] reported that roughly 24 million metric tons of WEO are generated annually in China. Approximately 12, 16, 39.9, 71, and 250 million liters of used oil is generated annually in Manitoba, Saskatchewan, British Columbia, Alberta, and Ontario, respectively [9]. A total of 1.7–3.5 million tons of waste lubricating oil is gathered annually in Europe and the USA [10,11]. In 2014, around 451.8 million liters of used lubricating oil was collected in Brazil. In 2015, 291 ktonnes of waste oil was generated in Spain. These statistics indicate that a significant quantity of WEO is being produced in different parts of the world.

The historical practice has been to dispose of WEO through burning or dumping. One study by Teoh et al. [12] reported that approximately 60% of WEO generated annually in Malaysia is disposed of as landfill without proper treatment. However, such an approach of ad-hoc disposal leads to environmental damages [13-18]. For example, direct disposal in landfills often pollutes the soil immediately. Further,

WEO will penetrate into the subsurface water system leading to water contamination. The layer of WEO on the surface of water blocks oxygen supply to the aquatic life [19]. Again, direct burning of WEO results in the release of pollutants to atmosphere and consequent degradation in air quality [16,19,20,21]. Hence, it is imperative to find an alternative strategy to deal with WEO.

In recent years, most of the countries/agencies have emphasized on the construction and maintenance of pavement infrastructure through sustainable practices. These include recycling of pavement materials, use of marginal materials, and use of alternative materials. Use of recycled asphalt pavement (RAP) is one of the popular approaches to increase utilization of old materials while reducing the consumption of virgin materials. However, binder within RAP typically has higher viscosity [22,23]. The higher viscosity of binder in RAP can be attributed to the aging of hydrocarbon components. This higher viscosity can often lead to workability issues during construction, performance issues such as rutting, and fatigue during its service life [24-27]. Hence, materials such as viscosity reduction agents, rejuvenators are used to compensate for the changes in properties (or constituents) occurring during aging.

Due to its common source of origin, the structure of individual components (such as hydrocarbons, aromatics, saturates, and asphaltenes) of WEO is similar to that of asphalt binder [15,19,28]. This similarity makes WEO a suitable material that can be used in conjunction with the asphalt binder. Even, partial usage of WEO along with asphalt binder can lead to significant savings, conservation of natural resources, and reduction in energy consumption. The schematic diagram showing typical life cycle of WEO is presented in Fig. 3.1.

This chapter discusses various aspects of WEO with specific reference to its usage in conjunction with asphaltic materials. This section introduces the topic with details regarding sources of WEO, its production around the world and the environmental issues created during disposal of WEO. Section 3.1.1 discusses the various processes involved in the refining of WEO. Details regarding physical properties, its chemical composition of WEO are presented in Section 3.2. Section 3.3 explains the various aspects related to possibility of incorporating WEO into binder and mixture. Challenges related to use of WEO and concluding remarks are presented in Sections 3.4 and 3.5, respectively.



Figure 3.1 Typical usage of waste engine oil in conjunction with asphaltic materials.

#### 3.1.1 Refining process of waste engine oil

The waste engine oil contains a considerable quantity of contaminants including heavy metals and other chemicals. In order to use it in conjunction with asphalt, one has to ensure that the contaminants within WEO conform to the safer limits. For example, the U.S. environmental protection agency and the Malaysian department of environment have set specifications for the permissible limits of contaminants in WEO to be used in asphalt pavements [14]. The process of modifying WEO before its reuse is often referred to as re-refining. The process of re-refining can be proudly classified into chemical, physical, and solvent extraction processes. Botas et al. [29] estimated that 1 L of fresh oil can be extracted from 1.6 L of WEO by proper treatment. Various re-refining processes that are carried out can be broadly classified into physical and chemical processes.

The basic principle of physical refining process is to allow heavy molecules to settle down while vaporizing the low molecular fractions under constant temperature and pressure without using chemicals. Distillation and thin-film evaporation are some examples of physical re-refining processes [10,29-31]. The distillation process can be atmospheric distillation or vacuum distillation. Atmospheric distillation or dehydration process removes water and light hydrocarbons. This process separates the volatile components at a temperature of about 160°C-180°C and under atmospheric pressure [32]. Further, by-product from dehydration is fed into the vacuum distillation. The vacuum distillation is performed at higher temperature of around 250°C [10] and pressure. It separates higher fractions of oil. The gasoline is separated at around 200°C and the temperature is increased up to around 400°C until all the volatile components are separated. These volatile components can be used as fuel. Thin-film evaporation removes the high molecular weight asphaltenes from the waste oil. The process involved in the various physical refining techniques is shown in Fig. 3.2. Usually, this physical process is expected to be environmental friendly. However, during the process the equipment will be contaminated and cracking of hydrocarbons (breaking down of higher molecular weight components) may occur. Also, the physical process is found to be laborious and expensive [10]. Membrane technology removes the particles from used oil. However, the process is expensive, and larger particles are clogged in the membrane [30].

Due to expensive and time-consuming physical processes, people have resorted to chemical-based separation process. In general, the chemical used in refining is selected so that they react with contaminants when compared to WEO fractions. Some of the commonly used chemical processes include acid refining [31], treatments using sulfuric acid [10] and acetic acid [10,33], acid-clay treatment [10,29-31], and hydrogenation [10,31]. Researchers observed that the treatment of WEO using acetic acid brings an economical and environmentally friendly solution when compared to treatment using sulfuric acid [10,31]. Hydrogenation removes the heteroatoms in the WEO such as sulfur, nitrogen, and some of the aged constituents. The chemical process causes pollution while refining and is found inadequate to remove large asphaltene contents [10]. A combination of vacuum distillation and hydrogenation, the Kinetics Technology International process, also found hugely priced [10,32].



Figure 3.2 Physical refining process of waste engine oil (WEO).

Solvent extraction is considered to be eco-friendlier as it extracts useful oil from WEO without expelling any toxic constituents with steam or chemicals [31]. The solvent dissolves the heavy molecular aromatics and asphaltenes while retaining the saturates. Researchers have used a mixture of toluene, butanol, methanol, ethanol, isopropanol, *n*-hexane, petroleum ether, 1-hexanol, carbon tetrachloride, and acetone (solvent extraction) to remove the physical, chemical, and mechanical impurities [10,29–31]. Adsorption is generally used with solvent extraction [31,33]. Adsorption has been tried with almond shell, walnut shell, eggshell, and acidactivated clay. The solvent extraction process starts with filtration. The oil is extracted from the mixture of clear oil and solvent in a rotatory evaporator. Activated alumina is mixed to obtain the new oil [31,33].

## 3.2 Properties of waste engine oil

Due to continuous wearing of engine parts and high-temperature oxidation, significant changes in engine oil are observed [31]. Due to these chemical reactions and physical changes, WEO appears black in color [28]. The difference in color between base engine oil and WEO is apparent from Fig. 3.3. The properties of WEO can be evaluated through physical and chemical evaluation [12,28]. In general, properties of WEO are significantly affected by its initial properties and conditions it experienced during its service life.

#### 3.2.1 Physical properties

Depending on temperature, WEO exhibits semi-solid to liquid state. Thus, a pour point is used to identify its physical state. As WEO is lighter than water, lower density and specific gravity values are observed. Due to its hydrocarbon composition, flash and fire points are used to quantify its combustible nature. Percentage loss on heating is used as an indicator of percentage of volatiles. Zhang et al. [8] reported higher and lower heating values of 40,384 and 37,402 J/g, respectively. Due to its



Figure 3.3 Base engine oil (A) and waste engine oil (B).

Property	Reported value	Reference
Pour point, °C	-5	Hamawand et al. [10]
Flashpoint, °C	120	Kamaruddin et al. [14]
	158	Hamawand et al. [10]
	193	Nurul Hidayah et al. [34]
	220	Liu et al. [16]
Fire point, °C	210	Nurul Hidayah et al. [34]
Loss on heating, °C	4.45	Nurul Hidayah et al. [34]
Specific gravity	0.85	Borhan et al. [20]
	0.8697	Hamawand et al. [10]
	0.92	Teoh et al. [12]
	0.93	Kamaruddin et al. [14]
Density at 15°C, g/cm <sup>3</sup>	0.756	Borhan et al. [20]
	0.8816	Liu et al. [16]
	0.9116	Shoukat and Yoo [35]
Viscosity at 40°C, centistokes	29.6	Borhan et al. [20]
	107.48	Shoukat and Yoo [35]
	136.6	Hamawand et al. [10]
Viscosity at 60°C, cP	33.25	Nurul Hidayah et al. [34]
Viscosity at 100°C, centistokes	4.2	Borhan et al. [20]
	12.93	Shoukat and Yoo [35]
	13.5	Hamawand et al. [10]
Viscosity index	89.11	Hamawand et al. [10]
Refractive index	1.4763	Hamawand et al. [10]

Table 3.1 Summary of physical properties.

temperature-dependent state, WEO exhibits viscoelastic behavior. As WEO exhibits fluid state above subzero temperature, viscosity is used as an indicator of its overall properties. Kamaruddin et al. [14] evaluated viscoelastic properties of WEO using complex shear modulus and phase angle measurements. They reported thermorheologically simple behavior of WEO through master curves. The numerical values of various physical properties reported by various researchers are summarized in Table 3.1.

#### 3.2.2 Chemical composition

Owing to its petroleum origin, WEO primarily consists of hydrocarbons. Among various hydrocarbons, aliphatic and aromatic compounds are predominant [36]. Hamawand et al. [10] reported that the specific gravity of WEO shows an increasing trend with an increasing percentage of aromatic content. However, the opposite trend was observed with increasing saturated aliphatic content.

Due to continuous wearing of engine components and elevated temperature, WEO contains very fine particles of heavy metals and impurities [12,14]. The percentage of impurities (on weight basis) ranged between 0.362 [16] and 0.49 [8]. Ash content in WEO was found to be less than 1%. Hamawand et al. [10] reported a carbon residue of 0.55% (on weight basis).

#### 3.2.2.1 Elemental level analysis

Due to its crude oil origin, WEO contains a significant amount of hydrogen and carbon. Researchers have found elements such as P, Ca, Fe, Mn, Cu, Zn, Pb, Mg, Cr, Sn, Cd, and Mb. Techniques such as X-ray fluorescence analysis, atomic absorption spectrometry, and inductively coupled plasma atomic emission spectroscopy have been used for this purpose [10,14,37,38]. Arsenic, cadmium, and polychlorinated biphenyl were found to be within permissible limits in WEO [39]. The elemental composition of WEO as reported by various researchers is summarized in Table 3.2.

#### 3.2.2.2 Molecular level analysis

It is a well-known fact that molecular structure influences the physical and chemical properties of the binder. With aging, the molecular structure within asphalt undergoes significant changes. The chemical constituents react with each other to form high molecular weight components. For example, maltenes transform themselves into asphaltenes [40]. Also, the polar molecular structure formed during aging is large compared to the molecular size of WEO [41]. These long chain molecules in turn offer more resistance against any movement and make binder more stiff [28]. WEO helps the binder to compensate these lost maltenes to a certain extent. It was observed that the molecular arrangement of saturates changes with addition of WEO. WEO alters the balance of molecular interactions that decides the physical properties of binder [41]. The polar molecules contribute to the viscosity of binder [41]. The elastic component of binder is appertaining to the polar molecules and for the viscous nature, the binder is characterized by the nonpolar molecules. WEO lowered the glass transition temperature [42]. Researchers have used gel permeation chromatography (GPC) to analyze the molecular structure of WEO. Saturates and naphthene aromatics in WEO share a similar molecular size fraction with asphalt. WEO contains a wide range of branched and aromatic hydrocarbons, nitrogen and sulfate compounds [12,14], and polycyclic aromatic hydrocarbons

Element	Reported value	Reference
Nitrogen, %	0.28	Zhang et al. [8]
	30.68	Liu et al. [16]
Carbon, %	12.9	Liu et al. [16]
	83.14	Zhang et al. [8]
Hydrogen, %	0	Liu et al. [16]
	13.67	Zhang et al. [8]
Sulfur, %	0	Liu et al. [16]
	1.03	Zhang et al. [8]
Oxygen, %	56.42	Liu et al. [16]
	1.88	Zhang et al. [8]

Table 3.2 Summary of elemental composition of waste engine oil.

[28]. The nuclear magnetic resonance and infrared spectrometry observed the presence of polyisobutylene (PIB) in the WEO [38,43]. The asphaltene and polar fractions of WEO are higher in molecular size when compared to asphalt [18,41]. Studies indicated that the quantity of hydrocarbons remains almost unchanged and it is the composition of the hydrocarbon changes during WEO's service life within the engine [44].

#### 3.2.2.3 Analytical methods

Various analytical methods have been used to evaluate the suitability of WEO. Some of these tests are at the component level while the rest are at the blend level. Some examples of these analytical methods are Fourier transform infrared (FTIR) spectrometry, environmental scanning electron microscopy (ESEM), GPC, and modulated differential scanning calorimetry (MDSC). FTIR spectrometry explores the functional groups and molecular weight of constituents present in the binder [10,43]. In other words, FTIR reveals the macromolecular structure of the materials [45]. FTIR analysis can provide a qualitative and quantitative measure of oil constituents. Different functional groups absorb different wavelengths in the infrared spectra while oscillating at a particular frequency. Due to oxidation, the functional groups in WEO will be mostly changed to carbonyl and sulfoxide groups and this can be detected by FTIR as a measure of aging [28]. This also indicates the conversion of low molecular weight maltenes to high molecular weight asphaltenes. Researchers have found that FTIR results can be used to calculate the total acid number and total base number. These numbers have been used to quantify the quality of oil [30]. WEO contains aromatics and polyolefins [16]. The aromatics contribute to the viscous part of the asphalt mixture. The functional groups such as alkanes, aromatic, aldehyde, CH<sub>3</sub>, carboxylic acid, and carbonyl compounds were found in the used engine oil [10]. Kaskow et al. [38] identified the PIB in the WEO, which contributes to the segregation of asphaltenes, and thus, the binder hardens and leads to low-temperature cracking. PIB is an additive used in engine oil. It adheres tightly to the aggregates and is difficult to separate by treatments. The aromatic groups showed significant changes due to oxidation. However, paraffinic and naphthalic components are inert to changes during engine operation.

## 3.3 Utilization of waste engine oil

Asphalt concrete is one of the main components in flexible pavement. While acting as binding agent, asphalt holds aggregate particles together. However, when exposed to environment, asphalt binder changes its chemical and physical properties. This process is referred to as aging. Due to aging, the maltenes will be converted to asphaltenes and the binder becomes hard and brittle. This leads to a situation where binder in RAP has higher viscosity. This necessitates the usage of viscosity reducing agents/rejuvenators to enhance properties of RAP binder. As the asphalt and WEO are from the same source, WEO has been used to replace either a



Figure 3.4 Methodology adopted while blending waste engine oil (WEO) with asphalt binder.

portion of binder or the chemical additives [34,46]. The aromatic content and low molecular weight maltenes (present in WEO) can be used to replace these components (present in RAP) that are lost during oxidation [45]. At a broader scale, chemical composition and the quantity of WEO added to binder affect the performance of binder and mixture significantly [20]. The general methodology adopted while mixing WEO with asphalt binder is described in Fig. 3.4.

### 3.3.1 Incorporation of waste engine oil into the asphaltic binder

Various researchers have studied the effect of WEO on properties of RAP binder. Due to similar molecular structure, WEO can be easily blended with asphalt binder [7,13]. Addition of WEO into asphalt binder increases the penetration value [7,9,19,20,45] and reduces the softening point [7,9,20,45] and ductility [20] of the binder. The reduction in softening point of unaged and short-term aged binder is shown in Fig. 3.5 [47]. Ragab and Abdelrahman [41] found that the WEO will atome the lost maltenes in the binder due to aging.

Rotational viscometer [15,46] and dynamic shear rheometer have been used to test the rheological performance of asphalt binder blended with WEO [9,14,16,28,34,39,48,49]. WEO acts as a cohesive agent in the binder. It will help to reduce the viscosity of aged binder [7,13,14,20,39,50]. The data from the research work by Qurashi [47] as shown in Fig. 3.6 indicate the general trend of (1) viscosity reduction with increasing WEO (at a particular aging condition and temperature), (2) increase in viscosity with aging (at a particular temperature and



Figure 3.5 Variation of softening point of the asphalt binder with waste engine oil (WEO) content.



Figure 3.6 Variation of asphalt binder viscosity with test temperature, aging, and WEO content.

WEO content), and (3) viscosity reduction with increasing temperature (at a particular aging condition and WEO). Qurashi [47] used viscosity aging index to quantify aging. The same is presented in Fig. 3.7. In general, for a particular WEO content, an increasing aging index was observed with a decrease in temperature. Further, the lowest aging index was observed at 2% WEO (when compared to other blends). This implied that the addition of 2% of WEO is sufficient to improve aging resistance of asphalt binder.

Dynamic shear rheometer has been used to conduct the multiple stress creep recovery (MSCR) test for determining the high-temperature rut resistance [18,28,36,49,51,52] and to characterize the elastic recovery and linear amplitude sweep test [18,28,36,42,52] for evaluating the intermediate temperature fatigue cracking resistance [18].



Figure 3.7 Variation in aging index of the binder with the addition of WEO.



Figure 3.8 Variation of rut parameter with the addition of WEO: (A) Variation with temperature and (B) variation with aging.

Loss of low molecular components from the binder makes it prone to low-temperature cracking and brittleness. The binder performance at low temperature was improved by the addition of waste lubricating oil [18,28,39]. The binder showed better resistance to thermal cracking [35]. The moisture damage characteristics of the WEO-modified asphalt binder showed improved resistance when compared to control [50]. Also, the addition of WEO resulted in a reduction in temperature susceptibility [15].

With straight run asphalt, the mixing of WEO doesn't show any susceptibility to cracking [20]. Some studies showed an improvement in the fatigue resistance of binder by the addition of WEO [15,16,40,42]. Apart from the initial susceptibility to aging, the rut resistance was improved after short-term aging [14]. For the same reason, resistance to plastic deformation increased with incorporation of WEO [20,34]. The variation of rut parameter with WEO content and temperature as observed by Qurashi [47] is shown in Fig. 3.8. It is clear from Fig. 3.8 (A) that

rutting parameter ( $|G^*|/\sin\delta$ ) reduces with an increase in temperature and dosage of WEO. Fig. 3.8 (B) shows the variation of rut parameter with aging. The nonlinear trend indicates the possibility of interaction of other parameters apart from temperature for determining the rutting parameter.

Addition of WEO resulted in decrease in complex modulus and increase in phase angle values [7,15,16,45]. This trend was also observed by Qurashi [47] and the same is presented in Fig. 3.9. The trend lines in Fig. 3.9 (A) exhibits the variation in complex shear modulus and phase angle at a particular reduced frequency with the addition of WEO. Fig. 3.9 (B) illustrates the changes according to the reduced frequency at a particular dosage of WEO. Another study observed that the resilient modulus and phase angle did not show significant changes [45]. To assess the lowtemperature cracking performance, researchers have used bending beam rheometer [9,18,37,42,48] and single-edge notched bending tests [18]. The test results showed that the low-temperature grade of binder was extended with the addition of WEO. Even though the low-temperature grade is improved, the high-temperature grade was negatively affected and the overall temperature cracking resistance was exacerbated [9]. GPC [18,42] test, MDSC test [42], and FTIR spectrometry [28,42,51,53] were used to ensure the chemical compatibility of WEO-modified binder. GPC showed that the low and medium molecular size fractions decrease with the addition of WEO and this leads to an improvement in low-temperature cracking performance and enhances the intermediate temperature fatigue resistance. The molecular structure of binder varies according to the source, and for the same source, the molecular size distribution changes with the degree of oxidation. MDSC test is used to study the glass transition temperature  $(T_g)$  of binder. Being an agglomerate of complex molecular interactions, the phase transition of the structure is difficult to capture. MDSC explores the thermal susceptibility of binder from its  $T_g$ . Researchers have observed a reduction in  $T_g$  in the presence of WEO [42]. This increases the low-temperature cracking resistance of binder.



**Figure 3.9** Variation in the viscoelastic properties of the binder with the addition of WEO: (A) at reduced frequency of 10,000 Hz and (B) at 8% WEO.

Contrarily, several researchers found that the addition of WEO into the asphalt binder has a negative impact on the binder performance [9,20,28,37,48]. WEO adversely affected the adhesion of binder to aggregate, thus leading to premature stripping and raveling [9,20]. The use of WEO as an additive in asphalt binder may lead to premature cracking in the pavement [17,28,54]. Recycled engine bottoms affect the binder negatively as the strain failure characteristics deteriorate gradually [17]. Researchers have reported that the addition of WEO affects the hightemperature performance [20,40]. MSCR tests showed that the WEO increases the nonrecoverable creep compliance of the asphaltic binder. Studies also showed that there is a reduction in elastic recovery and the rutting resistance [15,16,45]. This in turn leads to low rut resistance [28,45]. Several studies found that fatigue resistance may increase or decrease depending upon the applied strain [18,28,36]. Even though the WEO improves the low-temperature cracking performance of binder, the hightemperature performance will be affected significantly [18,36]. The large-sized molecules and carbonyl functional groups will be reduced by the addition of WEO to asphalt binder [53]. A similar study [28] showed an increase in carbonyl functional group, making the binder susceptible to aging. Some studies showed that the higher amount of paraffin (in WEO) makes it difficult for asphaltenes to dissolve in maltenes. This in turn increases the physical and chemical hardening of binder [48,54]. Double edge notched-tension test [17,36,37] was used to characterize the modified binder performance. The presence of oil adversely affects the thermal cracking resistance and the fatigue cracking resistance by reducing the ductility. The complex structure of the WEO-modified asphalt binder was analyzed by ESEM [36]. The asphaltene structure showed a wider tubular structure. This accelerates the gel formation and results in the reduction in ductile resistance of binder.

Incorporation of WEO into RAP increased the rut resistance, stiffness, and failure temperature of binder. The same research reported increased brittleness of binder at a low temperature and cracking potential. Complex modulus of the binder at unaged condition is reduced after WEO modification of RAP binder. However, increased stiffness and rutting resistance and lowered phase angle were observed after the aged condition [34].

# 3.3.2 Incorporation of waste engine oil into the asphaltic mixture

Various tests such as Marshall mix design, indirect tensile strength (ITS) test [19,46,53], resilient modulus (MR) test, durability test, creep test, and fatigue test [19,20,51,52,55] were used to evaluate WEO-modified mixture performance. ITS gives an idea about the low-temperature and fatigue cracking behavior of pavement. MR test indicates the elastic recovery or rutting behavior [52]. Creep test [14], asphalt pavement analyzer test [52,53], and wheel tracking test [46,51] were used to measure the rutting performance of the mixture. Four-point bending test conforming to specifications was used in a study to investigate the fatigue performance (EN 12697-24) and stiffness modulus (EN 12697-26) of asphalt mixture [46].

Mixing of WEO into RAP binder involves a lot of complex (sometimes impractical) processes. Thus, researchers have attempted to directly mix WEO into RAP mixture. Several researchers have reported that WEO addition makes the asphalt production easier by lowering the compaction and mixing temperature [20,39]. The addition of WEO into cold or hot mixture makes it more susceptible to rutting [19,20]. This may be attributed to (1) reduced bonding between binder and aggregate, and (2) lower density of compacted mixture. To improve adhesion between aggregate and binder and consequent resistance against moisture resistance, Wang et al. [56] used antistripping agents along with WEO. Researchers have found that WEO improves the workability of asphalt mixture with RAP [19,51,55]. Borhan et al. [20] reported reduced Marshall stability and tensile strength with the addition of WEO. Jia et al. [28] reported that an increase in WEO leads to a corresponding reduction in hot mix asphalt mixture stiffness. This in turn increases the resistance of mixture against cracking [14,49]. However, Hesp and Shurvell [37] found that WEO causes physical and chemical hardening and reduced strain tolerance in ductile state. Consecutively the rutting potential increases and fatigue and freeze-thaw resistance of the mixture reduces [28,53]. Further, WEO addition makes the mixture more brittle. Consequently, a reduction in resilient modulus of mixture is observed. The presence of WEO resulted in a higher rate of rutting and inferior performance to static or dynamic creep (elastic and plastic deformation) and lower fatigue resistance [19,20,28,51]. However, researchers have found that WEO improves fatigue resistance and low-temperature cracking of asphalt mixture containing RAP [19,51,55].

## 3.4 Future trends and challenges

With projection of current trends, the number of motor vehicles is expected to increase in the near future across the world. Hence, one can conservatively expect WEO will also be generated in more volume. One of the major challenges has been the homogeneity of WEO-blended asphalt binder. Such homogeneity can only ensure reasonable performance prediction of asphalt concrete. Thus, generated WEO should be collected, organized, and stored under control conditions. Such an approach will help in identification of compatible WEO with intended binder and proper mixing. Due to the fundamental nature of WEO and asphalt binder, it is imperative that the performance of WEO-modified asphalt binder or mixture performance cannot be enhanced over entire range of temperature expected during its service life. This drawback necessitates the usage of certain additives and performance enhancers to improve overall performance. Irrespective of laboratory results, it is necessary to ensure that the finalized mixture performs satisfactorily in field conditions. A highly complex interaction between WEO and asphalt binder further necessitates superior field performance.

## 3.5 Conclusions

Issues such as (1) accumulation and disposal of WEO, (2) similar molecular structure between WEO and asphalt binder, and (3) search for sustainable pavement construction practices have resulted in the usage of WEO in conjunction with asphalt binder and asphalt concrete. Various physical and chemical testing practices have been used to evaluate suitability of blending of WEO with asphalt binder. Some of the major findings are summarized below.

- 1. Incorporation of WEO into asphalt concrete is found to be a viable solution when compared to direct disposal into environment.
- 2. At individual component level, asphalt binder and WEO have similar molecular structure. However, mixing of these two components will result in certain chemical changes and subsequent molecular restructuring.
- **3.** It was observed that the direct inclusion of WEO (without preliminary processing) into binder or mixture will have a detrimental effect on binder/pavement performance.
- **4.** The proportion of RAP content within recycled mixture can be increased with the addition of processed WEO to a certain extent. This indicates that there is an optimal percentage of WEO and RAP up to which reasonable performance can be expected when compared with control binder/mixture.
- **5.** The source, WEO usage history, composition, and physical properties of processed WEO and asphalt binder would have a significant impact on overall properties of binder/mixture. Hence, proper evaluation needs to be conducted before finalizing the extent of usage of processed WEO.

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# Microstructure and performance characteristics of cold recycled asphalt mixtures

4

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## 4.1 Introduction

With the development of the road industry, a lot of waste asphalt mixtures are produced annually all over the world. Waste asphalt mixtures not only take up lands, but also pollute the environment. On the other hand, the lack of high-quality raw aggregates and energy seriously constrains the construction and maintenance of asphalt pavement. Therefore, recycling of asphalt pavement has become a hot topic in the past decades. Nowadays, cold recycling of asphalt pavement is one of the most effective ways to reduce environmental impact and shortage of natural resources [1]. And cold recycling techniques have developed rapidly in countries including the United States, China, Italy, and South Africa. In this chapter, the microstructure development and its effect on the final field performance of cold recycled asphalt mixture will be reviewed and discussed.

Typically, cold recycling of recycled asphalt pavement (RAP) is a type of rehabilitation strategy generally used for the road with low-to-moderate traffic volume in the United States [2]. Cold recycled mixture is used in the inner layer of road due to its high air void and poor performance. Recently, the Virginia Department of Transportation has proved that cold recycling is also cost-effective for the rehabilitation of heavy traffic–volume roadways [3]. Moreover, cold recycling of RAP by emulsified asphalt has been successfully used in expressway with heavy traffic during asphalt pavement rehabilitation in China since 2006 [4,5]. However, there is also no worldwide accepted standard for structure design of cold recycled mixture. Some structural design methods are proposed for cold recycled mixture, including the American structural number method [6], the South African method, and the deviator stress ratio design method suggested in Germany for higher traffic levels.

Fig. 4.1 shows the typical application layer of cold recycled mixture in road all over the world. It is seen that cold recycled mixture is mainly used in lower layers in the road structure, and the application layer of cold recycled mixture is different all over the world.



Figure 4.1 Typical application layer of cold recycled mixture [7-10].

# 4.2 Material composition of cold recycled asphalt

### 4.2.1 Definition

Generally, recycling of waste asphalt mixture can be divided into cold and hot methods [11,12]. Cold recycling of asphalt pavement uses asphalt emulsion or foamed asphalt (FA) to mix the RAP at room temperature, which can take up 60%-90% of RAP and used as subgrade or in asphalt pavement structure [13,14]. Cold recycling of asphalt pavement can also be divided into two types: cold in-place recycling (CIR) and cold central plant recycling (CCPR). During the recycling process, the cold recycled asphalt mixture is formed and compacted as a structural layer in road.

### 4.2.2 Material composition

Typically, cold recycled asphalt mixture is composed of emulsified asphalt emulsion or FA, RAP aggregate, new aggregates, water, and other inorganic additives. Emulsified asphalt or FA and inorganic additives are the major active components in cold recycled mixture, which significantly influence the performance of cold recycled mixture.

For emulsified asphalt, cationic slow-setting asphalt emulsion (CSS) is usually used in cold recycling technology [15,16], which can improve the workability of cold mixture after mixing and prolong the construction duration, especially in summer with high temperature. However, CSS may cause early-stage strength and moisture-sensitive problems because it needs a long time to separate water from asphalt. For FA, half-time and expansion rate are the main parameters that are needed to be cared [17].

Inorganic additives are widely used in cold recycled asphalt mixture, which is beneficial to the strength and durability of cold recycled mixture [18]. Cement is

the most common material used in cold mixtures. The dosage of cement is 1%-4% by weight of cold mixtures. It is reported that cement can significantly improve the early-stage strength, rutting resistance, and fatigue resistance of cold mixtures. Lime is also used to improve the performance of cold mixtures [19,20]. Moreover, some pozzolans including coal waste, coal waste ash, fly ash, and ground-granulated blast-furnace slag can also be used in cold mixtures [21–23].

RAP aggregates obtained from asphalt pavement also influence the performance of cold recycled asphalt mixture. Moisture content of RAP, aging level and content of asphalt in RAP, RAP sources, and the aggregate gradation in RAP are also important factors to design a good recycled mixture. Additionally, the effect of particle size should be taken into account as well.

Water is also one of the important components of cold mixtures. Water is used to wet aggregates and improve the mixing and wrapping of emulsified asphalt [24]. Additionally, the microstructure and durability of cold mixtures are influenced by water content.

#### 4.2.3 Mixture design

Nowadays, there is no worldwide accepted standard to be used for mix design of cold recycled mixtures in spite of some existing manuals and guidelines, such as the Wirtgen Cold Recycling Manual, ARRA, and JTJ F41 in China [25,26]. The modified Marshall method used in China includes three processes. First, the optimum water content of cold recycled mixture was determined by the density of samples prepared by heavy hammer while the asphalt emulsion content was determined as 4.0%. Then, the optimum asphalt emulsion content was determined by indirect tensile strength (ITS) of samples prepared by the Marshall method with optimum water content. Lastly, the performance of cold recycled mixture by emulsion (CRME) was examined by ITS and moisture damage resistance.

Lee [27] developed a volumetric mix-design using the Superpave gyratory compactor for CIR in North America. The design process includes three stages: First, the specimens were cured for 24 h at 60 °C after compaction, and a minimum of four emulsion contents are used. Second, the number of gyrations used to compact the specimens should be adjusted to achieve densities similar to those found in the field. Lastly, the optimal emulsion content was determined as the emulsion content at which the unit weight is at its maximum value.

#### 4.2.4 Construction technologies

Cold recycling consists of two methods: CIR and CCPR. CCPR is a process in which the asphalt recycling takes place at a central location using a stationary cold mix plant. After the cold recycled mixtures are prepared in the plant, the mixtures are transported to the field and paved like the common hot mix asphalt (HMA).

CIR is performed in place with no heating required during the construction process. The process of CIR involves milling and removing the appropriate surface thickness in an existing section, injecting and/or adding modifiers, and recompacting the modified mixture to a specified density [28].

## 4.3 Microstructure in cold recycled asphalt mixtures

### 4.3.1 Analysis methodologies

Cold recycled mixture is a complex material that is composed of asphalt emulsion, Portland cement and water. The inner structure of cold recycled mixtures determines its final performance in road. Nowadays, the microstructure of cold recycled mixtures has caught increasing attention and is found to have a given correlation with its mechanical properties in previous studies [29]. The microstructure of cold recycled mixtures is mainly studied by scanning electron microscopy (SEM), environmental scanning electron microscopy (ESEM), and X-ray computed tomography (CT).

SEM is an observation method that can be directly used to obtain material surface topography and element composition at the microscale. This method is commonly used to analyze the microstructure of materials. In the SEM analysis, electrons are scanned on the specimen surface under the condition of high vacuum, and various types of signals come out of the surface such as secondary electrons, backscatter electrons, characteristics X-rays, and other photons of various energy levels. Thus, the morphology of the interface can be observed and can be then related to the performance of the microstructure.

ESEM is similar to SEM but removes the high-vacuum constraint on the sample environment. Therefore, samples with wet and nonconductive can be tested by ESEM without any damage to the surface. The formation process of microstructure under wet mode can be in-situ investigated.

CT technique, which can be conducted to obtain the internal information of cold recycled mixtures at the microscale, is widely used in engineering materials recently [30]. Air void and its distribution can be obtained during the test.

#### 4.3.2 Microstructure formation

Microstructure formation process of cold recycled mixture was investigated by using ESEM [31]. Fig. 4.2 shows the continuous observation images of asphalt emulsion mortar with RAP aggregate. The spherical particles of asphalt emulsion could be clearly seen in the fresh asphalt emulsion-cement mortar, which is attributed to the initial evaporation of surface moisture in a low-pressure environment of ESEM instrument. During the curing process, asphalt emulsion particles began to merge and the fuse angle was gradually increased between the particles. After 24 h observation, the asphalt emulsion particles gradually formed integral membrane asphalt, and the emulsified asphalt boundaries between the particles were disappeared. It was concluded that the microstructure formation of CRME may be controlled by molecular diffusion of emulsion particles.

Additionally, the asphalt emulsion-cement mortar cured at 20 °C with 60% humidity after 3 days was observed by using ESEM [31]. The ESEM picture is shown in Fig. 4.3. The asphalt emulsion particles and cement-mineral particles were mixed together. The integration of asphalt emulsion particles and hydration of cement particles occurred during the early-stage curing process, and an interface between asphalt emulsion-cement mortar and aggregate was also formed. It was concluded that the hydration of cement is faster than the integration of asphalt emulsion particles, which explains the fact that cement played the key role in early-stage strength during the first 3-day curing.



Figure 4.2 ESEM images of interface formation process in CRME within 24 h [31].



**Figure 4.3** ESEM pictures of interface after 3-day curing (20 °C, 60% humidity). (A) emulsion particles, (B) interface between aggregates and emulsion particles [31].



Figure 4.4 Volumetric composition of CRM and evolution over time [32].

### 4.3.3 Air void distribution

Air void has a significant influence on their mechanical performance and durability. Air void of cold recycled mixtures is significantly higher than that of HMA; the average air void content is usually between 7% and 14%. The high content of air void may cause lower strength of cold recycled mixtures. For cold recycled mixtures, the volumetric composition changes during curing by water evaporation, which is shown in Fig. 4.4.

Yu [35] used X-ray CT to study the microstructure of CIR mixture with emulsion. It was found that the air void size of CIR mixture becomes large with the aggregate gradation changes from fine to coarse. Both compaction method and aggregate gradation significantly influenced the size and the spatial distribution of air void in the mixture.

The air void distribution of cold recycled mixtures was investigated by Gao et al. [33]. Air void of CIR-20 and AC-20 was comparatively studied by X-ray CT. Fig. 4.5A and B shows the CT instrument and the 3D reconstruction of samples, while Fig. 4.5C shows the air void size proportion. The proportions of air voids in the size of 0.1-1 mm for CIR-20 and AC-20 were 13.7% and 7.5%, respectively. It was found that the highest proportion of air voids for CIR-20 mixture distributes in the size range of 1.-2 mm. This proportion was also much higher than that of AC-20 mixture. In addition, only a very small amount of air voids was larger than 5 mm for CIR-20, while the corresponding proportion for the AC-20 mixture reaches about 14% for air voids larger than 5 mm. It was concluded that the air void size of cold recycled mixtures was lower than that of HMA with similar gradation.

### 4.3.4 Morphology of interface

Morphology of interface is usually studied by SEM, and the performance of cold recycled mixtures correlates with its morphology at the microlevel. Many studies



Figure 4.5 Components of CT system: (A) X-ray computed tomography system, (B) 3D image of CIR sample, and (C) air void gradations for CIR-20 and AC-20 mixtures [33].

have been conducted to determine the characteristics of morphology and the interface of cold recycled mixtures, which is beneficial to explain the performance mechanism of cold recycled mixtures.

Dulaimi [36] studied the microstructure of CRME with active mineral powders at 3-day curing and 28-day curing. The results showed that active mineral powders produced more hydration products at 28-day curing, resulting in higher modulus of CRME. Lyu [37] used SEM to observe the microstructure of aggregate-mortar interface and cement-emulsified asphalt mortar. It was concluded that the cement-emulsified asphalt mortar improved the microstructure and the interfacial deformation of the mixtures and formed a 3D network together with the aggregates, which was of benefit for transmitting and dispersing the external stress, restraining the development of cracks, and enhancing the fatigue life of highway. Zhang [38] reported that after 10 days of aging at 60 °C or 5 days of aging at 75 °C, the cold recycled mixture could form a continuous and close-grained microstructure, which was composed of cement hydration products and asphalt binders.

Lin et al. [34] studied the morphology of interface in both early-stage and longterm stage of curing for cold recycled mixture. SEM analysis was conducted and images of CRME at different curing times were obtained. The interface between
cement and asphalt emulsion is a rough state, which indicates the hydrated products are produced at early stage (Fig. 4.6). It is shown in Fig. 4.6C and D that the hydration products were covered by emulsified asphalt membrane with the increase of curing time. Energy-dispersive spectroscopy was used to analyze the elements of the interface in cold recycled mixture. Table 4.1 shows the atomic proportion in the interface of CRME from Fig. 4.6C and D. The proportion of C atoms was relatively high, which may attribute to the fact that C was the main element in asphalt and C element in asphalt was evaporated by electron beams during the test process. The interface was mainly composed of emulsified asphalt membrane and hydration products as well as mineral filler. The interface between asphalt emulsion and cement of CRME was mainly covered by asphalt membrane after long-term curing. Hence, the visco-elasticity of CRME after curing was significantly clearer than that in the early stage and the results were consistent with the phase angle data of CRME at 20 °C.



Figure 4.6 SEM images: (A) and (B) 7-day curing, (C) and (D) fully cured at 60 °C, and (E) and (F) HMA [34].

Moreover, the microstructure of fracture face for CRME after 392-day curing at 20 °C is shown in Fig. 4.7. It is found that the content of C elements is highest in the fracture surface, which indicates that the fracture surface is coating with asphalt film, and the hydration products are coated by asphalt film in Spot 1. Asphalt emulsion was gradually coated on the interface between aggregate and mortar, which is beneficial to the strength development of CRME.

# 4.4 Laboratory performance of cold recycled asphalt mixtures

# 4.4.1 Analysis methodologies

Understanding the performance of cold recycled mixtures is the key to design a durable cold recycled asphalt mixture and predict its future performance.

Elements	Fig. 4.6C			Fig. 4.6D			
	Spot 1 Spot 2		Spot 3	Spot 1	Spot 2	2 Spot 3	
	Atom%	Atom%	Atom%	Atom%	Atom%	Atom%	
С	64.21	51.96	60.84	67.97	76.12	71.13	
0	20.68	18.43	30.4	24.19	17.85	20.59	
Al	0.34	0.47	0.57	0.38	0.59	0.45	
Si	1.64	0.65	0.39	0.35	0.23	0.35	
S	3.05	2.37	1.25	2.16	0.34	2.08	
Ca	10.07	5.82	1.44	1.83	0.62	2.02	

Table 4.1 Energy spectrum analysis on the left of Fig. 4.6C and D.



**Figure 4.7** SEM image of fracture face for CRME after 392-day curing (20 °C, 60% humidity). (A) asphalt mortar, (B) interface between aggregates and asphalt mortar [34].

Early-stage strength, dynamic modulus, rutting resistance, moisture sensitivity, and fatigue durability are extensively studied with the devolvement of cold recycling technology.

# 4.4.2 Early-stage strength

The early-stage (<14 days) strength of CRME is of great significance for saving construction time and reducing the early damage of road [40], which becomes a critical concern for using cold recycled mixture in cold regions or in winter [41,42]. Cold recycled mixture needs 3-7 days of curing in summer and much longer in winter due to the low early-stage strength of CRME [43], which prolongs the construction period and increases the difficulty for traffic organization. Some researchers focused on the early-stage strength of cold recycled mixtures. Extensive research indicated that the cement accelerates the breaking of bitumen emulsion and the setting of the mix, which increases the early-stage strength of CRME [44,45].

Brown found that cement influenced the initial curing process of CRME and increased the early-stage strength of cold recycled mixture [46]. Harvey and coworkers found that the interface between asphalt and aggregate was important for cold recycled mixture by FA and concluded that FA mixtures were weakly bonded granular materials [47,48]. Thomas and Karmas [49] also found that CRME behaved similar to granular materials at early-stage and to conventional asphalt mixture materials after curing. Yan et al. [50] used Hveem cohesion and raveling tests to evaluate the early-stage strength for cold recycled mixtures. It was found that higher cement content leads to a higher cohesion force and a lower raveling loss rate, indicating that adding cement provides a positive effect on the early-stage strength of cold recycled mixture with asphalt emulsion.

Lin et al. [39] studied the development process of early-stage strength for cold recycled mixtures. It was concluded that cement hydration plays the predominant role in early-stage strength of CRME in 0–5 days, while emulsion asphalt plays the predominant role in terminal strength for CRME. At the early-stage, the cement hydration and inner frictional resistance provided the strength for CRME, and the water present at the interface between emulsion asphalt and aggregate led to a low strength of CRME at the early-stage, while the continuous asphalt membrane formed at the interface after curing provided a higher strength for CRME after curing (Fig. 4.8). In addition, Lin et al. [31] found that cement content and moisture content had an obvious influence on the early-stage strength of CRME, while curing temperature and asphalt emulsion content had a lower impact on early-stage strength of CRME. Moreover, accelerating the demulsification of asphalt emulsion could significantly improve the 3-day and 7-day early-stage strength of CRME.

# 4.4.3 Dynamic modulus

Dynamic modulus is one of the most important structural parameters in road design, which has been studied by many researchers. The viscoelasticity of cold recycled

mixture is determined by its dynamic modulus at different temperatures and frequencies. Studies indicated that cold recycled asphalt mixtures exhibited less temperature and frequency dependencies than HMA, but still should be classified as thermo-viscoelastic materials [51,52]. Cizkova and Suda [53] found that the cold recycled mixtures with 3% of cement reached significantly higher values of the elastic complex modulus component at low frequencies and high temperatures, and cement causes the behavior of cold recycled mixture prone to be an elastic material. Gu [3] reported that cold recycled asphalt mixtures generally had lower dynamic modulus than the HMA in the entire frequency range. However, in the high frequency (or low temperature) range, the cold recycled asphalt mixtures showed comparable dynamic modulus.

As mentioned by Lin et al. [34], CRME may also obey the time-temperature superposition principle like conventional HMA, and the viscoelasticity of CRME is weaker than that of HMA. On the other hand, the dynamic modulus of CRME increases with increasing curing time. The master curves of CRM were obtained by dynamic modulus under different curing times. The master curve of CRM and HMA (AC-25) was shown in Fig. 4.9. According to the time-temperature



Figure 4.8 The strength development mechanism of CRME [39]: (A) early stage and (B) after curing.



Figure 4.9 Master curves of cold recycled mixture at different curing times [34].

superposition principle, CRME at low temperature (high frequency) modulus was lower, and thus, the shrinkage stress produced by low temperature was smaller than that in HMA, which was beneficial to improve the temperature shrinkage cracking resistance and rutting resistance.

#### 4.4.4 Rutting resistance

Stimilli et al. [54] studied the resistance to permanent deformation of CIR mixtures containing a high percentage of reclaimed asphalt. The results showed that CIR mixture containing a high percentage of reclaimed asphalt presented better resistance to permanent deformation than AC mixture. Babagoli [55] reported that the addition of cement, lime, or SBS polymer decreases the permanent deformation and improves the rutting resistance of cold recycled mixture. Gao et al. [56] found that there is no significant difference in high-temperature deformations between the CIR rehabilitated pavement structure (4 cm AC-13 + 6 cm Superpave-20 + 12 cm CIR) and the original newly constructed highway pavement (4 cm AC-13 + 6 cm Superpave-20 + 8 cm Superpave-25).

Gu et al. [3] studied the rutting resistance of cold recycled asphalt mixtures following the AASHTO Standard TP79. The CCPR-FA mixture exhibited the greatest rutting resistance, while the CCPR-emulsified, CIR-foamed, and CIR-emulsified asphalt mixtures exhibited less rutting resistance than the HMA. Compared to the CIR asphalt mixtures, the CCPR asphalt mixtures showed much less susceptibility to rutting. This might be because the CCPR asphalt mixtures had coarse gradations than the CIR asphalt mixtures (Fig. 4.10).

# 4.4.5 Fatigue durability

Fatigue durability has been focused due to its relationship with the pavement structure design. Some efforts are made to assess the fatigue performance of cold



Figure 4.10 Accumulated plastic strain curves for cold recycled asphalt mixtures [3].



Figure 4.11 Average fatigue lives versus stress levels for different mixtures [7].

recycled mixture. Leandri et al. [57] studied the effect of cement on the fatigue resistance of CRME. It was found that cement decreased the fatigue life of CRME under high-stress level and increased the fatigue life of CRME under low-stress level. Gao et al. [58] also got the same results of fatigue durability. Wang [59] used a recycling agent and an acrylic copolymer emulsion to improve the engineering performance of control CRME, and it was concluded that the method provided significantly better overall performance than the conventional method.

Sun et al. [7] studied the fatigue characteristics of in-service CCPR and HMA mixtures by indirect tensile fatigue test method. It was reported that there are good linear correlations between the average fatigue lives and stress levels in logarithm coordinates system, and the actual traffic loads led to the reduction in laboratory fatigue lives for both CCPR and HMA mixtures (Fig. 4.11). The laboratory fatigue equations of the mixtures are presented in Table 4.2.

Lin et al. [34] studied the fatigue life of cold recycled mixtures and HMA under different loading strains by means of four-point bending according to AASHTO T 321 specifications. It was found that cold recycled mixture has lower fatigue life than ordinary HMA, especially at high strain (strain > 500 m $\epsilon$ ). The fatigue life of cold recycled mixture was only 1/5–1/10 of ordinary HMA at high strain. Extension of curing time could improve the fatigue life of CRME because curing increased the effective asphalt content and decrease the air void in CRME (Fig. 4.12).

# 4.5 Field performance of pavement with cold recycled asphalt mixtures

A comprehensive field survey should be carried on before starting the recycling process. Failure types and their corresponding severity, reason of distresses,

 Table 4.2 Fatigue equation of CCPR and HMA mixtures.

Mixture types	ESALs of lane A	ESALs of lane B	Laboratory fatigue equations for lane A	Laboratory fatigue equations for lane B	σ <sub>ESAL</sub> (MPa)
CCPR mixture	496,052	3,225,885	$N_f = 918.9 \left(\frac{1}{\sigma_{\rm ESAL}}\right)^{4.82}$	$N_f = 440.6 \left(\frac{1}{\sigma_{\rm ESAL}}\right)^{5.06}$	0.43
HMA mixture	496,052	3,225,885	$N_f = 9642.5 \left(\frac{1}{\sigma_{\text{ESAL}}}\right)^{5.59}$	$N_f = 5980.9 \left(\frac{1}{\sigma_{\rm ESAL}}\right)^{4.72}$	0.50

ESAL, equivalent single axle load.



Figure 4.12 Fatigue life of CRME and HMA [34].

pavement condition index (PCI) parameter, and properties of aged asphalt mixtures should be studied and analyzed to guide the recycling design. There are widespread research works on the field survey of aged asphalt pavements that will be recycled [60,61]. Therefore, this chapter will only focus on the field performance of pavement with cold recycled asphalt mixtures, instead of field survey before recycling.

#### 4.5.1 Performance in the wear course

Section 4.2.2 has discussed the application status of RAP in both low and heavy traffic locations worldwide. There is also another saying that it could be better to reuse RAP in the base layer than in the wear course or top layer, due to the existed serious aging. Fig. 4.1 shows that the RAP was mostly used in lower layers. In Minnesota, roughly one-third of agencies excluded RAP from wear course mixtures. Johnson and Olson conducted a study to determine whether asphalt pavement with RAP in wear course is easy to failure [62]. Their research reported that the wear and nonwear course mixtures performed essentially the same, and the field performance depended more on the dynamic modulus of the underlying layer. The low-temperature performance grade of the new asphalt binder has a larger influence on the field performance than that of the RAP.

#### 4.5.2 Field properties

Papavasiliou and Loizos [63] compared the fatigue life between AC overlay and foamed asphalt (FA) with recycled pavement materials, with cores from the field. The laboratory-estimated fatigue characteristics of FA materials, from the field, were presented with cored FA, while FA ref represented the fatigue law for the FA materials. It is obvious to see that the fatigue characteristics of AC materials were better than that of the recycled materials (Fig. 4.13).

Rogge et al. [64] conducted case studies on the performance of CIR asphalt pavements in the 1980s. A total of 10 projects of CIR asphalt pavement were included, as shown in Table 4.3. The modulus tests were conducted at  $23 \degree C$  with a



Figure 4.13 Fatigue characteristics of the FA and the AC materials [63].

loading frequency of 1 Hz and 100 microstrains. It was found that the strength and fatigue life of recycled mixtures will increase during the first 2 years of service. The increase is the greatest in the first few months (3-5 months), while after that period, the modulus and the fatigue life will still increase but at a reduced rate. This phenomenon may be associated with continued curing of emulsion and the hardening of the original asphalt binder in the RAP materials. They also performed the service life estimation of 32 CIR projects with CIR serving as a wearing course. The service life estimation showed that 40% of projects did not result in significant failure after served for 4.5 years on average, and the CIR projects had a total average life of 7.9 years.

The same strength development trend at the early stage of CIR construction can be found in the study by Sangiorgi et al. [65]. Fig. 4.14 shows the development of stiffness (indirect tensile stiffness modulus) of two mixtures under traffic load, by testing field cores. It is clear that there was a gradual increase in stiffness from day 0 to 60, followed by a reduction until 365 days. As mentioned before, the initial rise of stiffness was probably due to the continued curing of emulsion in CRM, but also the pavement settlement resulted from the traffic load. Fig. 4.15 presents the ITS values obtained for the laboratory specimens and field cores. It shows a 90% increase in ITS for cored specimens of CRM when compared to laboratory specimens, with values close to that of HMA field cores.

Vázquez et al. assessed the dynamic stiffness of a CIR pavement after 2 months in service condition without asphalt mixture layer [66]. The test results were then compared with the results of a reference pavement type BBTM 11A (a type of gap-graded bituminous mixture) (Fig. 4.16). The coherence is an indication of the reliability of the measurement. The dynamic stiffness of the studied CIR pavement was considerably lower than that of the reference pavement within the testing frequency range.

One of the biggest concerns of the recycled asphalt pavement was raveling, which should be less than 2% mass loss with wet track abrasion test. Thomas and Karmas reported a new process CIR to improve the raveling resistance with

Project no.	Year of construction	Average modulus (ksi)		Fatigue lif (1000 cycl	fe les)	Marshall stability (J	I Marshall flow (Ib) (in/10)		flow	Life <sup>a</sup>
		1988	1989	1988	1989	1988	1989	1988	1989	
1	1984	Ι	713	-	138	Ι	2410	Ι	17	9
2	1985	499	531	62	98	1196	2049	22	20	8
3	1985	508	485	109	176	788	1607	33	19	7
4	1986	377	526	54	150	1106	1181	21	18	3
5	1986	607	479	47	58	928	1372	22	17	8
6	1986	530	727	79	250	1171	1597	24	17	9
7	1986	451	487	59	119	1392	1625	29	18	6
8	1988	603	780	28	41	1028	1816	21	19	7
9	1988	253	445	18	26	683	1566	26	17	-
10	1988	490	501	11	24	595	1023	24	17	8

 Table 4.3 Summary data for 10 selected projects of CIR asphalt pavement.

<sup>a</sup>Expected life as wearing course.



Figure 4.14 Development of stiffness under traffic load at 0, 60, 180, and 365 days [65].



Figure 4.15 ITS resistance for the two mixtures (CRM and HMA) after laboratory and trail field compaction [66].

improved chemistry emulsion and enhanced engineered design protocol [49]. In this new process CIR, the old asphalt was considered as black rock, instead of part of binder in the recycled mix. The improved chemistry emulsion was specially designed for quicker early-stage strength, higher residual binder content, and longer durability [66,67]. Table 4.4 compares the differences in raveling loss between conventional and new process CIR mixtures. The raveling loss from the conventional



Figure 4.16 Dynamic stiffness spectra of CIR pavement and reference pavement [66].

Percent raveling loss							
Location	New process CIR (%)	Conventional CIR (%)					
Blue Earth County, MN, CSAH 20 WA SR 270	1.6 1.5	25.7 16.0					

Table 4.4 Raveling test results for conventional and new process CIR mixtures.

CIR sections was more than 16%, while only 1.5% of raveling loss was observed in the sections paved with new process CIR. Thomas claimed that new process CIR with better chemistry emulsion and improved design protocol resulted in a more cohesive mix and hence reducing raveling significantly.

## 4.5.3 Pavement condition and evolution

Kim et al. [68] investigated the relationship between PCI and pavement age of 26 emulsion-stabilized CIR pavements. The thickness of these pavements was approximately 10 cm thick with CIR and overlaid with 5-6 cm of asphalt mixture. Fig. 4.17 plots the PCI of pavement versus age from all the studied CIR pavements. According to the PCI rating scale, 85-100 is good, 70-85 is satisfactory, 55-70 is fair, and 40-55 is poor. It was shown that most PCI values were fair or better, even after served for more than 10 years. CIR pavements might serve between 21 and 25 years. Jahren et al. [69] not only used PCI to evaluate the field performance of CIR asphalt projects, but also defined to predict the expected service life with regression equations, as shown in Fig. 4.18. In total, PCI data from 18 projects



Figure 4.17 Relation between PCI and pavement service life from Kim et al. [68].



Figure 4.18 Expected service life of CIR asphalt projects with PCI data [69].

were collected for the regression analysis (Fig. 4.18). Present serviceability index (PSI) and PCI data show 14-19 and 14-38 years of predicted service life, respectively. The mean predicted service life is 15-26 years under the consideration of both PSI and PCI.



Figure 4.19 Variation of PCI during the inspection period [60].

In Modarres et al.'s study, they performed a 5-year evaluation of a highway in Iran that had been rehabilitated by cold in-place recycling [60]. During those 5 years of inspection period, no considerable structural failure was detected throughout the project in both directions. A significant recovery of PCI was achieved from cold in-place recycling and maintained at a good level. (Fig. 4.19)

In 2005, cold recycled asphalt pavements were constructed in Alabama for field investigation [3]. Four sections of CCPR-F, CCPR-E, CIR-F, and CIR-E (F: foamed binder; E: emulsified binder) were included. According to the measure data within 3 years after construction and 15 years of prediction, it was found that CCPR-F section exhibited much better performance than the other cold recycled pavement sections. The CCPR-E had much more distresses such as rutting and fatigue cracks, although it can pass the performance criteria in the analysis period. Both CIR-F and CIR-E sections need major rehabilitation at the 10th and 11th year of service.

# 4.6 Conclusions and future perspectives

This chapter discussed the fundamental technologies for microstructure development and performance characteristics of cold recycled asphalt mixtures. The inner microstructures, laboratory performance, and field performance has been detailed, reviewed, and concluded as follows:

- 1. Bibliometric analysis shows a steady increasing trend of annual publications on cold recycling of asphalt materials, while China and the United States are leading the research in the point view of peer-review publications. Scientifically, China and the United States have also successfully used CIR not only on low-to-moderate traffic roads, but also on heavy expressways.
- **2.** There is no worldwide accepted standard to be used for mix design of CIR, except some existing manuals and guidelines. These manuals and guidelines explained detailed key technologies for CIR based on field construction experiences.

- **3.** Emulsified asphalt or FA and inorganic additives, such as cement and lime, are the major active components in CIR. Characteristics of RAP aggregate itself has a significant impact on the performance of CIR, while cement can be added to improve the early-stage strength, rutting resistance, and fatigue resistance of cold mixtures. Cement hydration accelerates the breaking of emulsion and leads to a higher cohesion force.
- 4. CIR is a complex material and many new analyzing methods have been applied to understand the hydration process, inner structure development, and interface morphologies. The microstructure formation of CRME may be controlled by molecular diffusion of emulsion particles, and the high air-void content is the key reason to lower the strength of CIR. Understanding the characteristics of aggregate-mortar interface and cement-emulsified asphalt mortar interface morphologies can give a clear prediction on the final performance of CIR.
- 5. Many field studies have been conducted on the performance of CIR on lower layer and wearing course. It was found that the strength and fatigue life of CIR will increase at the early-stage due to the curing of emulsion and the hardening of the original asphalt binder in the RAP materials. Using better chemistry emulsion and improved design protocol will lead to a more cohesive mix and hence PCI with reduced raveling.
- **6.** The qualitative relationship between microstructure and performance of cold recycled mixtures has extensively been discussed, but the quantitative relationship and the critical microstructure parameter need to be established in further research.

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# Life-cycle assessment of asphalt pavement recycling

# 5

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# 5.1 Introduction

The asphalt paving industry and transportation agency are continually searching for cost-effective and environment-friendly solutions to improve pavement sustainability and resiliency when facing the changing climate patterns. One of the most common approaches in practice is to use recycled materials in asphalt pavement [1]. According to the latest survey conducted by the National Asphalt Pavement Association, approximately 79 million tons of recycled asphalt materials were used in new asphalt pavement in the United States which resulted in more than \$2.2 billion saving of total cost as compared to the use of virgin materials [2]. Among the total 79 million tons of recycled materials, the majority (90%) is reclaimed asphalt pavement (RAP) that is the removed pavement materials containing asphalt and aggregates; while the rest are mainly recycled asphalt shingle (RAS). The high-quality and well-graded RAP can be achieved after properly crushing and screening. Most of the RAP can be used in the roadway construction in two ways, as asphalt paving material or aggregate material in base or subbase layers.

This chapter reviewed the recent literature focusing on life-cycle assessment (LCA) of asphalt pavement using recycled materials and provided recommendations for future research. The goal is to access current LCA studies on quantifying environmental impacts [mainly energy consumption and greenhouse gas (GHG) emission] associated with recycling material in asphalt pavement and to provide critical comments and point out the potential research direction.

# 5.1.1 Asphalt pavement recycling techniques

This section includes a brief description of methods used for asphalt recycling, because the knowledge of the detailed recycling processes is helpful for comprehensive understanding of the reviewed LCA studies. Based on the location of the recycling process, RAP can be processed at the central processing facility (in-plant processing) or on the job site (in-place processing). Depending on the temperature at which the reclaimed material is heated and mixed into asphalt paving mixtures, the processing techniques can be classified into cold (below 70°C), warm (70°C -120°C), and hot (above 120°C) recycling [3].

For in-plant asphalt recycling, the RAP material milled from the existing asphalt pavement will be first crushed into the desired gradation. Then, RAP will be incorporated into batch plants or drum-mix plants as substitution of aggregate to produce the new asphalt mixtures. The in-plant recycling can be also divided into cold, warm, and hot recycling. The long-hauling distance of RAP from and to the asphalt pant might be considered as one of the environmental burdens of recycling.

Hot in-place asphalt recycling is a recycling process to repave asphalt pavement using the specialized equipment at the job site for heating, scarifying, rejuvenating, lay down, and compaction. Compared to hot in-place recycling, cold in-place recycling does not require high-temperature heating of the milled pavement. However, additives need to be added for proper mixing at normal temperature [4]. Xiao et al. [5] compared different cold recycling techniques with a focus on different stabilization agents for cold recycling of asphalt pavement, such as bitumen emulsion, foamed bitumen, and cementitious stabilization agents. As the cold in-place recycling is limited to certain weather conditions, warm mixing technique is generally used with the recycling [3].

# 5.2 Life-cycle assessment on recycled asphalt pavement

#### 5.2.1 Life-cycle assessment methods

LCA is a technique to assess environmental impacts associated with a product's life cycle from cradle to grave [6]. The life cycle of pavement can be divided into different stages including raw material extraction, material production, construction, transportation, use phase, maintenance, and end-of-life [7,8].

The formal structure of LCA was framed by the International Standards Organization. It includes four basic stages: goal and scope definition, inventory analysis, impact assessment, and interpretation [6]. The goal of an LCA study is to answer the question of why the study need be conducted. The scope of LCA is to define system boundary, functional unit, data requirement, and study limitation. The inventory analysis includes quantifying the inputs and outputs associated with each component of the product's life cycle within the system boundary. The impact assessment categorizes the inventory data into potential environmental impacts such as energy demand, global warming potential, fossil depletion, acidification potential, smog creation potential, and so on. Additional steps of impact assessment might also include normalization, grouping, or weighting of the results. The last step of interpretation is to summarize all the previous steps including impact assessment results to reach conclusions and recommendations.

There are mainly three LCA approaches: economic input-output model (EIO), process-based LCA, and hybrid LCA. The EIO-based LCA uses the purchases information between industrial sectors to quantify the environmental impacts of the whole supply chain [9]. The process-based LCA disaggregates the project or product production cycle into individual processes or activities independently. The integration of EIO-based LCA and process-based LCA is hybrid LCA, which combines the strengths of both LCA approaches.

#### 5.2.2 Summary of most recent studies

The summary of the most recent LCA studies on asphalt pavement with recycling materials is presented in Table 5.1. The factors under comparison are LCA method, recycling method, recycling materials, inventory data, data uncertainty, and impact categories. The LCA methods used in these studies are mainly process-based LCA. The hybrid LCA of EIO and process-based method has also been adopted by three studies. The recycling methods include hot in-plant recycling, hot in-place recycling, warm in-plant recycling, cold in-place recycling, and recycling as unbound material. The uncertainty analysis of inventory data was only considered in a few studies. The impact categories are mainly energy consumption and global warming potential (GWP); otherwise, the ReCiPe framework is used for obtaining environment impact scores of multiple midpoint and endpoint indicators [20].

Most previous studies conducted LCA of pavement recycling using processbased LCA. Ideally, the system boundary of pavement LCA should include material/acquisition/production phase, transportation phase, construction phase, use phase, maintenance and rehabilitation phase, and end-of-life phase, as shown in Fig. 5.1. However, different combinations of phases in the pavement life cycle were considered in previous studies, as summarized in Table 5.2. All the studies under review have captured material phase. At the same time, transportation and construction phases are also well covered by most studies. Only two or three studies considered end-of-life or use phase in the LCA results; while half of the studies included maintenance phase.

# 5.3 Environmental impacts of asphalt pavement recycling at different phases

#### 5.3.1 Raw material phase

All the reviewed studies on recycled asphalt pavement have included the material phase in LCA. In raw material acquisition, the main benefit of using recycled materials in asphalt pavement is to reduce the usage of aggregate and asphalt binder, which saves natural resource and reduces negative environmental impacts associated with the acquisition processes.

Because approximately 95% (by weight) of AC mixtures is constituted by aggregate, the use of RAP or RAS can reduce quarry operation for the extraction of virgin mineral materials. Farina et al. [11] reported that by using 10% RAP as partial substitution of virgin aggregates, the energy consumption of quarry activities has been reduced by 39.51 MJ/ton and the global warming potential was 2.58 kg  $CO_2/$ ton lower than by using conventional hot-mix asphalt (HMA) mixtures.

The material acquisition of virgin asphalt binder has significantly greater environmental impacts than that of quarry activities. Although the reuse of old asphalt binder could reduce the use of virgin asphalt binder, previous studies have shown that the environmental benefits depend on asphalt binder replacement (ABR) rates,

LCA method	Recycling method	Recycling materials	Inventory data	Data uncertainty	Impact categories	Reference
Process	Hot in-plant	RAP	Literature review	N/A	Energy consumption; GWP	[10]
Process	Hot in-plant	Crumb rubber, RAP	Ecoinvent, literature, interview	N/A	ReCiPe method	[11]
Process	Hot in-plant	RAP, RAS	Public databases, surveys, SimaPro 2014	N/A	Energy Consumption; GWP	[12]
Process	Warm-mix asphalt Cold in-place, hot in-plant	RAP	Material suppliers and contractors, literature review, Ecoinvent	Monte Carlo simulations	ReCiPe method	[13]
Process	Hot in-plant	RAP	Experimental data, literature review	N/A	Energy consumption; GWP	[14]
Process	Hot in-plant, hot in- place, unbound material	RAP	Literature review, interview	Sensitivity analysis of inventory data and transportation distance	Energy consumption; GWP	[15]
Process	Hot and Warm in- plant	RAP	Field data, literature review	Monte Carlo simulations	ReCiPe method	[16]

 Table 5.1 LCA methods and scope in the literature on asphalt pavement recycling.

Hybrid	Hot in-plant	RAP	PALATE Plant data, US EIA, US Bureau of Labor Statistics	N/A	Energy consumption; GWP	[17]
Eco- LCA	Warm in-plant	RAP	Input–output data from Carnegie Mellon University	Monte Carlo simulations	Mass; energy; industrial exergy consumption Ecological exergy consumption	[18]
Hybrid	Cold in-place, hot-in-place	RAP	PALATE	N/A	Energy consumption; GWP	[19]

GWP, Global warming potential; LCA, life-cycle assessment; N/A, not applicable; RAP, recycled asphalt shingle; RAS, recycled asphalt shingles.



Figure 5.1 Illustration of various phases of pavement life cycle in life-cycle assessment.

which is also known as the blending efficiency [10,12]. Here, the ABR is defined as the percentage of virgin asphalt binder that can be replaced by recycled asphalt binder from RAP or RAS. A previous study also showed that the blending efficiency might have a critical impact on the stiffness of asphalt layer and long-term pavement performance [21]. In the study conducted by Yang et al. [12], the ABR varies from 0% to 59% in 11 asphalt mixtures. It was found that the mixture with the higher ABR has lower energy consumption and GHG emissions due to the reduction in virgin asphalt binder used in the mixture. The same trend could be found in the study by Chen and Wang [10], as shown in Fig. 5.2. The higher ABR caused a greater GWP reduction. For example, for the asphalt mixture with 50% RAP, the increase of ABR from 50% to 100% resulted in 10% GHG reduction. Although the results of environmental assessment will be more accurate with the consideration with ABR, not every study from the reviewed literature have included ABR effect in the material and production stage.

Although it is recognized that warm and cold mix technologies are more environment-friendly due to the lower mixing temperature in plant and laydown temperature on site, the production of additives added in the mixing process has a significant environmental impact instead. Vidal et al.[16] concluded that with similar asphalt mixtures, warm-mix asphalt with additive of synthetic zeolites had the higher environmental burden than HMA in the material stage, despite only a small rate of 0.24% (by weight) was added. The reason is that the production of synthetic zeolites is highly intensive in energy consumption, resulting in much higher environmental impacts than asphalt binder. Therefore, cautions need to be made when analyzing the environmental benefits of alternative asphalt mixtures with different chemical additives.

#### 5.3.2 Production phase

In material production phase, hot in-plant recycling has better control in asphalt mixture quality during the production process than in-place recycling. The process of hot in-plant recycling includes grounding the existing asphalt layer, hauling from

Life-cycle components							Reference
Mater	rial	Transportation	Construction	Use phase	Maintenance	End-of-Life (EOL)	
Acquisition	Production						
		•	•	•	•	•	[10] [11] [12] [13] [14] [15] [16] [17] [18] [19]

 Table 5.2 Life-cycle components of reviewed papers.



Figure 5.2 Reduction of GHG emission for different blending efficiencies and RAP contents [10].

job-site to plant, and mixing RAP with virgin asphalt binder and aggregate to produce new asphalt mixtures [22].

Heating temperature and moisture content are the two main driving forces for energy consumption and environmental impacts associated with hot in-plant recycling. If RAP or RAS has a higher moisture content than virginal aggregate, additional heating energy might be required in the mixing process [23]. For RAP, the aggregate with the higher moisture is wrapped by an aged binder that will prevent moisture evaporation when it is exposed to air. A previous study found that RAP had an average moisture content ranging from 3% to 6%, while virgin aggregate usually had the lower moisture content ranging from 2% to 4% [24]. Yang et al. [12] found that water was added to the process of grinding, resulting in significantly higher moisture content in RAS than in RAP.

There are mainly three methods reported in the literature used to calculate energy consumption in hot in-plant recycling process: data-based regression analysis, thermodynamics-based approach, and specific field data from plan production. Yang et al.[12] used multiple linear regression models to build the relationship between energy consumption, heating temperature, and moisture content. The regression model was based on regional plant data in the state of Illinois and a previous study of energy conservation in HMA production provided by Young [25]. Chen and Wang[10] used the thermodynamic model based on heat transfer mechanism to model the thermodynamic of each material in the mixing process. The heat transfer model considered the energy consumption of heating aggregate, asphalt binder, water, steam, and latent heat for moisture vaporization. The energy consumption of HMA with RAP in plant production varied with different RAP contents and moisture contents in RAP, as shown in Table 5.3. The study also included a

RAP content	RAP moisture content						
	2%	3%	4%	5%	6%		
0% RAP 10% RAP 20% RAP 30% RAP 40% RAP 50% RAP	224 224 224 224 224 224 224	224 227 230 233 237 240	224 230 237 243 249 255	224 233 243 252 261 270	224 237 249 261 273 286		

 Table 5.3 Energy consumption of HMA with RAP in plant production with different RAP contents and moisture contents (MJ/ton) [10].

HMA, Hot-mix asphalt; RAP, recycled asphalt shingle.

correction factor of 76% to reflect the energy consumption efficiency in plant production with heat loss.

The studies by Yang et al.[12] and Chen and Wang[10] showed that the energy consumption to produce HMA with recycling material partially depended on the material properties and was not necessarily lower than the one to produce virgin HMA. Giani et al. [13] compared the energy demand of RAP and virgin HMA in plant production stage. The data were from material suppliers and contractors, which showed the electricity needed to produce HMA with RAP was 3% lower than the one to produce virgin HMA. The study collected plant data without modeling the effects of mixing temperature and moisture content.

## 5.3.3 Construction phase

The on-site construction of asphalt pavement using hot in-plant recycling has a similar process to the one using virgin HMA but is different from in-place recycling. The in-place recycling brings the advantage that the milled asphalt pavement can be reused at the same pavement section being rehabilitated, reducing the hauling distance from the job site to asphalt plant or to landfill.

The construction process of hot in-place recycling includes heating, scarifying, mixing, and paving, which demands significantly more energy than the conventional construction process of hot in-plant recycling. A study conducted by Miliutenko et al. [15] indicated that only considering material, transportation, and construction phases, hot in-place recycling caused slightly lower GHG emissions and energy consumption than hot in-plant recycling. The result also showed that the environmental benefits of hot-in place recycling were mainly due to the avoided hauling of paving materials. For using RAP as aggregate material in base layer, the study found that the transportation distance could be the major factor to impact GHG emission and energy demand.

For cold in-place recycling, the construction process includes milling of the existing pavement. After mixing the milled pavement with stabilizer and pulverized materials, a recycling machine lays down the mixture, followed by compaction.

Giani et al. [13] found that cold in-place recycling had a slightly higher impact than in-plant recycling in construction phase, because the recycling machine had the highest diesel demand among different construction equipment. In the study conducted by Cross et al. [19], the energy consumption of material transportation for overlay construction with cold in-place recycling was found to be 30%-44% lower than that of the alternative of milling and overlay and 51%-60% lower than that of the alternative of overlay without milling.

#### 5.3.4 Maintenance phase

The National Center for Asphalt Technology conducted an analysis using the field data collected in the long-term pavement performance program and concluded that asphalt pavement containing less than or equal to 30% RAP had equal overall performance as virgin asphalt pavement [26]. However, in other studies, asphalt pavement with recycled materials was reported to have a greater tendency of deteriorations such as thermal cracking, longitudinal cracking, and fatigue cracking [21]. The shorter life span of recycled asphalt pavement might result in more frequent maintenance and rehabilitation activities, which might offset the environmental benefits of using recycled materials brought from other life-cycle phases.

As LCA should be conducted with the assumption that all comparable alternatives perform equally well to meet the function unit defined in the scope of LCA, the impact of recycling materials on the field performance of asphalt pavement in long run should be considered. One of the challenges is to accurately predict pavement deterioration and determine the maintenance activities within the analysis period. Some studies such as Vidal et al. [16] simply assumed that maintenance activity occurred every 15 years. Giani et al. [13] determined the maintenance strategy based on experts' input: the surface layer was expected to be completely replaced every 5 years, and the base asphalt layer was replaced every 15 years. In the study by Chen and Wang [10], different performance levels were assumed ranging from 70% to 100%, which was defined as the percentage of service life that asphalt pavement with RAP can reach as compared to virgin asphalt pavement, as shown in Table 5.4. The lower performance level indicates more frequent maintenance activities. To maintain the advantage of fewer GHG emissions and less energy consumption, Aurangzeb et al. [17] first suggested using breakeven point of performance level for the asphalt pavement with RAP to outrace virgin asphalt pavement in total life-cycle environmental impacts. The study considered material, construction, transportation, and maintenance stages, but without use phase.

It is worth to mention that the study conducted by Chen and Wang [10] considered temporal aspect of global warming potential in their study. It is the only and the first study to include the time effect of GHG emission in maintenance phase in pavement LCA study. The study used time-adjusted LCA method to adjust one of the major limitations of traditional LCA in accounting GHG emission. The traditional LCA method fails to consider the physical decay patterns of greenhouse gases, lowering the accuracy of LCA results. Using time-dependent decay function of  $CO_2$ , the result of the scenario at 30% RAP content showed that the traditional

Maintenance treatment		100 % performance		90% perfo	rmance	80% performance	
Milling (in)	Overlay (in)	Activity number	Time (year)	Activity number	Time (year)	Activity number	Time (year)
3	4	1	8	1	7.2	1	6.4
6	7	2	16	2	14.4	2	12.8
3	4	3	24	3	21.6	3	19.2
6	7	4	32	4	28.8	4	25.6
3	4			5	36	5	32
6	7					6	38.4

 Table 5.4 Maintenance activities considering different performance levels of asphalt pavement with RAP [10].

RAP, Recycled asphalt shingle.

LCA method overestimated the total GHG emission, leading to 30%–40% greater global warming potential than the result with time effect. However, the study only considered material, construction, and maintenance phase; time-depended GHG emission in use phase should be considered in the future study.

### 5.3.5 Use phase

Use phase is often neglected by most LCA studies on recycled asphalt pavement, although it is known to have significant contribution to the total environmental impact throughout the pavement's life cycle. The most common environmental impacts considered in use phase of asphalt pavement are vehicle fuel consumption and GHG emission due to pavement deformation and surface characteristics. A key question here is what are the impacts of RAP on these impact assessment components in use phase as compared to virgin asphalt pavement. However, many previous studies that included use phase in LCA study failed to answer the question for the following two reasons: first, pavement surface characteristics and service life are not related to the properties of asphalt mixture without or with RAP, and second, if RAP is not in the surface layer, its impact will be minimized.

Vidal et al. [16] made an attempt to include the use phase in the LCA study of HMA and zeolite-based warm-mix asphalt (WAM) with RAP. The study included fuel consumption and emission from vehicles and land occupation change in use phase of pavement. The inventory was based on the database in the EMEP/EEA Air Pollutant Emission Inventory Guidebook 2009. However, the traffic volume and service life were assumed to be equal for all comparable alternatives in the study, neglecting the effect of asphalt mixture properties on pavement performance. The fuel consumption and emission were calculated as a function of vehicle speed and road gradient, simply reflecting the effects of traffic condition and highway geometry. The study found that the use phase has an overall impact of 79%–91% depending on the impact categories of climate change, ozone depletion, human

toxicity, photochemical oxidant formation, particulate matter formation, ionizing radiation, terrestrial acidification, freshwater eutrophication, marine eutrophication, terrestrial ecotoxicity, freshwater ecotoxicity, marine ecotoxicity, agricultural land occupation, urban land occupation, natural land transformation, water depletion, metal depletion, and fossil depletion. Santero and coworkers [27,28] suggested that the impacts of use phase that only considered traffic volume might not be fully attributed to the impact of pavement life cycle, because the impact assessment was unassociated with the structural performance of pavements. Still, these results clarified the relative impacts in use phase within pavement life cycle and the total impact of pavement when compared with other infrastructures.

One challenge of calculating energy consumption and GHG emission in use phase of asphalt pavement is the quantification of contribution by tire rolling resistance due to pavement surface roughness, texture, and deflection. Several models have been developed to describe the relationship between vehicle and tire configuration, climate condition, speed, pavement surface characteristics, and fuel consumption, such as HDM-4 [29], calibrated HDM-4 [30], and MIRIAM [31], which were developed based on vehicle tests on field sections. It is noted that the U.S. Environmental Protection Agency has developed Motor Vehicle Emission Simulator (MOVES) that can be used to calculate emissions from all on-road vehicles at different geographical scales, such as national, county, state, and multistate [32]. The user inputs include road type, grade, vehicle type, speed, fuel type, and time period. However, the effect of pavement surface characteristics cannot be considered.

Araújo et al. [14] emphasized the importance of considering use phase in LCA of pavement. Laboratory tests were used to evaluate the energy consumption associated with tire rolling resistances on the surfaces of different asphalt mixtures. The assessment was conducted using an adapted wheel tracking test (WTT) device, in which the energy required by electrical motor to move the wheel on specimens was measured. It is noted that in this study RAP as recycling material was only added to the asphalt base layer, thus the pavement surface texture was not affected by RAP. The result showed that the use phase of pavement has a dominant influence on the results of LCA. The energy consumption in use phase was 700 times higher than that in construction phase, while the GHG emissions were 1000 times higher than those in construction phase. The authors suggested applying the pavement surface with lower tire rolling resistance for long-term environmental benefits.

Yang et al. [12] considered two models to quantify the impact of pavement surface roughness in use phase. The first model is to describe the International Roughness Index (IRI) progression over time, and the second model is to determine the relationship between IRI and vehicle fuel consumption. The study adopted an available IRI-fuel consumption model [33] that integrated HDM-4 model into MOVES, which was also used by Wang et al. [34] to compare the impact of pavement preservation on GHG emission in use phase. It was found that the increase in IRI from 60 to 123 inch/mile would result in increased energy demand of 0.9% for large trucks and 3.7% for passenger car. In their study, the surface layers of all comparable mixtures were assumed to be the same, RAP was used in the binder course with different percentages. As a result, recycled materials in asphalt pavement had limited impact on use phase. The study focused on the impact of different service lives and maintenance's timings on the development of IRI. The progression of IRI is categorized into two models: well-performing model with service life longer than 15 years and poor-performing model with service life shorter than 15 years. All mixtures with service life ranging from 10 to 17 years are studied. The results showed that in low-volume traffic condition with average daily traffic (ADT) of 6000, if the pavement service life was shorter than 13 years, the additional energy in use phase (as compared to virgin asphalt pavement) would be offset by energy savings of using recycled material from material and production phases. However, in high traffic condition with ADT of 60,000, the breakeven point of pavement service life would be 15 years. It can be concluded that pavement surface roughness and traffic condition are the two critical factors affecting energy consumption and GHG in use phase.

#### 5.3.6 End-of-life phase

At the end of pavement service life, the old asphalt pavement can be recycled in new pavement construction, recycled as other products, or simply landfilled. For landfill, the environmental impact of end-of-life phase should reflect the removal, transportation, and disposal process. If the old pavement is recycled, the impact analysis is not as straightforward as the disposal process of landfill.

One method is that the system of which pavement is being recycled should receive partial or full credit of avoiding environmental burdens brought by virgin material production and landfill process [35]. For example, the full credit of recycling asphalt pavement as aggregate in an unbounded layer is reducing the environmental impact of producing virgin aggregate, transportation associated with the production, and the landfill process of the milled pavement.

The most widely used method of considering end-of-life phase in pavement LCA study is the "cut-off" approach, because it is difficult to predict exactly how the recycled pavement would be reused in the future [36,37]. The "cut-off" approach defines that the environmental benefits and burdens of recycling are assigned to any system that uses the recycled pavement; thus, the current system would not be given any credit of recycling at the end-of-life phase.

Aurangzeb et al. [17] adopted "cut-off" method, ending the study scope with removing all pavement after the analysis period. But the authors excluded the result from the impacts of demolition process at end-of-life phase, because they assumed that all the alternative pavements had similar removal processes. However, the study by Giani et al. [13] included pavement milling and transportation of RAP to the asphalt plant for recycling at end-of-life phase. The study also used "cut-off" approach and assumed that all pavement materials were recycled at asphalt plant. The result showed that the end-of-life constituted 1%-3% of the total energy consumption that included the summations from material, construction, maintenance, and end-of-life phases. In the study by Vidal et al.[16], the end-of-life process was separated into recycling and landfilling. The authors concluded that a part of the

impact reduction of asphalt pavement with RAP was due to the recycling strategy at end-of-life phase. The normalized impact result showed the process of landfill caused 1.8 times GWP as compared to recycling in plant. It is noted that the impacts from recycling process are attributed to material production phase for any system that uses recycled products.

Other techniques of end-of-life assessment include "closed-loop method," "50/50 method," and "substitution method." These methods are fully explained in the study by Nicholson et al. [37]. For "closed-loop method," it is suitable to use when the recycling material has insignificant weight loss. The environmental burden is the average impact of recycling times. The "50/50 method" equally assigns the environmental benefits and burdens to the system that generates the waste and to the system that uses the recycled materials. For "substitution method," all the recycling burdens are associated with the system that uses the recycled material as substitution to virgin materials. Huang et al. [36] conducted sensitivity analysis of different methods for considering end-of-life phase in pavement LCA. The authors concluded that the "cut-off" and "substitution" methods are two extremes of end-of-life allocation. In addition, the supply and demand for recycled materials should be considered in disposal scenarios when choosing the appropriate end-of-life allocation method.

# 5.4 Conclusion and recommendations for future research

The use of RAP or RAS saves the virgin asphalt binder and aggregate, reducing the negative environmental impacts associated with the acquisition processes of raw materials. It is noted that for warm and cold mixing technique, the production of additives added in the mixing process has a significantly high environmental impact. Thus, the inventory of additives in LCA cannot be neglected although only a very small portion of material is added.

In material production phase, the mixing temperature and moisture content of recycled materials are critical in quantifying the environmental impacts. Depending on these two factors, the impacts of using recycled material in the mixing process are not necessarily lower than that of virgin materials. It is suggested that the moisture content of RAP needs to be measured for accurate calculation of heating energy.

In construction phase, hot in-place recycling has the highest energy consumption due to the process of heating, scarifying, and mixing at job-site. The construction equipment for recycling demands significantly more energy than conventional construction process using HMA from in-plant recycling. The cold or warm in-place recycling has slightly higher impacts than conventional paving method in the construction stage, because extra milling and grading processes are needed [13]. For in-place recycling, the reduced hauling distance of paving materials is the major factor causing reduction of environmental impact in construction stage. Therefore, the accurate counting of hauling distance should be included. The use phase of pavement may dominate the environmental impact over the pavement life cycle depending on traffic volume. However, it has often been neglected by most pavement LCA studies. Field data need to be collected to investigate whether using RAP in the surface layer has significant impact on pavement service life and the IRI progression over time. In addition, the appropriate model of tire rolling resistance related to pavement surface characteristics should be used in quantifying use phase impact. Apart from vehicle fuel consumption due to pavement structure and surface roughness, there are other variables that could be included in the use phase, such as radiative forcing due to albedo change, leachate pollution, and land use change.

The end-of-life phase is particularly important for LCA of asphalt pavement recycling. The selection of end-of-life allocation method mainly depends on how the waste material will be reused. The "cut-off" method that is able to leave the answer of the question open is mostly used in previous. The demolishing and land-filling processes are usually assumed to have a limited impact on pavement's life cycle. However, it would be helpful to find out which end-of-life option, recycling or landfill, is the best option under various circumstances.

There are challenges and problems in choosing the method of LCA to access environmental impacts of pavement in general. The inconsistencies in functional unit, system boundary, inventory data quality, and environmental impact categories create great difficulty in comparing the LCA results from different studies to draw any comprehensive conclusions [27,28]. Due to the issues of data reliability, it is critical for pavement LCA study to perform uncertainty analysis for more credible LCA results. For example, deterministic and stochastic analyses of Monte Carlo simulations have been adopted in the reviewed studies. In addition, future studies should consider the time aspect in quantifying GHG emission and other possible time-related impact categories throughout all phases in pavement's life cycle.

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# **Further reading**

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# Part 2

# Pavements for climate change mitigation

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# **Cool pavements**



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# Nomenclature

$\alpha$	albedo
e	emissivity
Ε	evaporation rate
H	sensible (convective) heat flux density [W/m <sup>2</sup> ]
1	latent heat of vaporization of water [kJ/kg]
λ	thermal conductivity [W/m/K]
L	LW radiation [W/m <sup>2</sup> ]
LW	longwave (3–100 μm)
NIR	near-infrared $(0.8-3 \mu\text{m})$
PCM	phase change material
$p_0$	atmospheric pressure [Pa]
<i>p</i> <sub>S</sub>	water vapor saturation pressure at $T_S$ [Pa]
$p_{v}$	ambient water vapor pressure [Pa]
PV	photovoltaic
$R_n$	net radiation [W/m <sup>2</sup> ]
σ	Boltzmann constant [W/m <sup>2</sup> /K <sup>4</sup> ]
S	SW radiation [W/m <sup>2</sup> ]
SW	shortwave $(0.3-3 \mu\text{m})$
$T_z$	pavement temperature at depth $z [^{\circ}C]$
T <sub>air</sub>	air temperature [°C]
$T_{S}$	surface temperature
TEG	thermoelectric generator
UHI	urban heat island
UHII	urban heat island intensity
Ζ.	depth [m]

# 6.1 Introduction

In response to the public health threat posed by global warming and heat waves, cities around the world are looking for adaptation solutions to limit the impacts of these global challenges. Among other solutions, so-called "cool pavements" have been proposed and studied for decades with the aim of countering urban heat islands (UHIs).

The term "cool pavement" is loosely defined, generally referring to pavements engineered to limit their surface temperature and thus their contribution to the UHI effect compared with standard materials. Cool pavements belong to the broader family of cool urban materials, which also include building rooftops and façades.

Initially, cool materials mostly consisted of white or light-colored coatings and materials. Research on this topic began decades ago with high-albedo roofing materials, with a number of papers focusing to determine their potential to reduce urban heat island intensity (UHII) [1–3]. More recently, cool-colored materials have been developed, which are highly reflective in the near-infrared (NIR) band, spanning from 0.8 to 3  $\mu$ m in wavelength. This allows for a wide variety of colors while still providing cooling performance [4]. In addition to reducing urban temperatures, shortwave (SW) reflective materials create a small one-off negative radiative forcing that counters the effects of increasing greenhouse gas concentrations [5]. Similar solutions exist in the case of pavements.

In the case where materials have low emissivity, such as metallic roofing materials, high emissivity coatings can be used to increase longwave (LW) radiant cooling [6]. Due to their relatively high emissivity, this kind of approach is not frequently found in pavements.

Another category of cool material is so-called green materials. These are typically applied to building roofs and façades or street surfaces and rely on the use of green walls and roofs or lawn surfaces [2,7,8]. Indeed, while the benefits of lawns, parks, and trees have been studied and promoted for several decades [1,9], the more recent green facades and roofs make it possible to green both horizontal and vertical building surfaces [10-12]. In addition to cooling cities, green materials have other benefits such as promoting urban biodiversity and reducing rainwater runoff. Green pavement solutions can be found for low-traffic applications.

For high-traffic surfaces that cannot be vegetated, permeable paving materials have been developed [13-16]. These mainly consist of concrete or asphalt concrete with high void content that allows water to drain into the sublayers of the pavement [15,16].

Another way to decrease the surface temperature of urban materials is to use the latent-heat storage properties of a phase change material (PCM) to increase the material's thermal inertia [4].

Cool pavements are of growing interest to cities across the world, which have broadly adopted the goal of countering the combined effects of heat waves and UHIs. This awareness is accelerated by climate change forecasts, which predict an increase in these phenomena and their impacts, with certain cities even facing the risk of becoming uninhabitable all or part of the year [17–19]. These challenges have spurred the interest of urban decision-makers and planners for cooling techniques including cool pavements, consequently stimulating research in the field.

The present chapter seeks to present an overview of the most frequent cool pavement designs and the physical processes their performance is based on. To this aim, Section 6.2 will briefly present the UHI effect with a focus on the urban energy budget. Section 6.3 will present an overview of different cool pavement technologies and describe their future research needs.

## 6.2 The urban heat island effect and the urban energy balance

#### 6.2.1 Urban heat island effect

Urban areas, through a combination of radiative trapping, increased heat storage, wind obstruction, reduced presence of vegetation, low surface permeability, and high concentrations of human activity, create a localized warming effect known as the UHI effect [20,21]. UHI effect is observed as increased urban air and surface temperatures compared with surrounding rural areas, in the order of  $1^{\circ}C-3^{\circ}C$  on average [2]. The air temperature difference, measured as that of the urban area minus that of the rural area, is referred to as UHII.

UHII varies with time and local weather conditions. Typically, high wind speeds and cloud cover tend to decrease it, while clear skies and low winds amplify it [22]. On nights with clear skies and calm winds, UHII can reach as much as 12°C [23]. These conditions are typically reached during heat waves.

While UHI may reduce building energy consumption during winter, the opposite is true in the summer during which cooling demand is increased [24,25]. For Athens, Greece, for example, building cooling energy has been found to double when UHII reaches 10°C, while peak cooling electricity demand is tripled [26].

In addition to negative effects on energy use, UHIs also tend to exacerbate ozone and smog pollution [27], as well as the intensity of heat waves [28]. As a result, their health impacts are increased, making heat waves of particular concern for dense urban areas with intense UHIs, as seen in Europe during the 2003 heat wave, particularly devastating in Paris, France [29].

Luke Howard, frequently referred to as the father of meteorology and the namer of clouds, was the first scientist to formally observe this phenomenon, keeping a strict record of air temperature measurements between his home in the outskirts of 19th century London and the Royal Society building in the city center [30]. Since then, knowledge and understanding of the UHI phenomenon have gradually shifted from empirical descriptions and predictions [23], toward a finer understanding of the physical processes involved [21]. Currently, several urban canopy models have been developed and are able to represent the effects of low-level vegetation, green roofs, trees, and even urban hydrology [31-34]. These tools have proved to be invaluable to evaluate the impact of different urban cooling scenarios at the city scale [35].

#### 6.2.2 The urban energy budget

There are two relevant approaches to considering energy exchanges in cities: the energy budget of an urban volume and the energy budget of an urban surface, also known as urban facet.

#### 6.2.3 Urban volume energy balance

Fig. 6.1 illustrates the energy balance of an urban volume whose upper limit is above the urban canopy layer and whose depth is such that the conductive heat flux is negligible over the time scale considered.

The term *H* represents the convective exchange between the ground and the atmosphere; *lE* is the latent heat flow due to the (possible) evaporation of water present at the surface. The latter is the product of the latent heat of water vaporization *l* and the evaporation rate *E*. Net downward radiation is noted as  $R_n$ .  $R_n$  is composed of four components (see Fig. 6.2): *S* and *L* for incident short and long wavelengths (SW and LW), respectively, and  $S_{up}$  and  $L_{up}$  for SW and LW radiosity, respectively, summarized in the following equation:

$$R_n = S + L - L_{\rm up} - S_{\rm up}R_n = S + L - L_{\rm up} - S_{\rm ref}$$
(6.1)



Figure 6.1 Urban volume energy balance.



Figure 6.2 Pavement surface energy balance.

In addition,  $Q_F$  refers to the atmospheric anthropogenic source term, that is, waste heat due to human activities;  $\Delta Q$  is the heat storage term within the urban materials present in the volume. The term  $Q_A$  represents nonatmospheric heat advection outside of the volume. This exchange can be horizontal, for example, heat transport by a river, but can also be vertical, for example, in the case of geothermal energy. Atmospheric heat advection, when the area around the urban volume is homogeneous, is included in the term *H*.

The energy conservation equation for an urban volume is

$$R_n + Q_F = H + lE + \Delta Q + Q_A \tag{6.2}$$

#### 6.2.4 Pavement surface energy balance

The urban energy balance of a pavement surface is shown in Fig. 6.2.

The term V represents surface conduction into the pavement. This component is apparent at this scale and was internalized in the term  $\Delta Q$  in Eq. (6.2) when considering an urban volume.

The energy balance of an urban facet is given by:

$$R_n = H + lE + V \tag{6.3}$$

Pavement temperature, which influences each of the terms in the previous equation, automatically varies to compensate any imbalance that may arise.

To illustrate the relative importance of these terms, Tables 6.1 and 6.2 provide orders of magnitude of their intensity for a mid-latitude city under clear skies in summer, with Table 6.2 focusing on the terms of the radiative balance.

Clearly, the most important incoming flux is the net radiation  $R_n$ , more specifically solar irradiance S. Compared with a natural area, the main differences in the energy balance in the city are the positive values of day and night H and the importance of the storage term  $\Delta Q$  at night [36]. In addition, latent flows are lower in cities than in the countryside, while convective flows are higher.

 Table 6.1 Clear sky summertime heat flows for a mid-latitude city in W/m<sup>2</sup> [36].

	R <sub>n</sub>	$Q_F$	lE	Н	$\Delta Q$
Day	516	30	158	240	148
Night	-80	20	13	7	-80

Table 6.2	Clear sky	summertime	radiative l	balance for	a mid-latitude	city in	W/m <sup>2</sup>	[36].
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	S	L	Sup	$L_{ m up}$
Day	760	365	106	503
Night	0	335	0	415

When considering the heat balance of a pavement surface, it is more appropriate to expand the radiative term such as to explicitly present inbound and outbound flows in Eq. (6.3):

$$S + L = S_{up} + L_{up} + H + lE + V$$
(6.4)

In this form, the terms on the left represent the inbound flows, while the terms on the right represent the outbound flows. From the perspective of pavement design, the former are fixed by site selection and the presence or absence of radiative masks, while the terms on the right are dependent on the pavement's thermosphysical properties, as detailed below.

As mentioned in Section 6.1, the goal of cool pavements is to limit their contribution to urban warming compared with standard materials. In other words, they seek to modify the surface energy balance so as to reduce the term H in Eq. (6.3). This term is governed by Jürges' [37] formula:

$$H = h(T_S - T_{\rm air}) \tag{6.5}$$

The parameter *h* is the convective heat exchange coefficient, which is dependent on the air circulation conditions.  $T_{air}$  is the ambient air temperature, while  $T_S$  is the surface temperature. Given that *h* and  $T_{air}$  are fixed parameters from the perspective of the choice of pavement material, reducing the term *H* is equivalent to reducing its surface temperature.

Detailing the other terms in Eq. (6.4) provides the following set of equations:

$$S_{\rm up} = \alpha S \tag{6.6}$$

$$L_{\rm up} = (1 - \epsilon)L + \epsilon \sigma T_S^4 \tag{6.7}$$

$$V = -\lambda \frac{\partial T_z}{\partial z}\Big|_{z=0}$$
(6.8)

$$lE = 0.622 \frac{lh}{c_p p_0} T_s \left(\frac{p_s}{T_s} - \frac{p_v}{T_{\rm air}}\right)$$
(6.9)

Eqs. (6.5) and (6.6) relate the physical laws that define SW and LW radiosity, respectively. As can be seen, SW radiosity is solely due to SW reflection, directly governed by the pavement's surface albedo  $\alpha$ . Furthermore, LW radiosity depends both on LW reflectivity and LW emission as governed by the Stefan-Boltzmann law. The latter parameter is dependent on surface temperature and emissivity  $\epsilon$ .

Eq. (6.7) expresses the Fourier heat conduction law under 1D conditions in the vertical direction, mainly impacted by the vertical temperature profile  $T_z$  and the material's thermal conductivity  $\lambda$ .

Finally, Eq. (6.8) governs the latent heat flux for a water film with  $c_p$  the specific heat of air and  $p_0$ ,  $p_s$ , and  $p_v$  representing total air pressure, saturation vapor pressure at the water film temperature  $T_s$ , and partial air vapor pressure at  $T_{air}$ , respectively. A simple description of its physical meaning states that the evaporation rate increases with the water film temperature, the principal driver of the vapor pressure deficit. For better clarity, the latent flow is left in its initial form in Eq. (6.9), obtained by substituting Eqs. (6.5) through (6.8) into Eq. (6.4):

$$S + L = \alpha S + (1 - \epsilon)L + \epsilon \sigma T_S^4 + h(T_{air} - T_S) + lE - \lambda \frac{\partial T_z}{\partial z}\Big|_{z=0}$$
(6.9)

As mentioned in the previous section, the terms on the right depend on the pavement's properties, more specifically on its albedo  $\alpha$ , emissivity  $\epsilon$ , and thermal conductivity, in addition to the presence of water in or on the pavement to permit evaporation. It is by acting on these properties that cool pavements can offer improved thermal performance in cities. In addition, surface temperature is a fundamental parameter.

Modeling this last parameter more fully requires a slight modification of the previous heat budget, obtained by considering a thin layer of pavement of depth dz as illustrated in Fig. 6.3.

The following equation is then obtained, with C the specific heat and  $\rho$  the density of the pavement material:

$$S + L = \rho C \frac{\partial T_S}{\partial t} dz + \alpha S + (1 - \epsilon)L + \epsilon \sigma T_S^4 + h(T_{air} - T_S) + lE - \lambda \frac{\partial T_z}{\partial z} \Big|_{z=dz}$$
(6.10)

This differential equation illustrates the role of pavement heat capacity on regulating the thermal power of the heat flows in Eq. (6.4).

Having explained the fundamental physical laws that govern pavement thermal behavior, we now proceed to analyze specific kinds of cool pavements: reflective, green and evaporative, high-inertia, and high-conduction and heat-harvesting pavements.



Figure 6.3 Energy balance of a pavement slab of thickness dz.

### 6.3 Overview of cool pavement technologies

We begin by focusing on reflective pavements.

#### 6.3.1 Reflective pavements

In order to reduce their surface temperature, reflective pavements focus on limiting their absorption of solar energy, principally by an increased albedo compared with standard pavements.

Albedo is defined as the proportion of radiation reflected by a horizontal surface. This parameter is dependent on the spectral composition of the incident radiation. On Earth, albedo is determined according to the spectral composition of solar radiation. Usually, the spectral composition of global irradiance for air mass 1.5 (AM1.5) at sea level is considered, available from ASTM G173 and illustrated in Fig. 6.4. The dashed vertical lines delimit the visible (VIS) light spectral range (380–740 nm).

Two principal ASTM standards are used to measure albedo: ASTM E903 for laboratory measurements using a UV-VIS-NIR spectrophotometer and integrating sphere [38] or ASTM E1918 for measurements in the field using a pyranometer or an albedometer [39].

Solar energy at sea level is partitioned as follows: 3% in the ultraviolet (UV) range, 49% in the VIS range, and 46% in the NIR range. Consequently, engineering efforts focus on increasing the reflectivity of pavement materials in the VIS and NIR range.

There are two types of reflection: specular and diffuse. Specular reflection is similar to that of a mirror, that is, incident light is reflected in a specific direction



Figure 6.4 Standard solar spectral irradiance for air mass 1.5 (AM 1.5).

only. Diffuse reflection is similar to that of a matte-white painted wall, that is, reflected light is scattered in all directions. In reality, most materials display a combination of both behaviors, which may vary according to the angle of incidence. Typically, asphalt concretes display mainly diffuse reflection at close-to-normal angles of incidence and specular reflection at grazing angles of incidence.

Standard paving materials span a range of possible albedo values. Asphalt pavements have the lowest values, typically 0.05 for new asphalt concrete, which increases with aging and soiling, reaching up to 0.15. Concrete pavements are brighter and can reach an albedo of up to 0.35 for standard Portland cement concretes. On the contrary, their albedo decreases with time due to soiling, typically reaching 0.2 within a few weeks or months following installation. For pavers, typically used for pedestrian applications, their albedo varies with their composition. Reflective concrete pavements have been shown to reach albedos of up to 0.77, while cool coatings applied to asphalt concrete can have an initial albedo of above 0.5 (Fig. 6.5).

For asphalt surfaces, aggregate albedo is an important factor in pavement albedo, while for concrete surfaces, this depends greatly on cement albedo. This is caused by the structure of both pavements and how they age with wear. While road traffic tends to wear away the bituminous binder, which has low albedo, from asphalt concrete, thus exposing its aggregates, cement is not worn off from the concrete. Its aggregates are therefore less exposed, even after road wear. On the other hand, the cement becomes soiled and stained from road traffic.



Figure 6.5 Cool pavement test samples under study at the Lawrence Berkeley National Laboratory.

To increase the albedo of a pavement, several solutions have been developed. These range from the use of light-colored binders or aggregates to deploying a reflective coating or thin surface layer for asphalt pavement structures [40]. The added layer can be composed of concrete, in which case the process is known as "whitetopping." Light-colored aggregate chip seals can also be used [41]. Alternative designs can use a resin-based binder and alternative aggregates [42]. For concrete, white cement can be used although its production is significantly more energy-intensive than regular gray cement. Alternatively, concrete doped with TiO<sub>2</sub> or slag cement can also be used. The surface layer of both asphalt and concrete pavements can also be painted over with a reflective coating.

To limit the glaring effect of reflective pavements for pedestrians or drivers, cool-colored pavements have been proposed, such as a NIR-reflective paint coating for pavements studied experimentally by Kinouchi et al. [43] or Wan et al. [44] or thin-layer asphalts by Synnefa et al. [45]. The latter reported surface temperature reductions ranging from 5°C to 15°C depending on the coating compared with standard asphalt concrete. Anting et al. [46] tested the performance of NIR-reflective ceramic waste tiles, which were up to  $4^{\circ}C-6^{\circ}C$  cooler than a reference asphalt concrete tile.

By reducing their surface temperature, reflective pavements have been found to reduce air temperatures by up to  $0.9^{\circ}$ C for cities in California, results varying per city according to local conditions [47]. Taleghani et al. [48] obtained comparable results in their study of reflective pavements applied to Los Angeles. Many numerical studies of the phenomenon can be found in the literature, which report 1-2 m height air-temperature cooling in the order of  $1^{\circ}$ C or less [45,49–51].

One of the limits of reflective materials is surface aging and soiling, which cause performance degradation [52], though accelerated aging methods have been developed to determine this evolution ahead of time [53,54].

This is a very important factor for pavement materials that are exposed to very intense urban pollution and to high levels of abrasive wear and soiling associated with road traffic [41,51,55,56]. In addition to encountering significant difficulties in obtaining high-albedo aggregates and in deploying high-albedo cement for cool asphalt and concrete pavement construction, Rosado et al. [41] observed rapid albedo degradation due to traffic in under 10 weeks. In their experiment, chip seals with high-albedo aggregates had the most durable results compared with coatings. Furthermore, in cases when a surface layer is added onto a traditional pavement, mechanical issues may arise, limiting the pavement life span [57].

Another concern in the application of reflective materials is their impact under winter conditions. Indeed, although it is only to a lesser extent, cooling observed in the summer also occurs in the winter, thus potentially increasing building heating demand. Recent work on cool roofs has indicated that the benefits in summer are not offset by winter penalties for cold climates [58]. More recently, thermochromic coatings, which change color depending on their temperature, are being developed and studied in the laboratory. These can be designed to increase their albedo above a certain temperature and remain dark below it. These coatings offer a chance to enjoy the cooling properties of high albedo in the summer without affecting energy

demand in the winter [4]. These materials, which already face short life spans for rooftop or façade applications, have being investigated by a handful of research teams when added to asphalt binders [59].

Another avenue for future work on reflective pavements involves assessing and reducing their environmental impacts. Indeed, it has been found that in certain cities, cool coatings, chip seals, or bonded concrete overlaid on asphalt provide fewer life-cycle gains than penalties [60].

Another challenge that has been identified is the risk that under certain conditions, the increase in reflected radiation caused by high-albedo materials can have net negative impacts on pedestrian thermal comfort, defeating their purpose altogether [61]. In their findings, Erell et al. [61] found that it is high-albedo building façades under certain urban and insolation configurations that are the principal source of the increased stress for pedestrians.

The same phenomenon of street canyon multireflections limits the street-scale impact of diffuse reflective pavements as part of the energy reflected away by the reflective pavements is absorbed by the adjacent buildings. In principle, this is not a concern for retroreflective pavements, which preferentially reflect incident radiation back in the same direction, that is, back toward the Sun [62].

#### 6.3.2 Green and evaporative pavements

Green and evaporative pavements do not rely on higher albedo to reach lower surface temperatures. In fact, evaporative pavements are often less reflective than their standard counterparts, particularly in the case of pervious pavements. Instead, they release absorbed solar heat in the form of latent heat because of the evaporation of liquid water. This is accomplished by the evaporation of water trapped in the pavement material, with (green) or without the help of a herbaceous vegetation layer and the evapotranspiration of water they provide during photosynthesis. In both cases, water must be present in the pavement and available for evaporation or evapotranspiration. The more water is available in the material, the longer the cooling effect will last.

Evaporative pavements often rely on pervious materials, with or without water retention. The terms porous, pervious, and permeable are often used interchangeably to describe a material that allows water to flow through it. However, porosity is a measure of the proportion of voids in a given material, while its permeability is a measure of its ability to allow a fluid to pass through it, as illustrated in Fig. 6.6.

Porous pavements are usually obtained by increasing the proportion of large aggregates while reducing the proportion of fine aggregates in the pavement mix. The binder can be cement-, asphalt-, or resin-based. The void content is typically in the 15%-25% range. Due to the presence of these voids, the mechanical strength of pervious pavements is usually lower than that of equivalent impervious pavements, typically 4-15 MPa. Increasing the proportion of binder can compensate for this with compressive strengths of up to 32 MPa, with limited reductions in permeability [63]. In addition, thicker layers of pervious pavement can be used to reach the same resistance.



Figure 6.6 Examples of pervious concrete textures (top left and right) and demonstration of permeability (bottom).

Permeability can be measured in the field using the ASTM C1701 test method based on the constant head principle for pervious concrete pavements [64].

To maintain their permeability, pervious pavements may require cleaning by vacuum sweeping, particularly in low-speed traffic areas, while pervious highway pavements are considered to clean themselves with high-speed traffic. However, this may not be a problem with regard to lowering evaporative pavement surface temperature as clogging tends to promote capillarity to a certain degree. However, good drainage, principally ensured by sufficient pore size, is necessary to avoid issues with freeze-thaw in winter.

When dry, the air included in the voids of pervious pavements acts as a thermal insulator, increasing its surface temperature amplitude while dampening the temperature signal in the base layers. This is positive in winter, resulting in fewer occurrences of freezing temperatures under the surface course as observed by Kevern et al. [63] with cement concrete pavements. However, under hot weather conditions, this can lead to warmer surface temperatures for dry pervious pavements than for standard ones, that is, a negative impact for urban climate [16,63,65].

Pervious pavements are often promoted as efficient tools to reduce stormwater runoff and improve runoff water quality, in addition to reducing rolling noise from traffic and improving road safety by providing a drier surface during rain events. However, while pervious materials are very useful in offsetting rainwater runoff into sewer systems, they are not necessarily very efficient at providing evaporative cooling if they do not include a mechanism for water retention. Indeed, only water stored close enough to the surface will evaporate rapidly due to thermal and aeraulic resistance of the porous material [66,67].

Water-retaining materials make up for this by storing water close to the surface. This can be done because of capillary action that keeps moisture present at or near the pavement surface. This can be achieved by grouting the pores in pervious asphalt with a cement-based material that remains porous but with higher capillarity. This property has been clearly demonstrated by several studies that compared water-retaining pavements with porous ones, the former reaching surface temperatures up to 13°C lower than the latter [14]. Water-retaining pavers can also be created by sealing the bottom and sides of pervious concrete slabs, as studied experimentally on individual pavers by Qin et al. [68]. Storing approximately 9.5 mm of water, that is, 9.5  $L/m^2$ , the water-retaining block paver is found to maintain cooling for approximately 30 h as opposed to its pervious and impervious counterparts, which provide only significant cooling immediately after sprinkling. This performance is reached despite the fact that the water-retaining paver has lower albedo, that is, approximately 0.22 versus 0.35-0.40, respectively. This evaporative cooling resulted in surface temperatures 2°C-10°C cooler than pervious and impervious pavers. When controlling for the difference in albedo by painting all surfaces black, this cooling effect is prolonged to 60 h.

A number of studies of the potential for evaporative pavements to cool cities can be found in the literature [14,16,66,69–74]. These indicate that surface temperatures can be reduced in the order of  $5^{\circ}C-15^{\circ}C$  compared with standard impervious asphalt concrete, depending on the thermal properties of the dry pavement. The best and longest performance is obtained with water-retaining pavements.

In addition, several authors have studied active evaporative cooling techniques of standard pavement structures, often referred to as pavement-watering or sprinkling [75–84]. Hendel et al. [77,80], relying on street-cleaning trucks to sprinkle water in their experiment, reported surface temperature reductions of  $10^{\circ}C-15^{\circ}C$  during pavement insolation, resulting in air temperature reductions of up to  $1^{\circ}C$ . Fixed infrastructures, such as used in Japan by Kinouchi and Kanda[83] and Takahashi et al. [75], could be included in the design of future pavement retrofits in strategic urban areas.

Green materials are not generally included among cool materials, although they do provide lower surface temperatures compared with their standard counterparts. Few green pavements are available, though structures known as grid pavements can be found with grass-planted soil in the interstices or reinforced turf. Similar structures can be obtained with pavers by simply leaving openings between them. This can be as simple as increasing the joint width between pavers, as has been adopted in a number of cities such as Paris, France. Fig. 6.7 presents a picture of a grass paver structure.

A thorough comparison of the cooling effects of 37 different kinds of green pavements has been conducted by Takebayashi and Moriyama[85] in a parking lot in Kobe City, Japan. The authors reported surface temperatures of up to 25°C cooler than standard asphalt concrete parking spaces. The solar reflectance of these



Figure 6.7 Photograph of a grass paver (credit: I. Giel).

structures ranges from 0.165 to 0.246, illustrating the significant cooling impact of evapotranspiration. Similarly, grass lawns generally exhibit surface temperatures approximately 20°C cooler than standard pavements [66,74].

Grass lawns, which could be considered to represent an extreme of the spectrum of green pavement materials, generally offer the best performance with up to  $20^{\circ}$ C in surface temperature reduction compared to standard pavements. However, it should be noted that this performance is entirely canceled out once the grass is dry and has died out, as illustrated in Fig. 6.8.

Generally speaking, green pavements are not suited to high mechanical stress applications such as high traffic and will not fare well in areas that require snow plowing or the use of deicing salts in winter.

#### 6.3.3 High-inertia pavements: phase-changing materials

To reduce their surface temperatures, high-inertia pavements have neither higher reflectivity to limit solar heat absorption nor porosity to release absorbed heat by evaporating water. Instead, they attempt to offer a higher-than-normal thermal inertia. It is quite difficult to increase the inertia of concrete or asphalt concrete by changing formulations, for example, aggregates. However, it is possible to add PCM in their mix, which can be chosen or designed to change phase at a desired temperature. The inclusion of PCMs aims to increase the thermal mass C of the pavement surface layer, thus dampening the rate of temperature change as per



Figure 6.8 Infrared photographs of dried out (left) and healthy (right) grass in the sun (top) and shade (bottom).



Figure 6.9 Cross section of a high-inertia (PCM) pavement.

Eq. (6.10). The layer including PCMs is not necessarily the surface course, as illustrated in Fig. 6.9.

From a thermodynamic point-of-view, the phase change of a pure substance takes place at constant temperature and pressure. In other words, a pure substance solid melts into a liquid at constant temperature, while absorbing heat. If the process is reversed, resolidifying the obtained liquid releases exactly the same amount of heat at the same constant temperature. This is the same process when evaporating water from a liquid to a gas or melting water ice to a liquid. However, changing from a liquid to a gas is impractical as the specific volume of the substance changes very significantly. This is not the case when changing from a solid to a liquid, which are both condensed forms of matter. PCMs therefore focus on the latter phase change and are also called solid-liquid PCMs.

Contrary to the evaporation of water in green and evaporative pavements, PCMs are trapped inside the pavement. Therefore, as the pavement cools down at night,

the melted PCMs will resolidify, releasing the same amount of heat absorbed during the day. The amount of energy stored and released by the pavement is therefore not modified by the inclusion of PCMs. However, they do lower the rate of sensible heat exchange with the atmosphere.

Effectively, adding PCMs to a pavement increases its thermal inertia. While they absorb heat during the day, the temperature of phase-changing pavements will rise more slowly than traditional pavements. At night, the process is reversed: their temperature will reduce more slowly as they release the heat absorbed during the previous day. On average, this results in temperatures lower than standard pavements during the day, but higher at night.

As such, phase-changing pavements will likely not be effective means of limiting the UHI effect, because they will tend to exacerbate the reduced rate of nocturnal temperature decrease in cities. However, they are effective at limiting urban heat, that is, limiting maximum temperatures during the day.

Typical PCMs are organic (bio- or petroleum-based paraffins, carbohydrates, or lipids), inorganic salt hydrates, or eutectics, that is, not a pure substance. The principal design characteristics of PCMs are their melting temperature, latent heat of fusion, cyclability, and thermal conductivity. Melting temperature determines the temperature at which phase change occurs and should fall in the range of temperatures reached within the pavement during the climate cycle being addressed. Latent heat of fusion is a measure of the amount of heat absorbed to fully melt the PCM or released to fully solidify it; desirable values are generally considered to be 200 kJ/kg or higher. Cyclability describes the number of fusion-solidification cycles that a PCM can undergo before its performance degrades. Finally, a PCM's solid-phase thermal conductivity is an important parameter in order to ensure rapid heat conduction into the solid PCM to facilitate melting.

PCMs can be included in materials directly, although this is generally not recommended as they may flow through the material's pores when in liquid phase. This may also pose issues with mechanical strength, specifically for concrete [86]. PCMs are often microencapsulated to prevent this, but this is not resistant to hotmix asphalt temperatures. To solve this issue, PCMs can be included in lightweight aggregates used in asphalt concrete mixtures, as detailed by Ryms et al. [87].

Several studies focused on the use of PCMs to reduce freeze-thaw in concrete and asphalt pavements [88-93]. In addition, some studies focused on the use of PCMs to reduce concrete curing temperatures or asphalt setting temperatures [94-96].

However, relatively few articles focus on their use to mitigate urban heat [87,97-101]. In the laboratory, Chen et al. [101] observed temperature reductions of 2°C for PCM pavement samples. Surface temperature reductions of about 5° were reported by Ryms et al. [87,98] for PCM asphalt concrete containing 3% PCM by total weight. Athukorallage et al. [102] investigated the use of adding a PCM layer beneath the surface course in asphalt concrete pavement structures. This was found to be counterproductive due to the low thermal conductivity of PCMs compared with pure asphalt concrete. However, a maximum surface temperature

reduction of  $4^{\circ}$ C was found if the surface course includes an optimum 30% PCM by volume.

These surface temperature cooling effects are significantly lower than what can be obtained with reflective or green and evaporative pavements. Furthermore, adding PCM to concrete and asphalt concrete pavements poses challenges in terms of workability, mechanical strength, and PCM inclusion.

Further research is necessary to achieve sufficient surface temperature cooling for phase-changing pavements to merit application and become competitive with reflective or green and evaporative pavements.

#### 6.3.4 High-conduction and heat-harvesting pavements

High-conduction and heat-harvesting pavements both increase the surface conduction term in Eq. (6.10). They reach this goal either by increasing the thermal conductivity  $\lambda$  of the pavement structure or by increasing the temperature gradient by reducing temperatures below the surface layer, typically via a heat exchanger. In the latter case, the heat exchanged can be harvested and put to productive use. Overall, high-conduction pavements have been studied to a lesser extent than heat-harvesting pavements.

A number of different approaches have been proposed to increase the conductivity of pavements, which is principally determined by the conductivity of the binder and aggregates, as well as the void content (Fig 6.10). In certain cases, highly conductive powder can be added, such as graphite. Indeed, Yinfei et al. [103] found that adding low-conductivity material to the surface layer of asphalt concrete pavement increases surface temperature, as opposed to the addition of graphite that had the opposite effect. This design was studied with the aim of reducing temperatures deep inside the pavement, for example, to limit permafrost thawing or rutting. Yinfei and Shengyue[104] further observed surface cooling in the order of 1°C following the addition of vertical steel rods in the mid-course of an asphalt concrete pavement structure with the same low-conductivity surface course. An improved version of the steel rod pavement was found to reduce surface temperatures by up to  $3.5^{\circ}$ C without increasing rutting [105]. Even better daytime surface temperature reductions can be expected if the conductivity of the surface course is increased instead of being decreased.

Heat-harvesting pavements belong to a larger category of energy-harvesting pavements. These include pavements that produce electricity such as photovoltaic (PV),



Figure 6.10 Conductive pavement based on embedded vertical metallic rods (left) or a high-conductivity layer (right).

thermoelectric, and piezoelectric pavements, which, respectively, harvest sunlight, heat, and traffic-induced mechanical vibrations, and heat-harvesting pavements, which directly harvest heat from the pavement structure. Heat-harvesting and PV pavements can also be referred to as solar pavements or solar collector pavements [106,107].

The energy produced by solar pavements creates an additional outbound flow  $\dot{q}$  not included in Eq. (6.10). By adding this term to the right-hand side of Eq. (6.10), we obtain

$$S + L = \rho C \frac{\partial T_S}{\partial t} dz + \alpha S + (1 - \epsilon)L + \epsilon \sigma T_S^4 + h(T_{air} - T_S) + lE - \lambda \frac{\partial T_z}{\partial z} \Big|_{z=dz} + \dot{q}$$
(6.11)

As can be seen, the energy produced by solar collector pavements directly contributes to reducing its temperature, provided that solar pavement design does not significantly affect the other preexisting outbound flows.

Noting  $\eta$  as the overall efficiency of the solar pavement and its components,  $\dot{q}$  is expressed by:

$$\dot{q} = \eta S \tag{6.12}$$

PV pavements are included in this section even though they do not actually harvest heat from the pavement but sunlight, which is the source of the heat harvested by the other solar pavement designs.

#### 6.3.5 Photovoltaic pavements

PV pavements consist of a PV panel and its electronics, reinforced to bear traffic loads, placed on the surface layer of a standard pavement structure, as illustrated in Fig. 6.11. Based on the photoelectric effect, the PV panels turn sunlight into electricity.

Few research publications are available on the energy efficiency and surface cooling potential of PV pavements [107-109]. The energy efficiency of PV pavements, due to the addition of a reinforced glass layer on top of the solar cells, is typically lower than that of standard PV panels or cells. Efthymiou et al. [109] found a 2% drop in energy efficiency due to the reinforced glass surface of the photovoltaic pavement, with an initial panel efficiency of 14%. Dezfooli et al. [108] reported that PV pavement efficiency is half that of the reference solar cell; however, its initial efficiency is very low (1.1%). Xiang et al. [110] indicated PV



Figure 6.11 Diagram of a PV pavement design.

pavement efficiencies in the order of 9% due to the negative impact of high pavement temperatures. They proposed a hybrid PV-thermal approach to improve electrical efficiency to 11.5% and reach a combined thermal and electrical efficiency of between 45% and 67%.

In terms of surface temperature cooling, Efthymiou et al. [109] reported a reduction of up to 8°C for PV pavements compared to standard asphalt concrete.

#### 6.3.6 Thermoelectric pavements

To turn heat into energy, thermoelectric pavements rely on the Seebeck effect, which can produce an electric field from a temperature gradient between the hot and cold junctions of a thermoelectric generator (TEG). TEGs can either be embedded in the pavement and exploit the pavement temperature gradient directly, as shown in Fig. 6.12, or they can be combined with a pipe-pavement for use with pavement-heated and cold water [106,111].

Few experimental studies of TEG pavements exist under realistic insolation conditions. However, Jiang et al. [112,113] studied a TEG system including horizontal aluminum heat conductors connected to TEGs placed on the side of the pavement structure, making use of a temperature difference of up to  $10^{\circ}C-20^{\circ}C$  between the pavement and the ambient air. The authors report production of up to 33 Wh/m<sup>2</sup>. In the best summertime conditions, with temperature differences in the order of  $20^{\circ}C-30^{\circ}C$ , this is expected to reach 16 Wh/m<sup>2</sup> over 8 h. This value is rather low, representing an average outbound heat flow of 2 W during the insolation period. This is explained by the TEG's low efficiency, that is, approximately 5% of the power available from the temperature difference.

In terms of pavement cooling, the TEG pavement design exhibited surface temperatures up to 10°C cooler than traditional asphalt concrete in summer. This temperature reduction is misleading and should not be considered out of context. Although this value is indeed reached in the described experiment, only 5% of the extracted heat is turned into electricity, while the rest is released to the atmosphere.

#### 6.3.7 Heat-exchanger pavements

The most studied solar pavements are those including a heat exchanging layer [114-131]. Embedded pavement pipes or a pervious pavement layer are used for this purpose, with either water or air circulating as the heat-transfer fluid, as



Figure 6.12 Embedded thermoelectric pavement cross section.



Figure 6.13 Cross section of an embedded-pipe (left) or porous-layer (right) heat-exchanger pavement.

depicted in Fig. 6.13. Harvested heat in summer can be used to supply hot water for nearby buildings or can be stored underground for use in winter for snow-melting of the heat-exchanger pavement.

Embedded-pipe heat-harvesting pavement performance in terms of surface temperature reduction has not been thoroughly studied to date. Zhou et al. [114] reported surface temperature reductions of  $3^{\circ}C-6^{\circ}C$  during the summer day. Using air as the heat-transfer fluid, Chiarelli et al. [119,120] reported pavement cooling of up to  $6^{\circ}C$ .

It is clear, however, that design parameters such as fluid temperature, fluid flow rate, pipe depth, and distance between pipes have a strong influence on surface temperature [115,116,127,129,132]. Embedded pipes pose a certain number of engineering challenges in terms of stress and corrosion resistance as well as maintenance. Typical solar energy efficiency with embedded pipes is in the order of 25%-40% [114,121,123].

Alternative approaches discard the need for pipes altogether by relying instead on a pervious sublayer in which the heat-transfer fluid flows [130]. Temperature reductions are more uniform with this approach and energy efficiencies of up to 85%.

Some systems are coupled with geothermal energy systems to store excess heat in summer for use in winter, including pavement deicing [106].

#### 6.3.8 Combined cool pavement designs

Recent work has outlined the improved impact of combined UHI mitigation techniques [133]. One such example has been studied by Higashiyama et al. [134] who combined porous asphalt concrete with a light-colored and absorbent sealer based on a ceramic powder made from waste. Although its impact on albedo was not reported, the authors reported surface temperature reductions from 9°C to 20°C compared to standard porous asphalt concrete for the dry pavements, that is, without rain or sprinkling.

Other venues can also be explored though they usually require a trade-off, for example, reflective or evaporative solar pavements that may offer even cooler surface temperatures in exchange for reduced harvesting efficiency. Another example, from Ref. [135], concerns heat, or rather coolth, harvesting from a pavement-watering infrastructure.

The multitude of potential combinations offers many future research possibilities.

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# Reflective coatings for high albedo pavement

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## 7.1 Introduction

With the development of urbanization and population density, roof and pavement count for 50%-60% of urban areas [1,2]. Conventional roof and pavement almost all applied solar radiation absorption materials, especially the asphalt concrete pavement in black with a high heat flux [3]. In summer, the peak temperature of new asphalt pavement could be higher than  $65^{\circ}$ C [4,5]. The potential way in geoengineering to mitigate the urban heat island (UHI) effect and reduce the greenhouse gas emission is adopting green roof and cool pavement [6–8]. Cool pavement can be classified into several types: reflective cool pavement, evaporative cool pavement, and heat storage pavement [3,9].

Reflective pavements with greater reflectance/albedo than conventional pavements could help to decrease the surface temperature and sensible heat release [10,11]. The conventional pavement materials have a low reflectance: 0.05 for new asphalt (the black tar uniformly covers the aggregate), 0.15 for the aged asphalt (aggregate emerges on the asphalt surface), and 0.25 for concrete [12]. A higher total solar spectral reflectance leads to a reduced pavement temperature. Several studies and application attempts have been proposed and verified its feasibility for cooling effect [9,13,14]. Different ways to improve the albedo of pavement consist of the reflective coatings, reflective aggregates, and reflective fillers used for asphalt or cement.

In 2002, reflective coatings were first applied on pavement in Japan [15,16]. Carnielo et al. [12] chose the photocatalytic cement material to reflect sunlight to obtain the decreased temperature between  $8^{\circ}$ C and  $10^{\circ}$ C and proved to be a cool pavement as an effective way to cool in an urban texture. Synnefa et al. [17] applied five different pavement coatings to cool pavement surface and found that when the reflectance is increased to 0.45, the surface temperature will decrease by  $1.6^{\circ}$ C. The life-cycle assessment has also been conducted to analyze the effect of reflectance on energy consumption [4,18,19]. Li et al. [4] found that reflective coatings have low environmental impact but do not improve the ride quality and the application problem exists in coating's service life. According to the product Life

Cycle Assessment (pLCA) tool from Lawrence Berkeley National Laboratory, it is recommended that cool pavement is an effective way to decrease the primary energy demand (PED) and global warming potential in the use stage. Considering the 50-year pavement life-cycle stages, energy consumption of cool pavement in materials and construction (MAC) stage should be paid more attention [18]. Baral et al. [20] assessed the photocatalytic cool cement pavement in the use phase and showed that this kind of pavement indeed decreases the electricity demand for buildings.

The existing reflective cool pavement coatings show some limitations that impede their extensive application although they are effective in decreasing heat absorption [21]. The key research objectives should include the following three aspects: the optical and temperature properties of reflective cool pavement coatings, the lifetime and ride quality influence of cool pavement coatings, and the thermal influence of cool pavement on adjacent building and pedestrian considering microscale zone. Current research focuses on the first research objective. Solar radiation consists of about 5% ultraviolet (UV) radiation (wavelength  $\lambda < 400$  nm), 46% visible (Vis) radiation (wavelength  $\lambda = 400-700$  nm), and 49% near-infrared (NIR) radiation (wavelength  $\lambda = 700-2500$  nm) (ASTM G173-03). This means that the Vis radiation and NIR radiation account for 95% of the total solar radiation. Thus, making the Vis and NIR reflectance increased is the crucial aim. However, too high reflectance will easily cause glare and aesthetic problems and increase the risk of eye illness, especially for the UV and Vis reflectance. Therefore, the reasonable and optimal design of reflectance in different regions is significant. Consequently, studies on the working principles of spectral reflectance of reflective coatings at the full solar spectrum are essential. Meanwhile, research on colored cool pavement coatings and nanomaterial coatings has attracted increasing attention [17,22,23].

Some studies focused on the influence of total albedo/reflectance on the temperature performance of coatings [24,25]. However, limited research studied the optical properties of reflective pavement coatings at the full solar spectrum and considered the influence of different spectral regions [26,27]. Additionally, lightness is also a vital factor in determining optical properties of reflective coatings [28]. According to the CIE (Commission Internationale de l'Eclairage) LAB system, lightness of materials is described as lightness index (L<sup>\*</sup>) in the laboratory. The lightness index (L<sup>\*</sup>) represents the differences between light color (L<sup>\*</sup> = 100) and dark color (L<sup>\*</sup> = 0), which illustrates the perceived lightness by eyes. Over the past decades, limited studies have been established on the relation between lightness of coatings and its spectral reflectance [16,29].

In summary, the study of reflective cool pavement mainly focused on its application and development of different materials; however, the studies on the reflective mechanism and influenced factors are still at the preliminary stage. To adjust the change of special spectrum in narrow scope to the full spectrum scope, the optical properties of coatings at the full-spectrum scales require more investigations and the variation regularity needs revelation. This chapter focused on developing colored reflective pavement coatings (solvent coating and water-based coating in yellow and red) and investigated the optical properties and temperature properties of the pigments and coatings. The optical properties at full spectrum were examined and the solar reflectance was calculated based on the standard solar spectra. Then, the temperature properties of different colored and particle-sized pigments and coatings were investigated. Finally, the variation regularity of reflectance, lightness index, and surface temperature were analyzed and summarized by statistical analysis.

# 7.2 Coating performance evaluation method

The characteristics of pigments and coatings were classified into two categories: the optical properties (spectral reflectance, lightness) and the thermal properties (temperature), which were measured in this study [27].

The spectral reflectance of the coating was measured, following ASTM E903-96. The spectral reflectance measurement device used was a UV-Vis-NIR spectrophotometer (Perkin Elmer Lambda 950, with an accuracy of  $\pm 0.08$  nm) equipped with an integrating sphere (150 mm diameter). The measuring interval was set as 5 nm [30]. The average reflectance value was obtained after three records for each sample. The polytetrafluoroethylene was chosen as the reference and the reflectance of it is almost 100. The solar reflectance was computed by integrating the measured spectral data weighted with standard solar spectral irradiance for air mass 1.5 (AM1.5) as shown in Fig. 7.1 (see standards ASTM E903-96 and ASTM G159-98). Additionally, the solar reflectance values of the



Figure 7.1 The standard solar spectral irradiance for air mass 1.5 (AM1.5).
UV radiation ( $R_{uv}$ , 200–400 nm), Vis radiation ( $R_{visi}$ , 400–700 nm), short-wave NIR radiation ( $R_{sw-nir}$ , 700–1100 nm), and long-wave NIR radiation ( $R_{lw-nir}$ , 1100–2500 nm) were calculated. The lightness of the coatings was measured using a gray-scale image processing to obtain their lightness index (L\*) according to the CIE LAB (Commission Internationale de l'Eclairage).

Infrared thermography was used to investigate the temperature distribution of the samples and to depict the differences in their temperature properties. The device used was FLIR E6 thermal imaging infrared camera. The infrared imaging quality was  $160 \times 120$  pixels, the thermal sensitivity was  $0.06^{\circ}$ C and the spatial resolution of it is 5.12 mrad. To compare the temperature properties of coating samples at the same time, the Vis and IR images were taken during peak surface temperature time on a hot summer day (July 2, 2018). These samples are put on the same slab on the top floor without any shelter to guarantee that these samples receive the same radiation. The height of the device was 1 m above samples, the real-time temperature was  $30^{\circ}$ C, the relative humidity was 50%, the wind speed was around 1.6 m/s, the atmospheric pressure was 1001 hPa, and the emissivity was set as 0.95.

As for the correlation analysis and statistical analysis method, this paper [27] selected the linear regression and multiple regression to depict the correlation relationships. IBM SPSS Statistics 24 was used for statistical analysis. Analysis of variance (ANOVA) test, *t*-test, and collinearity diagnostics were conducted for the statistical analysis.

### 7.3 Optical properties

### 7.3.1 Optical properties of pigment powders

Different inorganic fillers have different crystal structures and other features. However, the optical properties of inorganic fillers have not attracted enough attention although the chemical properties of the optical properties of powders have been investigated extensively in the previous studies [31]. As a critical note, the optical properties of pigments are of the most important factors of its color and reflectance.

Before discussing the optical properties of pigment, the first important thing that should be made is to distinguish between the specular reflectance and diffuse reflectance. Specular reflection is important for optically smooth surfaces. The direction of incident light and the reflected light is the same in specular reflection [32]. When the particle is not the complete spherical and homogeneous particles, the diffuse reflectance is the way of reflecting sunlight. As shown in Fig. 7.2, the particle size and the grain boundaries are essential factors in diffuse reflectance. In the current study [27], the samples that were described are not optically smooth and possess surface roughness. Hence, the contribution of specular reflection to the total reflected light is insignificant and only diffusely reflected light requires to be considered.



Figure 7.2 Schematic diagram of backscattered reflectance for transmitting particles.



Figure 7.3 The reflectance spectra of nano-sized and micron-sized TiO<sub>2</sub> and iron oxide red.

Fillers and pigments in nano-size have increased bandgap [31] and greater fluorescence intensity [33]. Fig. 7.3 shows the spectral reflectance and computed reflectance of two pigments in white and red with different particle sizes. Evidently, the reflectance increases with the decrease in the mean particle size. Next, this experimental observation will be explained and discussed in detail below.

Different theories illustrate the interaction of light with samples, which include the microscopical or macroscopical model. On the one hand, Mie theory [34] is a microscopical model that has been proposed based on the principles of electromagnetism and applies well to spherical pigment particles separated by distances large compared to the wavelength of light. On the other hand, the simplest and most popular continuum macroscopical model is the two-flux theory, which was introduced by Schuster in 1905 and popularized by Kubelka and Munk [35]. Eq. (7.1) explains the proposed model.

$$\frac{(1-R_{\infty})^2}{2R_{\infty}} = F(R) = \frac{K}{S}$$
(7.1)

Where *K* is the absorption coefficient, *S* is the scattering coefficient, and  $R_{\infty}$  is the reflectance of the sample at infinite thickness. *F*(*R*) is called the Kubelka and Munk (KM) function, also termed the remission function.

The reflectance of the sample depends on the ratio of K to S and not on the absolute values of K and S. There exists a linear relation between scattering coefficient S and the inverse of average particle size 1/d, that is, when the particle size decreases, S increases. Finally, this results in an increase in reflectance. Furthermore, nano-sized materials have more grain boundaries of a particle consisting of many crystallite faces to reflect incident light [36]. Thus, this principle proves the better reflectance effect of nano-sized pigments.

It is evident from Fig. 7.3 that the particle size influences the reflectance of pigment; moreover, its pigment color has a significant effect. The optical properties of different colored pigments in black, white, yellow, red, blue, and green were examined. Fig. 7.4 and Table 7.1 illustrate the influence of color influences on the reflectance spectrum. The reason for different pigments that has different colors is their different reflectance spectrums; however, different spectrums may present the same color. It can be apparently observed that the pigment that has the reflectance peak



**Figure 7.4** The reflectance spectra (200–2500 nm, which contains full wavelength of ultraviolet light, visible light, and near-infrared light) of different pigment powders.

Pigment type	R <sub>total</sub> (%)	<b>R</b> <sub>uv</sub> (%)	$R_{\rm visi}$ (%)	$R_{ m nir}$ (%)
Iron oxide blue	37.59	10.69	23.09	51.86
Iron oxide green	43.53	14.75	20.33	65.35
Iron oxide gray	24.87	13.72	25.15	25.66
Iron oxide red	34.89	13.14	13.57	54.72
Iron oxide yellow	61.98	13.56	35.97	88.52
Fe <sub>3</sub> O <sub>4</sub> (brown)	11.25	12.72	11.40	10.99
Ni <sub>2</sub> O <sub>3</sub> (black)	10.48	10.92	10.03	10.83
TiO <sub>2</sub> (white)	86.64	25.52	83.65	94.71

 Table 7.1 The weighted reflectance of different pigment powders in four wavelength regions.

*Notes*:  $R_{total}$  is the total solar reflectance (200–2500 nm),  $R_{uv}$  is the ultraviolet reflectance (200–400 nm),  $R_{visi}$  is the visible reflectance (400–700 nm), and  $R_{nir}$  is the near-infrared reflectance (700–2500 nm).  $R_{total}$ ,  $R_{uv}$ ,  $R_{visi}$ , and  $R_{nir}$  are the computed weighted reflectances in different radiation regions according to ASTM G159-98 and ASTM E903-96.

at the red light zone (640–780 nm) appears red. Similarly, the reflectance peak of powders at 470–550, 530–610, and 505–525 nm indexes appears blue, yellow, and green, respectively, which can also be indicated by the definition of reflectance as shown in Eq. (7.2); spectral reflectance (*R*) is the ratio of reflected light flux  $\Phi p$  ( $\lambda$ ) and incident flux  $\Phi i(\lambda)$ .

$$R = \frac{\Phi p(\lambda)}{\Phi i(\lambda)}$$
(7.2)

When the reflected light flux  $\Phi p$  (640–780 nm) becomes larger, the coating surface will appear more red. For black coating, it will absorb all the incident light so that the reflectance of it does not show any peak and the spectral reflectance is very low.

Reflectance in Vis region (400-700 nm) is relative to the color of pigments and it is determined by color. However, the reflectance in the NIR region (700-2500 nm) is not related to the color of pigments, especially in the long-wave NIR region (1100-2500 nm). From the reflectance results, pigments in yellow, green, and red are more recommended as the reflective pigment. When applying black pigment in reflective coatings, it is better to control its covering rate and combine it with other light colors.

The medium chrome yellow was selected separately with the pigment  $TiO_2$  in white and the iron oxide yellow that has the highest reflectance. As indicated in Fig. 7.5, the total solar reflectance ( $R_{total}$ ) of medium chrome yellow reaches 64.41%, higher than that of the iron oxide yellow. The medium chrome yellow has



Figure 7.5 The reflectance spectra of medium chrome yellow, TiO<sub>2</sub>, and iron oxide yellow.

a lower Vis reflectance  $(R_{visi})$  but the NIR reflectance  $(R_{nir})$  is reached to 95%, even a bit higher than white TiO<sub>2</sub> pigment. Furthermore, the medium chrome yellow is a type of medium color pigment ( $40 < L^* < 80$ ) while the iron oxide yellow is a pigment in light color ( $L^* > 80$ ). That is, the medium chrome yellow is darker than iron oxide yellow but still has a higher NIR reflectance.

### 7.3.2 Optical properties of coatings

As mentioned in Section 7.1, various functional pigments have different optical properties. Non-white (red and yellow) reflective coating samples were prepared and the spectra of these samples were studied by spectrophotometric methods. As shown in Fig. 7.6 and Table 7.2, the solar reflectance for all these coatings was between 24.7% and 40.4%. Furthermore, all these coatings have high NIR reflectance, which was in the range of 39.8%-55.8%. For red reflective coating, the increased content of TiO<sub>2</sub> and iron red oxide can both make the reflectance increase.

First, as the red coating, all nominated samples have peak values around 700 nm to appear red surface. This reflectance spectrum line can be called as "red edge." Second, the reflectance spectrum of sample #4 is obviously different from others. Although it was darker (L\* < 40) and the reflectance in the Vis region was low, the reflectance in long-wave NIR region was higher than that for the rest of all the samples. The reason is that  $Fe_2O_3$  in nano-size was applied to sample #4 as the functional pigment. The specific surface area of nano-filler is larger than that of micro-filler so that when the mass fraction is the same, it needs nanofillers in a larger volume. The hiding power will be stronger. In this way, using nanoscale pigment is a potential way to prepare the "cool dark" coating. Third, when comparing samples



**Figure 7.6** The reflectance spectra and weighted reflectance of red coating samples. *ER*, Epoxy resin; *IOR*, iron oxide red; *AD*, additive.

Coating sample number	R <sub>total</sub> (%)	<b>R</b> <sub>uv</sub> (%)	R <sub>visi</sub> (%)	<b>R</b> <sub>nir</sub> (%)	R <sub>sw-nir</sub> (%)	<b>R</b> <sub>lw-nir</sub> (%)	<b>L</b> *	a*	b*
#1 & #2	31.8	7.6	13.7	49.1	48.7	50.2	57	36	22
#3	36.5	7.6	17.0	55.5	54.4	57.8	70	38	22
#4	24.7	7.6	8.5	39.8	29.6	58.0	43	33	21
#5	40.4	8.5	22.3	58.6	61.7	53.4	73	35	20
#6	31.6	7.6	12.5	49.9	49.8	50.4	50	40	22
#7	62.8	10.9	62.6	67.7	75.5	54.0	100	0	0

**Table 7.2** The solar and spectral reflectance and the color  $(L^*/a^*/b^*)$  of red coating samples containing different weight content of pigments.

#3 and #5, the lightness of sample #5 is higher than that of sample #3 by 4% with the Vis reflectance higher by 22%, whereas the long-wave NIR reflectance was higher by 8%. It indicates that the NIR reflectance is not determined by its color lightness but the Vis reflectance is not. The relationship is discussed further in Fig. 7.7.

The spectral reflectance curves and the computed spectral reflectance for waterbased reflective coating in yellow are shown in Fig. 7.8 and Table 7.3. The total solar reflectance reached about 60%, which was higher than the red coating as



Figure 7.7 The relation between brightness index and visible radiation reflectance.



Figure 7.8 Reflectance spectra of yellow water-based coating samples. The solvent-based white coating #7 was chosen as the comparison sample.

shown in Fig. 7.4. At the same time, the UV reflectance was very low (less than 7%). In contrast, the Vis reflectance was around 40% while the NIR reflectance extended up to 80%-90%. When the mass fraction of medium chrome yellow increased from 16.7% to 50%, the total solar reflectance increased by 5%, the UV

Coating type	R <sub>total</sub> (%)	<b>R</b> <sub>uv</sub> (%)	R <sub>visi</sub> (%)	<b>R</b> <sub>nir</sub> (%)	<b>L</b> *	a*	b*
0% Medium chrome yellow	72.9	7.0	73.0	78.8	100	0	0
TiO <sub>2</sub> : medium chrome yellow = 5:1	59.4	6.6	46.3	75.5	89	6	40
TiO <sub>2</sub> : medium chrome yellow = 5:2	59.6	6.7	41.2	80.2	85	11	57
TiO <sub>2</sub> : medium chrome yellow = 1:1	62.5	6.9	40.8	86.2	81	13	54
Solvent-based white coating	62.8	10.9	62.6	67.7	91	- 4	- 5

**Table 7.3** The solar and spectral reflectance and the color  $(L^*/a^*/b^*)$  of yellow waterbased coating samples containing different weight content of pigments.

Notes: The solvent-based white coating #7 was chosen as the comparison sample.

reflectance almost remained unchanged, the lightness (L\*) decreased by 9%, the Vis reflectance decreased by 12%, and the NIR reflectance increased by 12%. This means that doped chrome is a feasible way to increase the solar reflectance, especially the NIR reflectance without increasing the perceived reflectance. This also demonstrates that NIR reflectance does not have effect on the coating's color lightness but the Vis region is determined by coating's color lightness. Thus, this relationship need more research.

The relationship between spectral reflectance and the brightness index(L\*) was also investigated. As mentioned in Section 7.1, limited researchers studied the relationship between reflectance and brightness. However, this is a crucial issue for pavement engineering to avoid glare problems and guarantee the drive safety [2,3]. In Fig. 7.7, the x-axis stands for the brightness of reflective coatings from dark to bright, and the y-axis stands for the Vis reflectance. It is observed that the first four test values show a linear trend, and, in contrast, the latter values show exponential growth. The variance rules change at which level is the key question. Therefore, the authors found the critical point in steps as follows. The maximum value of brightness index is 100, thus the increased rate reaches its limit value. The tangent at the point  $L^* = 100$  and the linear fitting line were the selected samples to find the intersection point as the turning point, and finally, the threshold level was at the point  $L^* = 81$ . This means that when the brightness index (L<sup>\*</sup>) is greater than 81, the relationship between Vis reflectance and the L\* value is not linear but show an exponential variance. Coincidentally, when the brightness is greater than 80, it is defined as the light color (JG/T 235-2014). Thus, it is better to control the Vis reflectance under 50% to control the brightness index under 80 to obtain the medium and dark colors. This supplements the relationship between reflectance and brightness at the



**Figure 7.9** Infrared (A) and visible (B) images of the 10 coating samples (#1 and #2 are the same coating).

whole brightness range. Meanwhile, it is valuable for choosing and designing the Vis reflectance range to obtain the coatings in medium and dark colors.

### 7.4 Temperature properties of the nominated coatings

This section describes the temperature of reflective coatings and its correlation will be discussed in the next section. The coatings were put on a platform without any obstruction shielding to be measured by the infrared thermography. Fig. 7.9 illustrates the Vis and IR images shot at the height of 1 m of these coatings.

In the IR image, each millimeter contains the information of a frame. Four hundred frames in each sample were chosen as the research range and the box plot of coatings temperature distribution as shown in Fig. 7.10. The values of the box are from different locations of the same sample. It demonstrates the quartile, the median, the mean value, and the interquartile range of the temperature testing values. Samples #9–#11 are water-based reflective coatings in yellow and samples #6–#11 are solvent coatings in red. It is obvious that the temperature distributions are divided into two groups, which indicates that the temperature of samples #1–#6 was much higher than that of samples #7–#11. Moreover, for the relatively, the boxes of sample #8–#11 was narrower than that of samples #1–#6. This describes that the distribution of water-based reflective coatings is relatively more uniform than that of the solvent-based reflective coatings are more complex, and it is more difficult to achieve the dispersion uniformity.

### 7.5 Correlation between the optical and temperature properties of reflective coatings

On the basis of the above optical properties and temperature evaluation results, the correlation between the two characteristics was explored. The objective is to build



Figure 7.10 The box plot of temperature distribution of coating samples #1 through #11.

models to describe the correlation relationship between temperature and reflectance in different solar spectral regions. The independent variables are total solar reflectance, UV reflectance, Vis reflectance, and NIR reflectance. The temperature was set as the dependent variable. The temperature test was conducted under the following conditions: real-time temperature, 30°C; relative humidity, 50%; wind speed, 1.6 m/s; atmospheric pressure, 1001 hPa. The conceptual model was presented in Eq. (7.3). This model can be applied for solvent-based and water-based reflective coatings under the above test conditions. The linear fitting and multiple linear fitting results are shown in Fig. 7.11. The statistical and ANOVA test results are presented in Table 7.4. It can be seen from Table 7.4 that  $R^2$  is close to 1 and P value is much lower than .05. This indicates that the regression result fits well and there indeed exists a linear regression between temperature and total solar reflectance, Vis reflectance, and NIR reflectance. The fitting line, 95% confidence interval, and 95% prediction interval are shown in Fig. 7.11.

Temperature =  $f(R_{\text{total}})$ ; Temperature =  $f(R_{\text{uv}})$ ; Temperature =  $f(R_{\text{visi}})$ ; Temperature =  $f(R_{\text{nin}})$ . (7.3)

Except for UV reflectance, which does not show any variation regularity with temperature, the reflectances in other spectral regions all have a linear correlation with temperature. The relationship in simplest model (linear trend) with  $R^2$  ranges from 0.82 to 0.93, and the best model was between the total solar reflectance and



**Figure 7.11** The scattered plot and linear fitting results of (A)  $R_{\text{total}}$ , (B)  $R_{\text{uv}}$ , (C)  $R_{\text{visi}}$ , and (D)  $R_{\text{nir}}$  and temperature of reflective coatings.

temperature. Therefore, these results displayed that an increase in reflectance by 10% can reduce the high surface temperature in summer by approximately  $2.4^{\circ}$ C for reflective coatings. In the premise of 95% confidence interval, the ANOVA test (*F*-test) was conducted and *P* values are shown in Table 7.4. It demonstrates that the regression equations pass the test and the fitting results are reliable.

Then the multiple regression was expected to be conducted to integrate reflectances in different regions and seek for the relation between them and temperature. When choosing the independent variable, the Vis reflectance, the short-wave NIR reflectance ( $R_{sw-nir}$ ), and the long-wave NIR reflectance ( $R_{lw-nir}$ ) were considered individually. The model concept is shown in Eq. (7.4).

$$Temperature = f(R_{visi}, R_{sw-nir}, R_{lw-nir})$$
(7.4)

It is worth noting that all the independent variables are not the significance factors. Thus, trying to obtain the significant variable is the first step in this section; therefore, the *t*-test was conducted first to exclude the insignificant parameter. The linear regression and *t*-test results are presented in Table 7.5. The significance values in the sixth column show that  $R_{\text{lw-nir}}$  is the insignificant variable (Sig > 0.05),

ANOVA test								
Model	$R^2$	Adjusted R <sup>2</sup>	Quadratic sum	DF	Mean square	F	Significance	
Regression	0.966	0.956	135.121	2	67.561	99.293	0.000007	
Residual error			4.763	7	0.68	-	-	
Total	]		139.884	9	-	-	-	
	<i>t</i> -Test							
Model	Nonstandardized coefficient		Standardized coefficient	t	t Significance		Collinear statistics	
	В	Standard error	$\beta$			Tolerance	VIF	
Constant	64.55	1.063	-	60.748	0	-	-	
R <sub>visi</sub>	- 0.107	0.023	- 0.565	- 4.677	0.002	0.333	3.003	
R <sub>nir</sub>	- 0.082	0.021	- 0.466	- 3.852	0.006	0.333	3.003	

Table 7.4	Significance	test result	of $R_{\rm visi}$ ,	$R_{\rm sw-nir}$ ,	and temperature.	

Model	Non-standardized coefficient		Standardized coefficient	Т	Sig.	Collinear statistics	
	В	Standard error	Beta			Tolerance	VIF
Constant	62.780	2.093	-	30.000	0.000	-	_
R <sub>visi</sub>	- 0.126	0.030	- 0.663	- 4.235	0.005	0.200	5.006
R <sub>sw-nir</sub>	- 0.077	0.022	- 0.440	- 3.557	0.012	0.319	3.136
R <sub>lw-nir</sub>	0.048	0.048	0.104	0.983	0.364	0.436	2.292

**Table 7.5** The linear regression and statistical results of the different examined parameters.

although the collinear statistics indicate that the variables do not have the multicollinearity (tolerance > 0.1 and variance inflation factor, VIF < 10). Therefore, the conceptual model was simplified in Eq. (7.5).

$$Temperature = f(R_{visi}, R_{sw-nir})$$
(7.5)

After the multiple regression analysis, the model was developed as shown in Eq. (7.6).

Temperature = 
$$64.6 - 0.082 R_{\text{visi}} - 0.107 R_{\text{sw-nir}} (R^2 = 0.96)$$
 (7.6)

Table 7.4 shows the significance analysis results after excluding  $R_{lw-nir}$  and it is the statistical description of the proposed model. According to  $R^2$ , the independent variables ( $R_{visi}$  and  $R_{sw-nir}$ ) can explain 95.6% variance of dependent variable (temperature). The value of  $R^2$  close to 1 means that the goodness of fit is significant. When compared to the single variable model of  $R_{total}$  and temperature in Fig. 7.11, the multiple regression displayed the best prediction. In the ANOVA test, the number of variables (k) is 2 and the number of samples (n) is 10, so that the  $F_a$  (k, n - k-1) =  $F_a$  (2,7) = 4.73. From the ANOVA testing results, F value (99.293) is larger than 4.73 and the significance is very smaller than .05. Hence, this test indicated that the proposed model is valuable and it can be applied for prediction.

In addition, the *t*-test was conducted and the results are presented in Table 7.4, which shows the statistical description of coefficients. The significance is very smaller than .05, which means that all the coefficients are significant and the fitting results are meaningful. Furthermore, the multiple collinearity test was conducted. The VIF and the tolerance show that these two independent variables do not show the multicollinearity (VIF < 10 and tolerance > 0.1).

In this section, the multiple linear regression between reflectance and temperature was rebuilt. Multiple influence model of Vis reflectance and short-wave NIR reflectance is more effective than the single regression model of total solar reflectance and temperature. In addition, the long-wave NIR reflectance does not have a significant impact on temperature of reflective coatings. Thus, when optimal designing and testing the optical properties of reflective coatings materials to evaluate its thermal effect, it is proper to just consider the reflectance of 400–1100 nm. When developing coatings with higher NIR reflectance, it is better to increase the short-wave NIR radiation reflectance between 700 and 1100 nm. Nevertheless, this does not mean that UV reflectance and long-wave NIR reflectance are not important; moreover, the investigations should be conducted to verify their impact on the environment and humans in the future.

### 7.6 Conclusions and future trends

The research results can be a reference for designing reflective cool pavement coatings and evaluating the optical and temperature properties of them. The results obtained could provide insights and references for optimizing the design of reflective cool pavement coating and developing the metameric matching colored coatings without glare and aesthetic problems to mitigate the UHI effect. Color lightness is the dominant factor for spectral reflectance in the Vis radiation region; however, it does not influence the spectral reflectance in the NIR light region. Reflective pigment with nanoparticle size is an effective technique to increase coating's NIR reflectance. This implies that it is possible to develop the metameric matching cool pavement coatings.

- Coatings-doped chrome is a feasible way to increase the solar reflectance (59% ~ 63%), especially the NIR reflectance (75% ~ 86%) without increasing the perceived reflectance (40% ~ 46%). That is, the reflectance enhancement does not make the lightness of coatings unfavorable to glazing, and the lightness index (L\*) is 20% lower than that of white coatings. However, due to the complex physical and chemical components of fillers and pigments, they may contain several elements harmful to the environment around the pavement field along with the rainfall runoff. More research should be conducted to investigate the biotoxicity and other ecological properties of this type of coating.
- 2. Higher lightness means higher Vis light reflectance. When the coating is in medium and low lightness, the variation is linear. When the coating is in high lightness, the variation conforms to exponential growth and the threshold level in this limited research is L\* = 81. To achieve the goal of visual comfort, the relationship between the optical characterization of coatings and the Vis response of drivers and pedestrians is important. Further research should focus on this problem and provide a proper solution for glare problems.
- **3.** The reflectance between 400 and 1100 nm is the main significant factor to influence the temperature properties of reflective coatings. The long-wave NIR light region could be excluded in reflectance optimal design when considering the cooling effect. Furthermore, except the thermal comfort, the radiation in different regions may influence the environment around the road. Thus, other effects on humans and surrounding buildings of this radiation region should be studied in future work.
- 4. Under the complicated road circumstances and the heavy load of vehicles, the coatings for pavement could have better durability performance, especially the skid resistance and

antiabrasion performance. The surface roughness and texture depth of pavement changed on account of coating. The comprehensive performance of reflective coatings applied for pavement should be evaluated and investigated further to guarantee the driving safety in practical applications. There are still no standards for cool pavement coatings' performance. Thus, investigating into the pavement performance of reflective coatings is the key step to introduce the coatings for pavement into engineering applications.

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# Influence of aging on the performance of cool coatings



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### 8.1 Introduction

During the last decades, the urban heat island (UHI) phenomenon, referring to the increased ambient air temperatures ( $T_{air}$ ) of the urban areas, compared to values reported in the peripheral zones [1,2], has become one of the basic research topics in the area of urban climatology [3–6]. Considering the severe negative impact of the urban warming not only on the human indoor and outdoor thermal comfort but also on the buildings' energy performance [7–10], researchers have paid great scientific attention not only on the key parameters governing the energetic basis of the UHI phenomenon but also on the establishment of suitable mitigation and adaption strategies [5,11,12]. In fact, one of the main, human-related parameters, contributing to the urban warming, refers to the construction materials, applied both on the urban ground and building envelope surfaces; due to their thermophysical and optical properties, they absorb and store large quantities of solar radiation, part of which is then re-emitted inside the urban districts, increasing the temperature of the ambient air [12,13].

To address this issue and aiming at the improvement of the outdoor thermal environment and the resilience of the urban areas, a great scientific interest has been paid toward the creation of cool coatings that can be applied on the urban ground and building envelope surfaces. [14–17]. According to Qin [18] and Pisello [15], the key benefit of the cool coatings relies on the increased solar reflectance (i.e., increased albedo) and the high infrared emittance, compared to the conventional coating materials. The term "solar reflectance" or "albedo," describes the total reflectance of a specific surface, considering the hemispherical reflection of radiation, integrated over the solar spectrum, while both specular and diffuse reflections are included; the term "infrared emittance" designates the ability of the surface to emit energy away from itself compared with a blackbody operating at the same temperature [19]. Consequently, the application of cool coatings on the urban ground surfaces would lead to lower amounts of absorbed and stored solar radiation and as a result to lower temperatures of the urban surfaces  $(T_{surf})$ ; the convection of heat from pavement to the surrounding air will be thus reduced, leading also to a decrease in the ambient air temperature [17,20]. When cool coatings are applied to the buildings' envelope, the reduction of the building components surface temperatures would also decrease the heat transfer toward the interior of the building, leading to lower cooling energy needs and more favorable indoor thermal comfort conditions [21,22].

When it comes to the cool coatings' applications on the ground surfaces, their important potential on regulating the urban warming arises from the large percentage of the total land cover that asphalt and concrete pavements account for. In fact, several examples of modern cities suggest that the pavements, including asphalt roads, circulation areas around buildings, parking areas, and so on, represent almost 30%-40% of the total surface cover [23]. In this context and considering the significant mitigation potential that arises, various techniques have been proposed to achieve a higher solar reflectance of coatings, for ground surfaces applications including:

- The addition of light color aggregates that can lead to an increase in the surface's albedo by almost 30% [20,24]
- The use of thin, light-colored toppings that are highly reflective in the visible wavelengths [25,26]. Various materials are commercially available with reported albedo values ranging between 0.30 and 0.55 for the asphalt pavements [24,27]; higher albedo values, reaching even 0.77 have been reported for cool concrete pavements applications [24].
- The use of colored, nonwhite pigments on coatings so as to increase the solar reflectance in the near-infrared wavelengths [17,28]. According to the results of a previous experimental campaign, testing 10 prototype cool colored coatings in comparison to conventionally pigmented coatings, a rise in the solar reflectance of the color thin-layer asphalt samples was reported, reaching 0.27 for the red and green samples and 0.55 for the offwhite samples [29].
- The creation of thermochromic coatings, which thermally respond to the conditions of the outside environment, changing reversibly their color as the outside  $T_{air}$  rises [30]; yet, their price is considerably high and thus, the respective applications are still rather limited.

A detailed description of the different techniques that aim to increase the asphalt and concrete pavements' albedo is provided by Tran et al. [31]. To date, several experimental campaigns and simulation studies have been conducted to assess the effectiveness of cool coatings in improving the outdoor thermal environment and the cooling energy demand in urban buildings. Surface temperature measurements in Athens suggested that the use of a cool, yellow asphalt with near-infrared pigments can reduce the surface temperature by 7.5°C compared to the conventional dark material, whereas similar magnitudes have also been reported by Carnielo and Zinzi [32] and Lontorfos et al. [33]. At a district scale, Taleghani et al. [34] suggested that an increase of the asphalt and concrete pavements' albedo by 0.30 in an urban area in Los Angeles would lead to an important peak  $T_{air}$  reduction at human level by 2.0 C, while similar magnitudes of maximum  $T_{air}$  cooling were also reported by Battista et al. [35]. Other existing studies have also demonstrated that the application of cool coatings on roofs may lead to a considerable reduction in the peak cooling energy demand in air-conditioned buildings, reaching even 27% [27,36]. A review of previous studies, assessing the improvement of the urban microclimate and the reduction of the cooling energy demand due to cool coatings' application is provided in Refs. [37] and [38].

However, given that the albedo value is determined by the optical properties of the outer surface layer of the materials, significant changes may occur over time [39], thus compromising the durability of the material. As highlighted by Li et al. [40], the life cycle of cool coatings' applications on sidewalks, roads, and highways may be rather limited due to the environmental stresses and the increased trafficking, considerably affecting the durability of the coatings in terms of the solar reflectance value and reducing their effectiveness as a mitigation strategy toward the improvement of the urban microclimate [41-43]. It can be thus claimed that the accurate evaluation of the cool coatings' thermal performance should be conducted for the whole life cycle of the material, while the albedo degradation because of weathering and aging is also considered. The following sections will discuss the parameters contributing to the aging of cool coatings and the methods to quantify the albedo degradation, while emphasis is also paid on their effect on improving the outdoor thermal environment and the buildings' energy performance, when the aged albedo values rather than the design ones are accounted for.

# 8.2 Investigating the aging and weathering of cool coatings

As previously mentioned, the high solar reflectance of cool coatings may be lowered over time due to weathering and aging phenomena, compromising the durability of the respective materials. To date, the available data in terms of the ability of the materials to resist in time are rather limited; experimental measurements of albedo changes due to aging and weathering are more often available either for cool roof coatings [41,44–47] or samples of cool paving materials, placed in specially modulated areas or platforms [17,30,48,49] rather than for real applications of cool concrete pavements and asphalts [33,42] mainly due to the difficulty of circulation and traffic in the latter case. In the next sections, the parameters that contribute to the aging of cool coatings are presented along with the existing evaluation and the proposed solutions for the initial albedo values' restauration.

### 8.2.1 Parameters contributing to the aging of cool coatings and evaluation approaches

The durability and the high solar reflectance values of cool coatings are considerably affected by weather and time. Especially for the cool roofing coatings, the key degradation parameters comprise of soiling, precipitation, ultraviolet radiation, moisture penetration, wind forces, and biomass accumulation [50-52], whereas another critical parameter is the microbial growth, considerably enhanced by the deposition of atmospheric carbonaceous particles [53]. A detailed overview of the parameters contributing to the weathering of cool roofing coatings has been provided by Berdahl et al. [54] In the case of cool coatings for asphalt road applications, the albedo degradation is attributed not only to the abovementioned parameters but also to traffic-related factors such as cracking, peeling of the coating, motor vehicles emissions, and rubber deposition by the vehicles' tires [33,40,42]. Similarly, in the case of the cool concrete pavements that are mainly applied at sidewalks, the deposition of dust, soil, and atmospheric pollutants, along with the exposure to the natural weather conditions and the dirt accumulation due to the pedestrians' circulation are the dominant parameters contributing to the decrease of solar reflectivity with time [55]. Interestingly, the existing evidence suggests that most of the albedo loss occurs during the first year and actually during the first few months of exposure, while the annual decline of albedo in the subsequent years is rather negligible [17,33,56].

To investigate and assess the aging phenomena and the respective loss of solar reflectance of cool coatings, the most widely applied methods include:

- Field measurements of the coatings' solar reflectance, using a pyranometer, according to the standard ASTM E1918 (*Test Method for Measuring Solar Reflectance of Horizontal and Low-Sloped Surfaces in the Field*) [57].
- Laboratory measurements of the spectral absorptance and reflectance of the cool coatings using spectrophotometers according to the standard ASTM E903 (*Standard Test Method for Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres*) [58].
- Field or laboratory measurements of the cool coatings' solar reflectance using a portable solar reflectometer according to the standard ASTM C1549 (*Standard Test Method for Determination of Solar Reflectance Near Ambient Temperature Using a Portable Solar Reflectometer*) [59].
- Definition of the solar reflectance index (SRI) of the cool surface, representing the relative temperature of the cool surfaces with respect to black and white reference surface temperatures. The approach for the SRI definition is described in the Standard ASTM E1980 (*Standard Practice for Calculating Solar Reflectance Index of Horizontal and Low-Sloped Opaque Surfaces*) [60].
- Laboratory measurements of the albedo of cool coatings' after being exposed to accelerated weathering and soiling procedure, mimicking the real environmental conditions. The approach is described in the Standards ASTM G151-10 (Standard Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources)
   [61] and ASTM G154-12 (Standard Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Nonmetallic Materials) [62].

A detailed description of all the abovementioned techniques is provided in Ref. [19]. In all cases, records are taken right after their installation and within a period of a few months or years, (depending on the study) and the obtained measurements are compared, in order to evaluate the reduction of the albedo value with time. A summary of relevant studies assessing the reduction of the cool coatings' albedo due to weathering and aging is presented in Table 8.1. Based on the existing evidence, it can be generally said that the albedo degradation is more prominent in the case of cool coatings that are applied on the ground surfaces and tested under real exposure conditions, compared to the roofing materials or samples in modulated platforms. Even if the respective experimental campaigns evaluating the cool pavements' aging are rather limited, their results provide valuable information about the

References	Initial solar reflectance values (albedo right after installation)	Exposure conditions of the cool coating	Applied method for measuring the aging	Results – quantification of aging
[42]	Cool asphalt pavements: a = 0.35 Cool concrete pavements: $a = 0.66$	Outside conditions: vehicles and pedestrians' circulation	Measures of spectral reflectance using a UV/Vis/NIR spectrophotometer (ASTM E903-96)	After 6 months of continuous use: reduction of asphalt solar reflectance (SR) by 48%. Negligible reduction of the concrete pavements' SR
[33]	Cool asphalt pavements: a = 0.26 Cool concrete pavements: $a = 0.47$	Outside conditions: vehicles and pedestrians' circulation	Measures of spectral absorptivity with spectrophotometers (ASTM E903-96)Measures of spectral reflectance with reflectometers (ASTM C1549- 16).	After 12 months of continuous use: reduction of asphalt SR by 42%. Reduction of pavements' SR by 15%
[55]	34 samples of colored concrete pavements: a = 0.35 - 0.67	Outside conditions Only pedestrians' circulation (no vehicular circulation)	Determination of the solar reflectance index (ASTM E1980-11)	After 12 months of exposure: reduction of the light- colored pavements' SRI by 25%
[14]	Cool yellow concrete pavements: $a = 0.60$	Outside conditions Only pedestrians' circulation (no vehicular circulation)	Measures of solar reflectance and solar reflectance index (ASTM G159-91) and accelerated aging tests	After the first months of continuous use: negligible reduction of pavements' SR
[17]	10 cool coatings, with near-infrared reflective pigments: a = 0.27 - 0.63	Outside conditions- Samples placed on a specially modulated platform.	Measures of spectral reflectance using a UV/Vis/NIR spectrophotometer (ASTM E903-96 and ASTM G159-98)	After 3 months of exposure: reduction of samples' SR by 3%-10%

 Table 8.1
 Summary of studies quantifying the albedo changes of cool coatings as a function of time.

(Continued)

Table 8.1 (Continued)

References	Initial solar reflectance values (albedo right after installation)	Exposure conditions of the cool coating	Applied method for measuring the aging	Results – quantification of aging
[48]	White concrete pavement sample: $a = 0.26$	Outside conditions- Ground test sections	Measures of solar reflectivity using dual-pyranometer (ASTM E1918)	After 3 months of exposure: reduction of sample' SR by 10%
[44]	8 specimens of cool acrylic coatings	Outside conditions- Samples placed on a low-sloped roof surface.	Measures of spectral reflectance with a double-beam spectrophotometer (ASTM E903-96)	After 18 months of exposure: Reduction of sample' SR by 7%-27%
[47]	White coatings for roof tiles applications $a = 0.82 - 0.83$	Outside conditions Samples of tiles placed on a horizontal roof	Determination of the solar reflectance index (ASTM E1980-11)	After 1 year of exposure: reduction of the SRI by 8%-12%
[63]	Gray concrete pavement sample: $a = 0.32$	Laboratory tests— accelerated weathering and aging	Measures of solar reflectivity with a solar-spectrum reflectometer (ASTM G154)	After 17 months of exposure: reduction of sample' SR by 15%

albedo changes as a function of time. Experimental results from a large-scale application of cool coatings in Athens suggested that, after 6 months of continuous use and traffic exposure, a cool yellow asphalt would lose almost 50% of its initial albedo value as a consequence of atmospheric pollutants and particles issued by vehicles emissions but also due to dirt and rubber from the vehicles' tires [42]. Similar remarks have been issued from another experimental campaign, measuring the solar reflectance of cool asphalt and concrete pavements implemented in the context of an urban rehabilitation project in Athens [33]. The obtained results indicated an important loss of solar reflectivity after the first year of exposure, reaching 40% and 15% for the bitumen and concrete pavements, respectively. Again, soiling, microbial growth, and rubber deposition are considered the main parameters affecting the solar reflectance values. Finally, the obtained results of another monitoring study, evaluating the albedo changes of various concrete pavements of different colors that occurred within a year of exposure at the pedestrians' circulation, suggested a considerable reduction in the SRI, even up to 25% [55].

### 8.2.2 Methods for the albedo restauration

Considering that the positive effect of cool materials toward the improvement of the outdoor thermal environment and the regulation of the urban warming can be attenuated due to the aging and weathering process, several methods have been reported to recover the initial values of solar reflectivity. Levinson et al. [46] assessed different cleaning methods of cool coatings, applied on roofs such as wiping with dry cloth, rinsing with water, scrubbing with detergent and water, and so on; the analysis of the solar reflectance measurements before and after the cleaning process suggested that the initial high albedo values can generally be recovered, with the wiping method being more effective at removing the black carbon, while water rinsing and detergent use may remove nearly all of the remaining soil, leading to increased albedo values for the cleaned samples. Similarly, Berdhal et al. [41] examined the potential of recovering the high albedo of cool roofing coatings using various techniques and concluded that an abrasive treatment of the surface would lead to similar albedo values to the ones of the unweathered surface. In the same context, Synnefa et al. [17] concluded that the initial high albedo of cool coatings, having near-infrared reflective color pigments, can be restored even up to 93% if the weathered samples are rinsed with water and washed with a sponge; the reduction of the solar reflectance has thus been mainly attributed to the dirt deposition rather than to permanent issues.

Regarding applications of cool coatings on the urban ground surfaces, Kyriakodis et al. [42] and Lontorfos et al. [33] also concluded that the scrubbing of the cool ground pavements with wet solvents would enable the full recovery of the initial value of solar reflectivity, even if a considerable reduction has occurred due to weathering, dust, and microbial growth. Yet, it is important to emphasize that various cleaning methods for the albedo restauration presented before, would be more suitable for the maintenance of cool roofing coatings rather than for cool asphalt and concrete pavements' application due to the large land cover of the latter ones.

## 8.3 Assessing the effect of aging on the thermal performance of cool coatings

As stated in Section 8.1, the use of cool coatings having high albedo values, either on the building envelope surfaces or on the urban ground surfaces, is considered as a strategy toward the regulation of the urban warming during summer and the attenuation of each consequent effects; a higher solar reflectance would lead to lower surface temperatures because higher amounts of solar radiation are reflected than absorbed. In its turn, this would lead to a reduction in the amounts of heat, transferred from the surface to the surrounding environment through convection phenomena compared to conventional paving materials, thus reducing the ambient air temperature [64]. When it comes to building envelope applications, the lower surface temperatures would also lead to a decrease in the amount of heat penetrating the building, thus improving the indoor thermal conditions and the required cooling loads [26].

It becomes however clear that the effect of the cool coatings on regulating both the outdoor thermal environment and the buildings' energy performance may be compromised because of the aging and weathering phenomena and the consequent albedo loss. In other words, a major issue that arises is the thermal performance of the cool materials during their whole life cycle, risking to be rather short, counterbalancing their positive contribution [43]. To date, numerous studies have assessed the durability of the cool coatings and their albedo decrease with time. On the other hand, the performance of the aged, "dirty" coatings with respect to the urban microclimate and buildings' energy needs has been far less evaluated, with the existing evidence providing however significant insight on the cool coatings' effect during their whole life cycle. What is also important to mention is that the respective studies, examining the thermal performance of the weathered coatings, have mainly used simulation means rather than experimental campaigns. This is because the establishment of numerical simulation approaches would allow the comparative assessment of the acquired results (i.e., microclimatic parameters and/or buildings' energy needs) before and after the cool coatings' application, both for the "clean" and the aged scenario, under similar boundary conditions.

### 8.3.1 Aged cool coatings and their effect on urban microclimate

As mentioned before, the potential of cool coatings in improving the urban microclimate of dense urban areas, while considering their optical properties during their whole life cycle, has been rather rarely examined. Indicatively, Kyriakodis et al. [42] performed microclimate simulations to assess the effect of replacing a dark conventional asphalt with a cool yellow one, having an albedo of 0.35, on the reduction of the ground surface and the ambient  $T_{air}$ ; the microclimatic conditions for the aged albedo values obtained after a period exposure have been also simulated (i.e., reduction of 50% of the initial albedo). The analysis, conducted on a hot summer day, showed that the use of a nonaged asphalt led to considerably lower  $T_{\text{surf}}$  values, compared to the conventional dark material with the estimated differences of up to 9.0°C; yet, the respective discrepancy did not exceed 6.0°C, when the aged materials have been accounted for. In parallel, replacing the dark asphalt with a cool one considerably decreased the peak ambient  $T_{\text{air}}$  value at the pedestrians' height by 1.5°C but the mitigation potential was substantially reduced by 50% when the albedo degradation has been considered.

Similar remarks have been made by Lontorfos et al. [33] who numerically assessed the effect of highly reflective pavements on decreasing the peak ambient air temperature during a summer day in a large urban area in Athens, Greece; again, microclimate simulations have been performed both for the nonaged (0.47 and 0.26 for the concrete and bitumen pavements, respectively) and the aged solar reflectance values, after a year of continuous use (0.40 and 0.15 for the concrete and bitumen pavements, correspondingly). Numerical simulations results revealed an important reduction in the maximum  $T_{air}$  values, reaching 1.7°C, when the conventional asphalt and concrete pavements were replaced with the highly reflective ones. Yet, when the aged albedo values have been accounted for, the mitigation potential of the cool coatings was substantially reduced by 25%, compared to the initially estimated values for the nonaged materials.

Aiming at a further analysis of the high albedo coatings' cooling effect during their whole life cycle, the abovementioned experimental results of Lontorfos et al. [33] have been applied for a parametric investigation, examining the influence of aging on the thermal performance of cool coatings in a dense urban area in Thessaloniki, Greece. The selected district is considered representative for the broader center of the city, in terms of the urban space's characteristics (i.e., geometry of the street canyons, surface cover properties, materials, and building height). The buildings of the study area, having a height of 22.0–25.0 m cover 35% of the total ground surface of the study area, whereas the average sky view factor of the district fluctuates between 0.20 and 0.50 (Fig. 8.1). The analysis has been conducted with the ENVI-met microclimate model for a representative summer day in July [65]—July 22, 2016—and the simulation scenarios involve (1) the current conditions, in which ground surfaces are covered by conventional dark asphalt and



**Figure 8.1** (A) Google Earth image of the study area indicating the main street canyons and (B and C) area input file introduced in the ENVI-met model.

concrete pavements with an albedo of 0.12 and 0.4, respectively, (2) the nonaged, cool coatings scenario, corresponding to the replacement of the conventional asphalt and concrete pavements with cool ones, having initial albedo values of 0.40 and 0.70, respectively, and (3) the aged scenario, considering an aged asphalt and concrete pavements' albedo of 0.23 and 0.60, respectively (considering an albedo loss by 40% and 15% for the asphalt and pavements, respectively, according to the reported values of Lontorfos et al. [33]). Regarding emissivity, no changes were considered due to weathering process over a year with the latter hypothesis being based on a previous experimental campaign, estimating the aging of cool colored concrete pavements by reporting changes on albedo and emissivity occurred within a year. The obtained monitoring results showed zero changes in emissivity for the majority of the samples while a maximum modification of 5% was reported for the rest of the samples [55].

The simulated scenarios are summarized in Table 8.2. Furthermore, the study area was modeled using a domain size of  $135 \times 135 \times 20$  grids (i.e., *x*-grids  $\times$  *y*-grids  $\times$  *z*-grids), corresponding to a grid size of  $1.5 \times 1.5 \times 3.0$  m and the meteorological boundary conditions in terms of wind speed, wind direction, hourly values of  $T_{air}$ , and relative humidity (RH) were obtained from the meteo station of the Aristotle University of Thessaloniki.

The absolute values of the estimated  $T_{surf}$ , occurring at 15:00 p.m. for the conventional asphalt and concrete pavements and for the cool coatings, both for the nonaged and the aged albedo values are shown in Fig. 8.2. The obtained simulation results suggest a substantial drop in the ground surface temperatures due to the application of the nonaged, high albedo coatings, with major  $T_{\rm surf}$  modifications of  $5.0^{\circ}C-7.0^{\circ}C$  and  $6.0^{\circ}C-9.0^{\circ}C$  for all the exposed parts of asphalt street and pavements, respectively; yet, for the areas kept in shadow by the building volumes, the  $T_{\rm surf}$  changes are of lower importance and do not exceed 3.0°C. The achieved  $T_{\rm surf}$ cooling potential is attributed to the significantly higher amounts of reflected solar radiation and the consequent lower absorption by asphalt and concrete paving materials; in fact, the increase of the asphalt's albedo by 0.28 resulted in a rise of the reflected shortwave radiation by 75%, compared to the conventional coatings. However, the assumption of an albedo decrease because of aging and weathering has led to lower amounts of reflected solar radiation and thus, to higher amounts of solar radiation henceforward absorbed, contributing to a less prominent reduction of the ground surface temperatures. More precisely, the achieved  $T_{surf}$  drop due to the

	Asphalt	Asphalt	Concrete	Concrete
	road	road	pavement	pavement
	albedo	emissivity	albedo	emissivity
Case A: current conditions	0.12	0.90	0.30	0.90
Case B: cool coatings—non-aged	0.40	0.90	0.70	0.92
Case C: cool coatings—aged	0.24	0.90	0.60	0.92

Table 8.2 Optical properties of the ground surface materials [24,66].



**Figure 8.2** (A) Absolute values of the simulated  $T_{surf}$ , occurring for the current conditions and (B) after the application of nonaged cool coatings, and (C) the aged coatings with lower albedo values at 15:00 p.m.

aged cool coatings has been now estimated close to  $1.0^{\circ}C-3.0^{\circ}C$  and  $3.50^{\circ}C-7.0^{\circ}C$  for the parts of the asphalt and concrete pavements exposed to the sun, revealing an important change in their cooling potential in terms of the ground surface temperatures.

The next step of the analysis focuses on the assessment of the aged cool coatings' effect, on the reduction of the ambient air temperature during the simulation day, and on the quantification of the decrease of their mitigation potential, compared to the nonaged, high albedo coatings. The diurnal evolution of the absolute  $T_{\rm air}$  values, estimated for point 1 (see Fig. 8.1C) in the center of the study area is depicted in Fig. 8.3, whereas the distribution of the respective  $T_{\rm air}$  discrepancies between the base case scenario and the cool coatings, at various distances from the ground level, is shown in Fig. 8.4. The obtained simulation results generally suggest minor  $T_{\rm air}$  modifications due to the cool coatings during night time, whereas their effect is more prominent during daytime; peak discrepancies have been generally found between 9:00 a.m. and 17:00 p.m., a period during which the specific part of the study area (i.e., point 1) is never shaded by building volumes. Regarding the cooling potential of the nonaged coatings, the highest  $T_{air}$ reduction has been mainly noticed at the pedestrian's height (i.e. at 1.5 m from the ground level) in which the increase of the albedo of the asphalt and concrete pavements by 0.28 and 0.40, respectively, led to a peak  $T_{air}$  decrease of around  $0.75^{\circ}$ C. The cooling potential of the high albedo coatings seems to remain rather stable till the height of 4.50 m, but after this level, their effect becomes of lower importance and a loss of performance of around 6%-8% for each additional 3.0 m has been estimated.

Furthermore, assuming an albedo reduction by 40% and 15% for asphalt and pavements, respectively, due to weathering and aging revealed a major decrease in the cooling potential of the coatings, reaching 50% of the initially estimated performance; the peak temperature decrease at 1.5 m did not exceed 0.40°C, while as with the nonaged scenario, the effect of aged coatings on lowering the  $T_{\rm air}$  values decreases as the distance from the ground increases. The loss of performance and the reduced mitigation potential of the aged cool coatings is attributed to the higher amounts of sensible heat, being henceforward released toward the air, as a



Figure 8.3 Diurnal  $T_{air}$  evolution of the  $T_{air}$  for the conventional coatings and for the nonaged and aged coatings, at different heights from the ground level.

consequence of the lower albedo value and the induced higher solar radiation absorption in the ground surface materials; the sensible heat, transferred from the asphalt surface toward the ambient air was reduced by almost 35% for the nonaged cool coatings compared to the base case scenario, while the respective reduction for the aged coatings was considerably lower and close to 12%.

To summarize, the abovementioned findings indicate that the performance of cool coatings, applied on the urban ground surfaces, may substantially vary as a function of their albedo value; the accurate assessment of their potential as a mitigation strategy against the urban warming should thus be conducted for a broader period, while potential changes in their optical properties due to weathering and aging are also accounted for. It is also important to highlight that microclimatic changes and the respective reduction of the ambient  $T_{\rm air}$  due to cool coatings' applications will not only affect the pedestrians' thermal balance but also the buildings' energy performance, given that the latter is strongly influenced by the microclimatic conditions of the area under investigation [67,68]. In the next section of the chapter, emphasis is given on the effect of cool coatings on the buildings' annual energy needs, considering both the nonaged and the aged values of solar reflectance.



Figure 8.4 Differences of the diurnal  $T_{air}$  evolution between the conventional coatings and the nonaged and aged cool coatings.

### 8.3.2 Aged cool coatings and their effect on the buildings' energy performance

To date, the impact of the cool coatings on the buildings' energy performance has been extensively investigated for cool roofing applications [69-73] but still, the existing studies mainly focus on the initial, high albedo values, whereas the aging phenomena are far more rarely examined. In this context, Mastrapostoli et al. [50] have analyzed the aging of cool roofing coatings, applied in two school buildings in Athens, Greece, by measuring the changes in their optical properties with time; in both buildings, an albedo reduction of 23%-25% has occurred within a period of 4 years and its effect on the buildings' energy demand has been then examined by simulation means. It was found that the aging phenomena resulted in higher cooling energy needs by almost 20%, compared to the "clean," cool roof coatings, while the opposite effect has been noted for the heating demand because the reduction of the albedo led to higher amounts of solar radiation, henceforward absorbed by the roof and transmitted toward the indoor spaces of the buildings.

Furthermore, when it comes to the cool coatings' applications on the urban ground surfaces, their impact on the nearby buildings' heating and cooling energy needs has been far rarely evaluated, with the respective studies primarily focusing

on the thermal comfort of the pedestrians [34,74,75]. However, based on the existing knowledge, microclimatic changes and the respective reduction of the ambient  $T_{\rm air}$  due to the use of high albedo pavements are expected to influence not only the human thermal balance but also the energy performance of the buildings located inside the area under investigation [67,76]. To this aim, a recent study has examined by simulation means the effect of the cool pavements' applications-both for the nonaged and the aged albedo values-on the annual heating and cooling energy needs of a generic, noninsulated building unit of 80 m<sup>2</sup>, located in a dense urban area in Thessaloniki, Greece [77]. Microclimate simulations with the ENVI-met model have been conducted for all seasons and (1) for the current conditions in which ground surfaces are covered by conventional asphalt and concrete pavements, (2) for the cool, nonaged coatings, assuming an increase in the asphalt and concrete pavements' albedo by 0.28 and 0.4, respectively, and (3) for the aged scenario, considering an albedo loss by 40% and 15% for the asphalt and pavements, respectively. The obtained results, reflecting the microclimatic conditions that occur in front of a specific first-floor building unit of the study area, before and after the cool coatings' applications, have been then used as an input boundary condition in the EnergyPlus simulation model. The simulation output of the annual heating and cooling energy needs of the investigated building unit, both for the existing conditions and the cool coatings scenarios, is depicted in Fig. 8.5.

It can be generally said that the replacement of conventional asphalt and concrete paving materials with the corresponding high albedo ones only provide minor changes in the building unit's energy performance, even in the case of the nonaged albedo values. On the one hand, the use of high albedo pavements contribute to lower ground surface temperatures and reduced amounts of sensible heat release toward the surrounding air, thus leading to a reduction in the ambient  $T_{air}$  in front of the examined building unit (see Fig. 8.6A). On the other hand, this effect is



**Figure 8.5** Estimated annual heating and cooling energy needs for the base case scenario and due to the cool coatings' applications, considering both the nonaged and the aged albedo values. *Source*: Image, based on the results of S. Tsoka, K. Tsikaloudaki, T. Theodosiou, Coupling a building energy simulation tool with a microclimate model to assess the impact of cool pavements on the building's energy performance. Application in a dense residential area, Sustainability 11 (9) (2019) 2519.



Annual heating and cooling needs of the examined building unit

**Figure 8.6** (A) estimated differences in the monthly  $T_{air}$  values due to the cool pavements' applications, having the design or the aged albedo values and (B) percentage difference on the solar gains of the building unit due to cool coatings application, compared to the base case scenario.

*Source*: Image, based on the results of S. Tsoka, K. Tsikaloudaki, T. Theodosiou, Coupling a building energy simulation tool with a microclimate model to assess the impact of cool pavements on the building's energy performance. Application in a dense residential area, Sustainability 11 (9) (2019) 2519.

counterbalanced by the rise of the solar gains transmitted into the thermal zone of the building unit, due to the higher amounts of the reflected solar radiation toward the surrounding environment (Fig. 8.6B); the EnergyPlus model accounts for both the direct and the reflected radiation from the exterior surfaces during the estimation of the solar radiation transmitted into the examined thermal zone and thus, a moderate rise of solar gains is reported during the heating period, ranging between 1.0% and 11.0% whereas in the cooling period, the thermal zone may receive even up to 20% higher solar gains, compared to the base case scenario.

When the albedo degradation is considered, the estimated reduction in the ambient  $T_{air}$  becomes of lower importance; peak differences are again noticed during summer, without however exceeding 0.2°C. This is due to the higher amounts of sensible heat, being henceforward released toward the air, because of the lower, aged albedo and the consequent higher solar radiation absorption from the ground surface materials, compared to the nonaged scenario. As anticipated, the aged albedo values would also lead to a less prominent rise of the solar energy, transmitted into the thermal zone, compromising again the small reduction of the ambient  $T_{air}$ , estimated in front of the examined building unit.

It can be thus said that, contrary to cool roofing coatings, the applications of cool materials on the urban ground surfaces may have a rather minor impact on the estimated buildings' annual heating and cooling energy needs with their effect risking being even minor for the higher floor levels, at the distance from the ground increases [78]. Given that the nonaged albedo values resulted in minor changes, the

aging phenomena and the albedo degradation did not substantially compromise the initial positive effect of coatings. Yet, it is important to emphasize that even if the previously mentioned remarks provide important insight on the effect of the non-aged and aged cool coatings on the buildings' energy needs, further evidence would be necessary to draw concrete conclusions on their mitigation potential and their impact on the buildings' energy performance.

### 8.4 Synopsis and conclusions

Aiming at the regulation of the urban warming and the improvement of the buildings' energy performance, the use of cool coatings either on the building envelopes or the ground surfaces of the urban areas has gained a lot of scientific attention. Their cooling potential primarily relies on their optical properties and the increased high albedo values of the outer surface layer, which may however substantially change over time due to weathering and aging of the material. Apart from the degradation that occurs due to natural weather conditions, other factors such as dust, increased bacterial loads, biomass growth, and also the deposition of rubber from the tires of vehicles in the case of ground surfaces applications are strongly affecting the values of the solar reflectance of the coatings and thus, their resistance over time. As a result, assuming constant albedo values during the whole life cycle of the cool coatings may be misleading when it comes to the assessment of their effect on the urban thermal environment. In other words, in order to comprehensively evaluate the thermal performance of cool coatings both with regards to the urban microclimate and the buildings' energy performance, it is of great importance to consider the temporal variation of the albedo value.

To date, several studies have measured the albedo changes over time but only a small number has evaluated the effect of aging on the thermal performance of the coatings with respect to the urban microclimate and the buildings' energy demand, with the reported results providing however significant knowledge on their long-term performance. Regarding the effect of aging on the urban microclimate, the reported results generally reveal a significant reduction in the mitigation potential of cool pavements, with a loss of performance even up to 50%, both with regards to the surface and the ambient air temperatures. In terms of the buildings' energy needs, the application of high albedo coatings does not seem to provide any substantial modifications and thus, their aged albedo values do not compromise their effect. Yet, when the cool coatings are applied on building roofs, an albedo loss will considerably change the energy balance of the building, leading to higher cooling energy needs, compared to the nonaged roofing coatings.

### 8.5 Future perspectives

The existing evidence suggests that the optical properties of cool coatings may significantly change with time, compromising their thermal performance. Even if the aging of cool roofing coatings has been extensively studied, this is not the case for the respective ground surface applications, thus revealing a great potential for further research. Conducting experimental campaigns so as to assess the weatherization and the albedo degradation of cool pavements under actual stresses and real vehicular or pedestrians' exposure is a necessary research step so as to further examine methods that would improve their durability. Especially for the applications that concern high-traffic roads, the life cycle of the reflective coatings may be even smaller, indicating the need for the establishment of innovative methods to assure their resistance over time.

Furthermore, the few scientific studies, examining the effect the coatings' aging on the outdoor thermal environment and the nearby buildings' energy performance, mainly concern applications in urban areas of Mediterranean countries; investigating the respective performance of cool pavements' applications in colder climates will provide valuable knowledge so as to obtain a global perspective of the cool coatings' performance under different climatic conditions.

To conclude, high albedo coatings are still an emerging technology toward the mitigation of the urban warming and the attenuation of its implicit effects. Yet, further research and effort are required by the scientific community and the industry to assure the durability and the long-term performance of the coatings.

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### **Conflict of interest**

The author declares no conflict of interest.

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# Part 3

# **Self-healing pavements**

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## Self-healing property and road performance of asphalt binder and asphalt mixture containing urea-formaldehyde microcapsule

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## 9.1 Introduction

Cracking is a common distress in asphalt pavement [1]. Under the combined action of traffic load and environmental factors, microcracks occur in the asphalt pavement. Then, the microcracks, with the continuous expansion and accumulation, expand into macroscopic cracks that are visible to the naked eyes. If not repaired in time, these cracks will affect the driving comfort and service life of pavement [2]. Self-healing asphalt pavement can detect and repair cracks by itself to some extent. In recent years, some techniques, including microwave heating, induction heating, microcapsule and ion polymer, and so on, have been used to heal the cracks in pavement [3–12]. For the healing technique using microcapsules, when microcracks occur and extend into the microcapsules in the matrix material, microcapsules will rupture and the repairing agent will be released to repair microcracks. At present, microcapsule technology has achieved good healing results in the field of polymer and has begun to be applied in the field of road engineering to improve the healing ability of asphalt pavement.

In the 1980s, the United States put forward the concept of self-healing polymers and then carried out more research in this area [13]. In 2001, White et al. [14] prepared self-healing microcapsules with urea-formaldehyde (UF) resin as the shell and dicyclopentadiene (DCPD) as the core through the in-situ polymerization method. The microcapsules and the catalyst Grubbs were added together to improve the inherent toughness of the polymer composites. And the fracture experiments yield as much as 75% recovery in toughness. In 2006, Keller [15] prepared the microcapsules with urea-resin as the wall and DCPD as the core and found that the microcapsules had good elastic deformation ability. In 2013, Su et al. [16,17] prepared microcapsules with the repair agent as the capsule core and methanol modified melamine resin as the shell and studied the thermal stability and mechanical properties of microcapsules and the interfacial stability between microcapsules and asphalt. Sun et al. [18] prepared microcapsules with melamine resin as capsule shell and rejuvenator as core in 2015 and found that the self-healing ability and fatigue life of asphalt can be improved by adding microcapsules. In 2015, Chung et al. [19] prepared microcapsules with UF resin as the wall and dimethyl phthalate as the core, observed the healing process of cracks in asphalt by scanning electron microscopy (SEM), and proved that the asphalt containing microcapsules has good mechanical properties. In 2016, Micaelo et al. [20] prepared microcapsules with sunflower oil encapsulated in calcium-alginate as the core and epoxy-cement composite as the shell, and the self-healing ability of asphalt mixtures containing the microcapsules was studied. In 2018, Al-Mansoori et al. [21] studied the self-healing ability of asphalt mastic by the action of calcium-alginate capsules containing sunflower oil.

The studies show that the use of microcapsules in asphalt materials is feasible and beneficial. After the addition of microcapsules, when microcracks occur next to the microcapsules, the rejuvenator is released from ruptured microcapsules to the cracks to increase the rate of self-healing. In this chapter, the self-healing microcapsules were prepared, and the properties of the microcapsules were characterized. Then, the microcapsule self-healing asphalt binder and mixture were prepared and the effect of microcapsule on the self-healing capacity, rheological property, and road performance was assessed.

## 9.2 Preparation of microcapsule

#### 9.2.1 Preparation of microcapsule

The ZS-1 asphalt rejuvenator was selected as the core material of microcapsules. UF resin was selected as the shell material. Self-healing microcapsules were prepared by in situ polymerization method. Fig. 9.1 shows the prepared UF microcapsule sample.

García et al. [11] made a microcapsule with the porous sand and the rejuvenator inside as core material and with the epoxy-cement matrix as shell material. The porous sand with particle size between 1 and 1.7 mm was designed to substitute part of the fine aggregate in asphalt mixture. The encapsulation procedure was as



Figure 9.1 UF microcapsule sample.

follows: To produce the core material, first, the sand was dried at 70°C for 24 h to remove the moisture, then both the sand and the rejuvenator were put in a container, heated at 105°C for 1 h, brought into a vacuum chamber for at least 30 min to remove the air, and then the rejuvenator penetrated the voids of the sand. To produce the shell, the sand with rejuvenators and epoxy were mixed by hand in a container. Then, they were added into another container with steel balls and cement together and the container rotate. As a result, the cement bonded to the epoxy and the epoxy-cement shell surrounding the sand was made.

Sun et al. [18] fabricated microcapsules by in situ polymerization process in an oilin-water emulsion with rejuvenator as core material and melamine-formaldehyde resin as the shell material.

Micaelo et al. [20] prepared three types of capsules: (1) capsule with three epoxy-cement coatings, (2) capsule with two epoxy-cement coatings, and (3) capsule without epoxy-cement coatings. The core of the capsules consisted of a structure made of porous calcium alginate that was encapsulated with the sunflower oil. In some cases, an outer shell made of epoxy cement was used to coat the calcium alginate. The procedure to fabricate the capsules composed of calcium alginate and rejuvenator was as follows: First, deionized water and oil were introduced in a glass container. Oil and water were homogenized by a gear-drive mixer. Then, sodium alginate was added and stirred. Simultaneously, a calcium chloride solution was prepared. Allowing the oil-in-water emulsion to drop into the calcium chloride emulsion, the microcapsule was formed. Finally, capsules were decanted, washed with deionized water, and dried by a fan. The procedure to coat the outer shell of epoxy cement was as follows: First, the calcium-alginate capsules were covered with epoxy. Then, the capsules coated by epoxy, steel balls, and cement were put in a container and the container was shaken by hand. After this process, cement, steel balls, and capsules were separated in a sieve. The epoxy-cement coating process could be repeated several times.

Aguirre et al. [22] made microcapsules via in situ polymerization. The core material of the microcapsules was a green bio-oil product. The double wall consisted of polyurethane and UF.

Su et al. [23] prepared microcapsules by in situ polymerization method using methanol melamine-formaldehyde as shell and rejuvenator as core material.

Garcia et al. [24] made the microcapsule with porous sand with sunflower oil as core material. The porous sand was designed with five different particle sizes of 0.60-1.18, 1.18-2.36, 2.36-3.35, 3.35-4.75, and 4.75-5.6 mm. The shell was made of epoxy-cement matrix.

## 9.3 Characterization of microcapsule

#### 9.3.1 Morphology

The SEM image of the prepared microcapsules is shown in Fig. 9.2. The UF microcapsule is basically a regular sphere without cracks and depression. And some fine



Figure 9.2 SEM image of UF microcapsule.

particles are adhered to and clustered on the surface of the microcapsules. Clustering phenomenon between some microcapsule samples is mainly due to the uneven precipitation of shell material prepolymer droplets on the surface of core particles droplets during the preparation of microcapsules.

SEM was used to analyze the morphology of the capsule consisted of porous sand and epoxy-cement shell made by García et al. [11]. Images obtained showed that the capsules appeared as aggregates.

The capsules made by Micaelo et al. [20] were approximately spherical, and the oil was contained in the micropores of the core. Furthermore, there was a gap between the shell and the core.

Su et al. [23] used an environmental scanning electron microscopy (ESEM) to study the morphology of microcapsules and found that the microcapsules had a regular global shape.

#### 9.3.2 Size distribution

The particle size of the microcapsule material was observed by using BT-2002 laser particle size analyzer. The microcapsule samples were dispersed in deionized water to form a solution by wet dispersion method. Then, the microcapsules were evenly dispersed in the circulatory system of laser particle size analyzer. And after a certain period of time, the particle size of microcapsules was measured by the analyzer.

The particle size distribution of microcapsule samples is shown in Fig. 9.3. The average particle size of UF microcapsule samples is 100.50  $\mu$ m, the particle sizes mainly concentrate in the range of 50–120  $\mu$ m. The wide distribution range of particle size distribution is mainly due to the different shear stresses at different locations of the droplets in the solution system during the microcapsule preparation process.



Figure 9.3 Particle size distribution of UF microcapsule.

In the microcapsule made by García et al. [11], the size of the porous sand was between 1.0 and 1.7 mm, and the size of the capsules was between 1.0 and 2.0 mm. The mean shell thickness was 0.10 mm.

Sun et al. [18] used laser diffraction particle size analyzer to measure the size of the microcapsules. The diameter of the single microcapsule was about  $2 \sim 3 \mu m$ . However, microcapsules were often agglomerated together. The mean size for agglomerated microcapsules was between 40 and 90  $\mu m$ .

The average size of capsule made by Micaelo et al. [20] lied between 4.6 and 6.8 mm and increased with the number of coatings. The microcapsule fabricated by Su et al. [23] had a mean size of  $20-50 \ \mu\text{m}$ . The average diameter of the five types of capsules made by Garcia et al. [24] was 2.23, 3.12, 3.64, 5.72, and 7.01 mm, respectively.

#### 9.3.3 Chemical structure

The microcapsule samples were dried, mixed with KBr, ground, and compressed by tablet press. The tableting was analyzed by FTIR-7600 Fourier transform infrared spectroscopy (FTIR) and then the chemical structure of microcapsules was obtained.

The FTIR spectrum of microcapsule is shown in Fig. 9.4. It can be seen that the core material has strong vibration in the vicinity of wavenumbers 3452.0, 2925.5, and 2854.1 cm<sup>-1</sup>, which are the characteristic absorption peaks of aromatic hydrocarbon (C–H) and carbonyl groups (-C = O). For the UF microcapsule material samples, the absorption peaks of imino (-NH), carbonyl (-C = O), and amide groups ( $-CO-NH_2$ ) are observed at 3417.2, 1743.3, and 1633.4 cm<sup>-1</sup>, respectively. Imino (-NH), carbonyl (-C = O), and amide groups ( $-CO-NH_2$ ) are characteristic functional groups of UF resin. The presence of these functional groups indicates that the UF resin shell has been successfully prepared. In addition, by analyzing the infrared data of the microcapsule material and the core material shown in



Figure 9.4 FTIR spectrum of UF microcapsule.

Fig. 9.4, it is found that the microcapsule sample has the characteristic peak of the core material, which proves that the core material exists in the microcapsule sample and has been successfully coated by the shell material.

#### 9.3.4 Thermal stability

The thermal stability of microcapsule samples was tested and analyzed by using HS-TGA-101 thermogravimetric analyzer to determine whether they can withstand the corresponding temperature during the construction of asphalt pavement. First, 10 mg microcapsule samples were weighed and protected by nitrogen. Then, test temperature was heated from ambient temperature to 300°C at a rate of 10°C/min.

The relationship between residual mass percentage and temperature is shown in Fig. 9.5. It could be seen that the mass loss percentage of microcapsule samples increases as the temperature increases. When the temperature rises from  $25^{\circ}$ C to  $100^{\circ}$ C, the sample mass loss percentage is 9.1%, and it is mainly caused by the evaporation of water during heating process. When the temperature rises from  $100^{\circ}$ C to  $250^{\circ}$ C, the mass loss rate of microcapsules keeps relatively stable and the mass decreases by 10%. During this temperature range, the mass loss is mainly due to the decomposition of the core material that is not covered by the shell. When the temperature of microcapsules and the decomposition of core materials. The temperature during storage, mixing, paving, and compaction process of asphalt mixture is between  $120^{\circ}$ C and  $180^{\circ}$ C, which indicates that the microcapsules are fairly thermally stable and can withstand the corresponding temperature during the construction of asphalt pavement to a certain extent.



Figure 9.5 Analysis of thermal stability of UF microcapsule.

García et al. [11] conducted thermogravimetry analysis (TGA) and differential scanning calorimetry (DSC) for the rejuvenator and the capsules. Results showed that the major decomposition of both epoxy and rejuvenator occurred at about 440°C. At the mixing temperatures between 160°C and 180°C, the losses in the capsules are between 0.8% and 1.3%, respectively. These losses were caused by the evaporation of the pore water in sand the dehydration of the capsule materials.

In the study of Sun et al. [18], TGA was also used to evaluate the thermal stability of microcapsules, shell material, and core materials. Core materials began to evaporate at 90°C and about 90% of the original mass was lost at 230°C. Shell materials began to evaporate at the beginning; however, only 25% of the original mass was lost when the temperature reached 270°C. As for microcapsules, the sudden mass loss occurred at 170°C. However, only about 20% of the original mass was lost when the temperature reached up to 190°C, indicating that the microcapsules were thermally stable during the construction of asphalt mixture.

In the study of Aguirre et al. [22], TGA results showed that there was a less than 8% of mass reduction in microcapsules.

Su et al. [23] evaluated the thermal stability of microcapsule in a fast heating process and an alternating temperature process. First, a fast heating process was applied to simulate a thermal shock for microcapsules. An asphalt sample was placed in an oven where the temperature was improved from room temperature to  $200^{\circ}$ C at a rate of 5°C/min. Then, a fluorescence microscope was used to observe the states of microcapsules in asphalt binder during the whole heating process. The image obtained showed that the microcapsules still kept their original shape and

was rarely ruptured. Second, an alternating temperature process was applied. Asphalt sample (MB-7) was heated to  $50^{\circ}$ C, and then the temperature decreased to  $-10^{\circ}$ C at a rate of  $2^{\circ}$ C/min. This process was repeated 50-100 times. A piece of the sample was peeled off and heated to  $150^{\circ}$ C; then the melting bitumen was evenly coated on glass slides and observed by a fluorescence microscope. The microcapsules could resist the temperature process repeated for 50 times but could not resist the process repeated for 70 times. Using TGA test, Garcia et al. [24] found that the oil almost did not lose mass at  $140^{\circ}$ C.

#### 9.3.5 Mechanical resistance

García et al. [11] applied both compression and tension tests on the capsules in which the displacement was applied at 5  $\mu$ m/s and the force-displacement curves of capsules were plotted. When under tension for three capsules, the ultimate resistance was 16.13, 14.48, and 11.15 N at 21.3, 16.5, and 13.75  $\mu$ m of deformation, respectively, and when under compression, the ultimate resistance was 10.92, 9.53, and 7.08 N at 44.02, 49.16, and 39.81  $\mu$ m of deformation, respectively.

Micaelo et al. [20] conducted a compressive test on the capsule. It was found that the compressive strength of microcapsule depended on the number of epoxycement coating and the testing temperature. When the temperature was low, the compressive strength of microcapsule was high. The average compressive strength of the capsules with three shell coatings was about twice the strength of other capsules with two shell coatings or without coating. The average compressive force of capsules was between 6.1 and 12.0 N at  $130^{\circ}\text{C}$ .

ESEM was used in the study of Su et al. [23] to observe the morphology and integrality states of microcapsules in asphalt mixture. The image obtained showed that microcapsules had a good bonding to bitumen so that debonding did not appear at the interface between microcapsules and asphalt binders. Microcapsules were rarely ruptured, which shows that microcapsule was not damaged by the agitation.

In the study of Garcia et al. [24], compressive resistance of capsules was measured at room temperature (20°C). The loading rate was 0.2 mm/min. The measured strengths of the capsules were 3.19, 3.02, 3.80, 2.01, and 1.67 N/mm<sup>2</sup> for capsules of 2.23, 3.12, 3.64, 5.72, and 7.01 mm, respectively.

#### 9.3.6 Capsule survival rate

Through scanning electron microscopy, Aguirre et al. [22] found that the developed microcapsules did not break during mixing with the aggregates.

In the study of Garcia et al. [24], FTIR was used to evaluate the capsule's survival rate after mixing and compacting. Bitumen was peeled off from the surface of the cores and measured using the FTIR. Pure sunflower oil and bitumen were also measured for comparison purposes. It is well known that there is a logarithmic relationship between FTIR transmittance and concentration. The strongest absorption peak was chosen to be compared in different materials. Results showed that the bigger capsules were easier to break than smaller capsules.

#### 9.3.7 Flowability behavior of core material

The flowability of the core materials is a key parameter for effective healing. Core material should flow out of the broken microcapsule and flow fast into the crack plane before consolidation of the agent. Sun et al. [18] used the laser microdissection to cut the microcapsules and to evaluate the flowability by microscopic visualization. When the shell was destroyed by the UV laser, the rejuvenator flowed out of the shell and proceeded quickly to the surroundings. The time needed for 55% of rejuvenator flowing out from the broken shell was only 10 s.

## 9.4 Self-healing property of microcapsule-containing asphalt binder

The neat asphalt binder (PG 58-16) was used and the properties of asphalt are shown in Table 9.1.

Matrix and self-healing asphalts of 2% and 3% UF microcapsule were prepared and the healing capacity at low and high temperatures was studied through the selfhealing ductility test and self-healing dynamic shear rheological (DSR) test.

#### 9.4.1 Self-healing ductility

The test specimens are shown in Fig. 9.6 and the specific test procedures were as follows: (1) The specimens of different types of asphalts were prepared. The cracks were manufactured by cutting a certain depth at the middle of the " $\infty$ " font of the asphalt sample, and there were 4.5 mm depth left not being cut. The asphalt samples with cracks would be divided into two groups, and each group included three specimens. (2) One group was performed ductility test at 10°C instantly, and the ductility of this group was recorded as  $L_{\text{Original}}$ . (3) Another group was placed at 25°C for 4 h, and then ductility test was carried out at 10°C. The ductility of this group was recorded as  $L_{\text{Healed}}$ . The ability of self-healing was quantified by the rate of ductility healing denoted as P [14], which was calculated by:

$$P = \frac{\left(L_{\text{healed}} - L_{\text{original}}\right)}{L_{\text{original}}} \tag{9.1}$$

Table 9.1	Properties	of neat	asphalt	binder.
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Test items	Value
Penetration (25°C, 100 g, 5 s) (0.1 mm)	90.8
Softening point (°C)	47.3
Ductility (50 mm/min, 10°C) (cm)	38.8
Density (g/cm <sup>3</sup> )	0.992



Figure 9.6 Specimens of self-healing ductility test.

 Table 9.2
 Self-healing ductility test results.

Asphalt	Matrix asphalt	Asphalt of 2% UF microcapsule	Asphalt of 3% UF microcapsule
Ductility before repairing, $L_0$ (cm)	8.4	6.9	6.0
Ductility after repairing, $L_{\rm H}$ (cm)	10.5	9.1	8.2
Ductility healing rate, P (%)	25.0	31.8	36.6

The self-healing ductility test results are shown in Table 9.2. It can be seen from Table 9.2 that the ductility healing rate P of asphalt-containing UF microcapsules of 2% and 3% increases by 6.8% and 11.6%, respectively. The reason for this phenomenon is that the cracks in the asphalt sample expand and the stress concentration occurs in the tip of the cracks; subsequently the microcapsules rupture and the rejuvenator released repairs the cracks during the healing interval. And the amount of released repair agent increases with the increase in the microcapsules content.

#### 9.4.2 Self-healing dynamic shear rheological test results

The self-healing DSR test performed in this paper was one of the fatiguehealing-refatigue tests. First, a certain fatigue damage (fatigue fracture) was produced in asphalt by the DSR instrument. Second, loading was stopped and the cracks were repaired at a certain temperature and intermittent time. Finally, the second fatigue damage test was carried out. The healing ability of asphalt was evaluated by the changes in the dynamic modulus and fatigue life of asphalt



Figure 9.7 Self-healing DSR fatigue curve of matrix asphalt.

samples during the healing interval and characterized by the healing index (HI). The higher the HI, the better the healing ability of the asphalt. The following parameters are involved in the calculation of HI, and their meaning and position in the fatigue test are shown in Fig. 9.7. In this figure,  $G^*_{\text{initial}}$  is the initial complex shear modulus of the sample before the first loading;  $G^*_{\text{before}}$  is the fatigue failure control point of the first loading test. When  $G^*$  drops to this value, the first loading test stops and the number of fatigue is denoted as  $N_{\text{before}}$ ;  $G^*_{\text{after}}$  is the complex modulus of the asphalt sample that is healed at 25°C for 1 h;  $G^*_{\text{terminal}}$  is the complex modulus of the sample, which drops to  $G^*_{\text{before}}$  again,  $G^*_{\text{terminal}} = G^*_{\text{before}}$ ; the number of fatigue corresponding to complex shear modulus  $G^*_{\text{terminal}}$  is recorded as  $N_{\text{after}}$ .

At present, self-healing properties are mainly evaluated by healing indices (HI), which means the changes in asphalt properties such as complex shear modulus, phase angle, fatigue life, and so on, during self-healing process. Three common definition methods of HI are adopted, which are shown in the Eqs. (9.2)-(9.5) [25].

1. HI<sup>1</sup>, defined by the asphalt modulus growth rate during self-healing interval:

$$HI^{1} = (|G^{*}|_{after} - |G^{*}|_{before}) / |G^{*}|_{before}$$
(9.2)

2. HI<sup>2</sup>, defined by the correction ratio of loading times during self-healing interval:

$$HI^{2} = (N_{after} - N_{before})/N_{before} * |G^{*}|_{terminal}/|G^{*}|_{initial}$$
(9.3)

**3.** HI<sup>3</sup>, defined by the ratio of dissipated energy (DE) before and after self-healing interval:

$$HI^{3} = \sum DE_{after} / \sum DE_{before}$$
(9.4)

where  $\sum DE_{before}$  and  $\sum DE_{after}$  are the accumulative DE before and after self-healing interval, respectively.

DE can be calculated as follows:

$$DE_i = \sin\delta_i * \pi\varepsilon_i^2 * G_i^* \tag{9.5}$$

where DE<sub>i</sub> is the dissipated energy by the *i*th loading cycle,  $\delta_i$  is the phase angle,  $\varepsilon_i$  is the strain (%) and  $G_i^*$  is the complex shear modulus under *i*th loading cycle.

The self-healing DSR test was carried out according to ASTM-D 4402 [26]. In the test, the stress controlling mode was used, the test temperature was 20°C and the load-ing frequency was 10 Hz. The fatigue stresses of 0.1, 0.2, 0.3, and 0.4 MPa were used.

The fatigue curves of the matrix asphalt under different loading stresses are shown in Fig. 9.8. It can be seen that the complex shear modulus  $G^*$  of asphalt decreases gradually with the increase of loading times. The occurrence of fatigue damage needs a very long time if using 0.1 or 0.2 MPa as loading stress, and a relatively short time if using 0.4 MPa as loading stress. When using 0.3 MPa as the loading stress, the test time is appropriate, and the fatigue properties of asphalt can be analyzed more conveniently. Therefore, 0.3 MPa was used as the loading stress in fatigue tests.

From Fig. 9.8, it can be found that the initial complex modulus  $G^*$  of the matrix asphalt under loading stress of 0.3 MPa is 5.1521 MPa. When the complex modulus



Figure 9.8 Fatigue curves of the matrix asphalt under different loading stresses.

Asphalt	Matrix asphalt	Asphalt of 2% microcapsule	Asphalt of 3% microcapsule
$G^*_{\text{initial}}$ (MPa)	5.23	5.1172	5.3436
$G^*_{\text{before}}$ (MPa)	3.1422	3.0326	3.1203
$G^*_{after}$ (MPa)	3.9568	4.1757	4.7307
$G^*_{\text{terminal}}$ (MPa)	3.1422	3.0326	3.1203
N <sub>before</sub> (time)	3419	3784	4207
N <sub>after</sub> (time)	3785	4306	4830
$DE_{before}$ (×10 <sup>7</sup> Pa · time)	7.87	11.62	10.86
$DE_{after}$ ( $\times 10^{6}Pa \cdot time$ )	9.47	18.97	20.91

Table 9.3 Self-healing dynamic shear rheological test result of asphalts.

 $G^*$  is reduced to 2.9738 MPa (about 58% of the initial modulus), the descent rate of  $G^*$  begins to increase obviously. It can be inferred that there is a certain degree of fatigue damage occurring at this time. Therefore, the loading times when the modulus decreased to 60% of the initial modulus was regarded as the fatigue failure control point corresponding to  $N_{\text{before}}$  in Fig. 9.7. The fatigue-healing-refatigue tests were carried out for matrix asphalt and asphalts of 2% and 3% microcapsules, and test results are listed in Table 9.3. The addition of 2% and 3% microcapsules makes the  $G^*_{\text{after}}$  increase by 0.3285 and 0.7958 MPa, respectively, and make the fatigue life ( $N_{\text{after}} - N_{\text{before}}$ ) increase by 156 and 257 times, respectively. The results prove that after adding microcapsules, the healing ability of the asphalt increases with the increase in microcapsule content.

The healing indices can be calculated according to the data shown in Table 9.3 and are illustrated in Fig. 9.9. It can be seen from this figure that the healing ability of asphalt increases obviously after the addition of microcapsules, and the increment of healing ability increases with the increase in microcapsule content. This is because the microcracks occur in the asphalt material during the fatigue damage. The stress on the tip of microcapsules rupture and accordingly the repair agent is released to repair cracks, restore the dynamic modulus of the asphalt to some extent and extend its fatigue life. As for the three healing indices, HI<sup>1</sup> can distinguish the healing abilities of different asphalts very well, followed by HI<sup>3</sup> and HI<sup>2</sup>.

Fatigue life recovery test utilizing DSR was also used to study the healing effect of microcapsules in asphalt by Sun et al. [18]. Results showed that the microcapsules could improve the healing capability of asphalt.

## 9.5 Rheological properties of microcapsule-containing asphalt binder

First, the original matrix asphalt and asphalts of 1%, 2%, 3%, 4%, and 5% UF microcapsule were selected in this paper for consistency tests. Then, after aged by



Figure 9.9 Self-healing indexes of asphalt.

rolling thin film oven test (RTFOT), the aged asphalts were subjected to consistency tests as well. Finally, according to the consistency property and durability, two microcapsule contents would be picked out from the five microcapsule contents, and the following property would be tested on the matrix asphalt and the microcapsule self-healing asphalts with the two selected microcapsule contents.

#### 9.5.1 Consistency property

The penetration test was carried out in accordance with ASTM-D5 [27]. The softening point test was conducted according to ASTM-D36 [28]. The ductility test at 10°C was carried out in accordance with ASTM-D113 [29]. The viscosity tests at 60°C and 135°C were conducted according to ASTM-D2171 [30] and ASTM-D2170 [31], respectively.

The penetration, softening point, ductility at  $10^{\circ}$ C, and viscosities at  $60^{\circ}$ C and  $135^{\circ}$ C are shown in Table 9.4 and Fig. 9.10. It can be seen from Table 9.4 that as the microcapsules content increase, softening point increases, penetration and ductility decrease. "Highway Engineering Asphalt and Asphalt Mixture Test Code" (JTG E20–2011) in China specifies that the minimum ductility of asphalt at  $10^{\circ}$ C is 20 cm. When the content of UF microcapsule is 5%, the ductility of asphalt cannot meet this requirement anymore. When the microcapsule content is 4%, the ductility of asphalt is 21.1 cm and it should be used in practice with caution. It can be seen from Fig. 9.10 that the viscosity of asphalt rises with the increase in microcapsule content.

Adding microcapsules into asphalt is similar to the incorporation of micron-sized solid particles in the asphalt, which makes the asphalt thicker. At the same time, part of

Microcapsule content (wt. %)	0	1	2	3	4	5
Penetration (25°C (100 g, 5 s) (0.1 mm)	90.8	85.7	78.4	73.3	68.9	65.4
Softening point (°C)	47.3	48.2	50.1	52.4	53.7	55.1
Ductility (50 mm/min (10°C) (cm)	43.8	37.1	31.6	26.5	21.1	18.2

 Table 9.4 Consistency properties of urea-formaldehyde microcapsule self-healing asphalt.



Figure 9.10 Viscosities of UF microcapsule self-healing asphalt at 60°C and 135°C.

aromatics and saturate in asphalt material were absorbed by microcapsules. The swelling of the microcapsule particles in the asphalt changes the composition of the asphalt; as a result, the content of the light component becomes less. And the more the swelled microcapsules, the more difficult the relative movement of microcapsules. So, the asphalt becomes hard, the penetration is reduced, and the softening point and viscosity are increased. Compared with modified asphalt, matrix asphalt is a more homogeneous system in which physical and chemical properties are more uniform. After adding the microcapsules to the asphalt, the homogeneous structure of the matrix asphalt sample is broken to form a new heterogeneous structure. The resistance to deformation of swelled microcapsules is poor, and stress concentration occurs in asphalt, which leads to the decreasing of the ductility with the increase of microcapsule content.

#### 9.5.2 Durability

The asphalt samples were aged by an RTFOT according to the method specified in ASTM-D 2872 [32]. After 75 min of aging process, mass loss of bitumen,

penetration, softening point, ductility at  $10^{\circ}$ C, viscosities at 60 and  $135^{\circ}$ C were tested and compared with these properties of asphalt before aging. Through comprehensive analysis, the effect of microcapsules on the antiaging properties of asphalt would be obtained.

The antiaging property of asphalt was evaluated by the viscosity index (VAI), residual penetration, increment of softening point, and ductility retention shown in Eqs. (9.6)-(9.9). The greater the VAI, residual penetration, and ductility retention and the lower increment of softening point, the more serious the aging of asphalt.

$$VAI(\%) = (V_{after} - V_{before}) / V_{before} \times 100\%$$
(9.6)

Residual penetration(%) =  $P_{\text{after}}/P_{\text{before}}$  (9.7)

Increment of softening point (°C) =  $S_{after} - S_{before}$  (9.8)

Ductility retention(%) = 
$$D_{after}/D_{before}$$
 (9.9)

where  $V_{\text{before}}$ ,  $P_{\text{before}}$ ,  $S_{\text{before}}$ , and  $D_{\text{before}}$  are the viscosity, penetration, softening point, and ductility of asphalt before aging, respectively, and  $V_{\text{after}}$ ,  $P_{\text{after}}$ ,  $S_{\text{after}}$ , and  $D_{\text{after}}$  are the viscosity, penetration, softening point, and ductility of asphalt after aging, respectively.

Tables 9.5–9.7 list the changes in penetration, softening point, and ductility of asphalts during aging process. The addition of microcapsules of 1%, 2%, 3%, 4% and 5% increases the residual penetration ratio by 2.5%, 3.5%, 5.2%, 5.9%, and 6.5%, respectively; decreases the increment of softening point by 1.0°C, 1.9°C, 2.7°C, 3.1°C, and 3.4°C, respectively; and increases the ductility retention by 11.9%, 15.8%, 20.3%, 22.1%, and 22.6%, respectively.

Table 9.5	Penetration of	f urea-formal	ldehyde	e microcapsul	e self-healing	asphalt	before a	nd
after aging	<b>5.</b>							

Microcapsule content (wt.%)	0	1	2	3	4	5
Before aging (0.1 mm)	90.8	85.7	78.4	73.3	68.9	65.4
After aging (0.1 mm)	63.8	62.4	57.9	55.4	52.5	50.2
Residual penetration ratio (%)	70.3	72.8	73.8	75.5	76.2	76.8

**Table 9.6** Softening point of urea-formaldehyde microcapsule self-healing asphalt before and after aging.

Microcapsule content (wt.%)	0	1	2	3	4	5
Before aging (°C)	47.3	48.2	50.1	52.4	53.7	55.1
After aging (°C)	53.6	53.5	54.5	56.0	56.9	58.0
Increment of softening point (°C)	6.3	5.3	4.4	3.6	3.2	2.9

 Table 9.7 Ductility at 10°C of urea-formaldehyde microcapsule self-healing asphalt before and after aging.

Microcapsule content (wt. %)	0%	1%	2%	3%	4%	5%
Before aging (cm)	43.8	37.7	31.6	26.5	21.1	18.2
After aging (cm)	14.4	16.9	15.4	14.1	11.6	10.1
Ductility retention (%)	32.9	44.8	48.7	53.2	52.0	55.5

**Table 9.8** Viscosity (at 60°C) of asphalt before and after aging.

Microcapsule content (wt.%)	0	1	2	3	4	5
Before aging (Pa · s)	178.3	183.5	199.1	211.5	217.2	225.8
After aging (Pa · s)	219.7	229.0	244.8	254.2	256.9	266.0
VAI (%)	23.2	24.8	23.0	20.2	18.3	17.8

The changes in the viscosity during aging process of asphalt are listed in Tables 9.8 and 9.9. It can be seen that the VAI values of UF microcapsules self-healing asphalt are lower than that of matrix asphalt and decreases with the increasing microcapsule content. The lower the VAI values, the better the antiaging ability.

According to the effects of UF microcapsules on the changes in penetration, softening point, ductility, and viscosity during aging process, it can be concluded that the addition of microcapsules can slow down the aging of asphalt. In aging process, the internal microcracks take place and the stress concentration phenomenon occurs in the tip of them. When the stress is greater than the rupture strength of microcapsules, microcapsules rupture, and repair agent is released to repair cracks and restore the partial characteristics of asphalt. This may explain the effects of microcapsule on the aging performance of asphalt.

The ductility values of the asphalt of 4% and 5% UF microcapsules are close to or less than the limitation specified in Chinese specification. Therefore, the matrix asphalt and the asphalts of 2% and 3% UF microcapsule will selected out and they will be conducted for subsequent tests.

#### 9.5.3 High-temperature stability

According to ASTM-D 4402 [26], the complex modulus ( $G^*$ ) and phase angle ( $\delta$ ) of the asphalt specimen were measured by the DSR test, which could adequately characterize the viscous and elastic behavior of asphalt binders at high and intermediate service temperature. In addition, the rutting resistance of the asphalt material is usually characterized by the rutting factor  $G^*/\sin\delta$ , and the higher the rutting factor, the smaller the flow deformation and the more resistance to rutting. In this test, the temperatures selected were 58°C, 64°C, 70°C, and 76°C.

Microcapsule content (wt. %)	0	1	2	3	4	5
Before aging (mPa · s)	325.1	351.8	379.4	399.8	415.6	420.2
After aging (mPa · s)	548.3	557.5	566.7	574.9	580.4	575.7
VAI (%)	68.6	58.5	49.4	43.8	39.7	37

Table 9.9 Viscosity (at 135°C) of asphalt before and after aging.

Table 9.10 DSR test results of asphalt.

Test item	Temperature (°C)	Matrix asphalt	Asphalt of 2% microcapsule	Asphalt of 3% microcapsule
Complex shear	58	3.359	3.502	3.652
modulus, $G^*$	64	1.645	1.717	1.774
(kPa)	70	0.840	0.884	0.913
	76	0.455	0.478	0.494
Phase angle,	58	83.6	83.57	83.41
$\delta$ (°)	64	84.23	84.25	84.18
	70	83.92	84.03	84.02
	76	82.26	82.51	82.59
Rutting factor,	58	3.380	3.524	3.676
$G^*/\sin\delta$ (kPa)	64	1.653	1.725	1.783
	70	0.845	0.889	0.918
	76	0.459	0.482	0.498

The DSR test results of matrix asphalt and asphalts of 2% and 3% UF microcapsule are listed in Table 9.10. After the microcapsules are incorporated, they exist in the asphalt in the form of particles. The microcapsule particles influence the shearing action significantly, so the loss of elasticity of the asphalt becomes relatively small, and the phase angle  $\delta$  gradually decreases. Therefore,  $G^*/\sin\delta$  increases after the incorporation of UF microcapsules, which implies that the antiaging ability of asphalt becomes better. And the more the microcapsule content, the larger the  $G^*/\sin\delta$  and the better the antirutting ability.

#### 9.5.4 Low-temperature crack resistance

The bending beam rheometer (BBR) test was carried out according to ASTM B6648 [33]. The bending creep stiffness modulus *S* and the slope of the creep curve *m* are the two parameters used to characterize the degree of flexibility of asphalt and to evaluate the low-temperature crack resistance of asphalt binder. Test temperatures were  $-12^{\circ}$ C,  $-18^{\circ}$ C, and  $-24^{\circ}$ C.

Classic beam analysis theory is used to calculate the creep stiffness of the asphalt binder beam at 60 s loading time, as follows:

$$S(t) = PL^3/4bh^3\delta(t) \tag{9.10}$$

where S(t) is the creep stiffness at 60 s, *P* is the applied constant load (100 g), *L* is the distance between beam supports (102 mm), *b* is the beam width (12.5 mm), *h* is the beam thickness (6.25 mm), and  $\delta(t)$  is the deflection at 60 s.

BBR test results are shown in Figs. 9.11 and 9.12. It can be seen that the creep stiffness modulus S increases and the creep curve slope m decreases slightly after the addition of UF microcapsules under the same temperature condition, which indicate that the low-temperature anticracking performance of asphalt is decreased due to the addition of UF microcapsules. The incorporation of UF microcapsules breaks the homogeneous structure of the asphalt sample and reduces the flexibility and ductility of the asphalt binder and makes the microcapsules swell, which result in greater temperature stress and lower low-temperature cracking resistance.



Figure 9.11 Creep stiffness modulus S of asphalt.



Figure 9.12 Creep curve slope *m* of asphalt.

# 9.6 Self-healing property of microcapsule-containing asphalt mixture

Continuously graded AC-13 was selected. The optimal asphalt content was 5.0%. The coarse aggregate, fine aggregate, and mineral filler were made from basalt, limestone, and limestone minerals, respectively.

#### 9.6.1 Fatigue life prolongation

Midpoint trabecular bending test was used to assess the antifatigue performance of asphalt mixture in accordance with ASTM D7460-10 [34]. Fatigue-rest-refatigue test was designed to assess the fatigue life prolongation introduced by microcapsule.

The asphalt mixture specimen size was  $250 \times 40 \times 40$  mm. In the self-healing ability test, two groups of asphalt mixture specimens were needed. One group was subjected to fatigue test and calculated the average fatigue life of the asphalt mixture, denoted as  $N_{f}$ . Another group was first subjected to fatigue test as well, and the loading times was a certain percentage of  $N_{f}$ , denoted  $N_{1}$ . Then, the specimens were placed at 25°C for 4 h. After the healing interval, fatigue test was carried out on the specimens continuously until broken, and the loading times was denoted as  $N_{2}$ .

The self-healing ability of asphalt mixture was quantified by the healing rate P as shown in Eq. (9.11).

$$P = (N_1 + N_2 - N_f)/N_f$$
(9.11)

Self-healing ability test results showed that the self-healing ability of asphalt mixture is improved significantly after adding 3% UF microcapsules. And the degree of improvement is related to healing temperature, healing time, and damage degree. The larger the healing temperature is, the larger the healing rate will be. The more healing the time is, the larger the healing rate will be. The larger the smaller the healing rate will be.

#### 9.6.2 Stiffness recovery

Micaelo et al. [20] evaluated the healing capacity of asphalt mixture with stiffness recovery. Asphalt mixture specimens were subjected to cyclic uniaxial compression test without lateral confinement. The test temperature was 20°C. The test included three stages: initial loading, resting, and reloading. During the initial loading stage, an obvious crack was induced in the specimen. The effect of the resting period was assessed according to the stiffness of the asphalt mixture. During resting time, the oil may flow out of the capsule, diffuse in the crack plane, and heal part of crack. As a result, the resting time may change the stiffness of the asphalt mixture. Different resting periods of 3, 6, 12, 24, and 48 h were adopted. Results showed that oil diffused in the bitumen at 20°C. Whether the resting time affected the stiffness of asphalt mixture or not depended on the epoxy-cement shell. The asphalt mixture containing capsules

with three epoxy-cement shells had the same stiffness after the resting period. On the other hand, for the asphalt mixture containing capsules without epoxy-cement shell, the stiffness increased by approximately 15% after resting for 48 h.

Aguirre et al. [22] also evaluated the healing capacity of asphalt mixture with stiff recovery. In their study, a three-point bending test was conducted for the damaged, undamaged, and healed asphalt specimens. The stiffness was calculated according to the test results. The damaged and healed stiffness values were compared to determine the stiffness recovery at the end of the healing period. The undamaged and healed stiffness values were also compared. Results showed that the healed stiffness was greater than the damaged stiffness, proving that there was a recovery in the mixture stiffness. But the healed stiffness was less than the undamaged stiffness. At higher temperatures, the stiffness recovery was greater.

#### 9.6.3 Crack observation

Aguirre et al. [22] evaluated the healing capacity of asphalt mixture through observation. In their study, during a three-point bending test the crack width was measured using an optical light microscope before healing and at different days of the healing period. Then, the healing process of specimens could be quantified as a function of time. The healing efficiency of the specimens at different healing periods was calculated as follows:

$$H_e = \left(1 - \frac{C_{wt}}{C_{w0}}\right) \times 100 \tag{9.12}$$

where  $H_e$  is the healing efficiency (%),  $C_{w0}$  is the initial crack width (mm), and  $C_{w1}$  is the crack width (mm) at the time of analysis. The images obtained showed a reduction in the size of the cracks in a resting period from 0 to 6 days. The healing efficiencies at room temperature were higher than that at high temperatures for all mixtures.

Su et al. [23] also evaluated the healing capacity of asphalt mixture through observation. Asphalt material was poured into the middle of two aluminum plates with a thickness of 1 cm. Liquid nitrogen  $(N_2)$  was dropped on the surface of asphalt sample to make microcracks in asphalt. Using the fluorescence microscope, it could be found that the microcapsules were pierced by a propagated microcrack successfully and the rejuvenator flowed out.

## 9.7 Mechanical and pavement performance of microcapsule-containing asphalt mixture

#### 9.7.1 Indirect tensile strength

Garcia et al. [24] measured the tensile strength of asphalt mixture containing capsules with different sizes using indirect tensile strength test at 5°C. It could be found that the addition of capsules decreased the indirect tensile strength by about 30%. In addition, the size of capsules had almost no effect on the indirect tensile strength. The reason for the reduced strength was that the tensile strength of the capsule was lower than that of the aggregate. On the other hand, if the tensile strength of the capsule was not lower than that of the aggregate, the capsule would not break when encountering a crack. So, the reduction of strength of asphalt mixture containing capsules could not be avoided.

#### 9.7.2 Indirect tensile stiffness modulus

Garcia et al. [24] studied the effects of microcapsules on the tensile stiffness modulus of asphalt mixture. The stiffness of the different asphalt mixtures containing capsules was measured using indirect tensile stiffness modulus test at 5°C. The load frequency was 40 cycles/min with 124 ms rise-time. Results showed that the addition of capsules reduced the stiffness of asphalt mixture and bigger capsules caused more stiffness loss. The reason maybe that some capsules broke during mixing and compaction processes and the leaked oil reduced the stiffness. So, it seemed that the reduction of stiffness of asphalt mixture containing capsules could not also be avoided.

#### 9.7.3 Antifatigue performance

Midpoint trabecular bending test was used to assess the antifatigue performance of asphalt mixture in accordance with ASTM D7460-10 [34]. The trabecular specimen size was  $250 \times 40 \times 40$  mm in the midpoint trabecular bending test. The sine-wave loading with a frequency of 10 Hz was applied to the asphalt mixture specimen at  $20^{\circ}$ C. And the loading peak is a certain percentage of the flexural strength of asphalt mixture, which was regarded as stress ratio. The fatigue life of asphalt mixture specimen is the times of loading applications before the specimen is broken under the cyclic sine-wave loading.

Midpoint trabecular bending test results showed that the fatigue life of asphalt mixture with 3% UF microcapsules increased, compared with that of matrix asphalt mixture. And the larger the stress ratio is, the larger the degree of increment will be.

Garcia et al. [24] applied indirect tensile fatigue tests to asphalt mixtures containing capsules. Twenty percent of the peak stress was loaded. The load frequency was 40 cycles/min with 124 ms rise-time. It could be found that the addition or size of capsules did not influence the total life of the asphalt mixture.

#### 9.7.4 High-temperature stability

The rutting test was conducted to evaluate the high-temperature stability of asphalt mixtures in accordance with the test method of JTG E20-2011 [35]. The test specimen size was  $300 \times 300 \times 50$  mm. During rutting test, the asphalt mixture specimens were applied 0.7 MPa loading at the speed of 21 cycles/min by rubber wheel, and the covered distance was 230 mm. High-temperature stability is mainly evaluated by dynamic

stability (DS), which refers to the loading times of the standard axle load when the asphalt mixture is deformed by 1 mm. And the DS was calculated by the deformations of specimens at 45 and 60 min at  $60^{\circ}$ C.

Rutting test results showed that the DS of asphalt mixture increased by 16.5% after adding 3% UF microcapsules, indicating that the addition of 3% microcapsules improved the high-temperature stability of asphalt mixture.

#### 9.7.5 Low-temperature crack resistance

The low-temperature bending test was performed according to JTG E20-2011 [35] of China. The test beam specimen size was  $250 \times 30 \times 35$  mm. The loading was applied to the center of the asphalt mixture specimen by creep testing machine at a speed of 50 mm/min until the specimen was broken. During the low-temperature bending test, the maximum loading and deflection at the center of specimen were recorded as  $P_{\rm B}$  and d, respectively. The flexural strength  $R_{\rm B}$  and tensile strain  $\varepsilon_{\rm B}$  were calculated by the maximum loading and deflection at the center of specimen. And the bending stiffness modulus was the ratio of  $R_{\rm B}$  and  $\varepsilon_{\rm B}$ .

Low-temperature bending test results showed that the bending stiffness modulus of asphalt mixture with 3% UF microcapsules increased 7.1% compared with that of matrix asphalt mixture. However, the flexural strength and tensile strain of asphalt mixture decreased 3.7% and 10.1% after adding 3% UF microcapsules, respectively.

#### 9.7.6 Water stability

The immersed Marshall test and freeze-thaw splitting test were conducted to study the water stability of the asphalt mixture in accordance with ASTM D6927 [36] and AASHTO T 283 [37], respectively.

Two groups of asphalt mixture specimens were needed for immersed Marshall test. One group was immersed in water at 60°C for 0.5 h and tested for the Marshall stability, denoted as *MS*. Another group was immersed in water at 60°C for 48 h and tested for the Marshall stability, denoted as *MS*<sub>1</sub>. The water stability was characterized by residual stability (*MS*<sub>0</sub>), which is the ratio of *MS*<sub>1</sub> and *MS*.

Immersed Marshall test results showed that the Marshall stability at 0.5 and 48 h of asphalt mixture increased after adding 3% UF microcapsules, and the residual stability of the asphalt mixture with 3% UF microcapsules increased by 2.27% compared with the matrix asphalt mixture.

Two groups of asphalt mixture specimens were needed for freeze-thaw splitting test. One group was placed at 25°C for 2 h and tested for the splitting strength, denoted as  $\delta_1$ . Another group was first placed under vacuum at  $-18^{\circ}$ C for 16 h, then placed in water at 60°C for 24 h, and finally placed in water at 25°C for 2 h. The splitting strength of the group was tested and denoted as  $\delta_2$ . The water stability was characterized by the freeze-thaw splitting strength ratio of  $\delta_2$  and  $\delta_1$ .

Freeze-thaw splitting test results showed that the splitting strengths  $\delta_1$  and  $\delta_2$  increased after adding 3% UF microcapsules, and the freeze-thaw splitting strength ratio of asphalt mixture with 3% UF microcapsules increased by 3.70%.

From the above results of immersed Marshall test and freeze-thaw splitting test, it could be found that the addition of microcapsules improved the water stability of asphalt mixture to a small extent.

### 9.8 Future trends

- **1.** In the future, test roads need to be constructed to verify the self-healing ability and the road performance of the asphalt mixture containing microcapsule.
- **2.** On the one hand, microcapsules should withstand the agitation during mixing; on the other hand, they should break when encountering a cracking. Better shell materials need to be explored.

## 9.9 Sources of further information and advice

Further information can be found at the following references:

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## Self-healing biomimetic microvascular containing oily rejuvenator for prolonging life of bitumen

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## 10.1 Introduction of biomimetic microvascular self-healing

Some irreversible aging and damage can be found because of the influence of environmental factors or stress in the course of using materials [1]. These injuries are often hidden in the material and are difficult to come to light. Long-term durability and reliability are critical to the materials used in the structure [2]. Self-healing function is one of the most basic characteristics of nature and self-healing materials are still considered to be an important area of research in next years [3,4]. The selfhealing research studies are carried out in nearly all types of materials, such as ceramics, metals, polymers, and cementitious materials. Self-healing can be divided into two classes according to the healing mechanism, namely intrinsic and extrinsic self-healing materials [5]. The intrinsic ones can be obtained through different chemical reaction approaches, such as photo inducement, molecular interdiffusion reversible bond formation, and recombination of chain ends [6]. On the contrary, the extrinsic ones may do not possess an intrinsic self-healing capability or owe a relatively weak self-healing capability. In other words, these materials need external healing components to achieve the self-healing function by deliberately embedding microcapsules and hollow fibers containing healing agents [7].

In recent years, the concept of self-healing has been extended to the engineering and construction fields [8]. Interestingly, it is worth mentioning that the self-healing bitumen has attracted increasing attention in self-healing materials because this smart self-healing technology is considered as a potential revolution for the pavements. Predictably, self-healing materials may convert the construction and maintenance of pavements by increasing the service time and decreasing the maintenance cost [9]. Self-healing bitumen can reduce the dosage of resources in pavement maintenance, decrease the traffic blockage during maintenance, decrease pollutant and greenhouse gases emissions, and elevate road safety and lifespan [10]. Undoubtedly, the self-healing technology will enhance the level of intelligence of roads for the future. At present, the bituminous pavement designs follow the principles of enhancing performance, increasing durability, and improving load-carrying capability. With the progress of intelligent science and technology, the bitumen pavement design may be motivated efforts to accomplish a goal allowing roads to repair themselves to a certain extent of original state [11].

In general, the self-healing capability of bituminous pavements can be improved by external additives. To achieve the above goal, these additives must survive and prevail in the harsh conditions during treatment process and service life in asphalt. [12]. To date, a literature review has shown that two methods are considered as the effective approaches for the self-healing bitumen. The first one is called as heat induction method, which has gained popularity in self-healing bituminous material research [13]. Electrical conductive fibers and fillers were added into bituminous pavement and heat produced by electric current enhanced the self-healing ability of bituminous materials. Another method is the usage of microcapsules containing oily rejuvenator [14] or hollow fibers containing rejuvenator [15]. The self-healing mechanism of bitumen applying microcapsules containing rejuvenator has been mentioned systemically in our previous works [11,14,16]. Several microcapsules could be punctured by the tip-stress of a microcrack in its propagation path. The encapsulated oily rejuvenator was then released and moved into the gap of the cracks through capillary. Small molecules of rejuvenator then diffused into the aged molecules of bitumen and softened the bituminous binders. The viscous flow facilitated a healing process and prevented a further propagation of the microcrack [17]. The self-healing processes mentioned above were also repeatable as a multi-self-healing, which has been proved in previous works [14]. In particular, the microencapsulated waste cooking oil was recycle-used as self-healing additive in asphalt. This idea has potential environmental value and technological value. Besides the capsules system, hollow fibers containing rejuvenator have also been fabricated for self-healing bitumen. Tabaković et al. [15] fabricated sodium alginate fibers within bituminous rejuvenator for self-healing bitumen by electrospinning method. Results showed that these compartmented fibers demonstrated good thermal and mechanical strength. Limited by the spinning technology, the hollow fibers did not have a continuous hollow cavity. When rejuvenator is encapsulated in fibers, this structure may block the smooth flow of liquid rejuvenator. Sodium alginate undergoes dehydration process at 60°C-170°C. Therefore, it can be deduced that these sodium alginate fibers may not resist the high temperature (180°C) of bitumen during pavements. In addition, the fibers prepared by electrospinning are difficult to ensure the strength of the fibers satisfying the application in bituminous pavement. As a biomaterial extracted widely from the cell walls of brown algae, the sodium alginate will also biodegrade in nature, losing its original shape and material. To overcome the above shortcoming, Su [18] reported a preparation method of continuous hollow fibers containing rejuvenator by a spinning technology using PVDF resin. Because of the presence of fluorine element, the fibers had an outstanding thermal stability and mechanical property.

Both approaches of using microcapsules or hollow fibers are based on the supplementation of small molecules into aged bitumen. Bituminous material unavoidably loses part of viscoelastic character after a few years. The molecules of rejuvenator will soften the aged bitumen and recover the viscoelastic capability. Self-healing phenomena normally appear in the binders of asphalt mixture. However, no released rejuvenator molecules can penetrate into aged materials without breaks of microcapsules or hollow fibers. In other words, the self-healing will not occur when no crack has appeared. The original properties of bitumen are lost during a long service time suffering external environmental factors. Predictably but disappointingly, the mentioned microcapsules or hollow fibers methods are only remedial measures when cracks have already generated. Therefore, it is a perfect approach to take preventive measures in advance. We know that human skin will be aging caused by many internal and external factors and it becomes thinner, less elastic, and more easily damaged. An effective antiaging method is using skin creams, which are designed to supply small molecules to skins, reducing or diminishing the effects of aging. Inspired by the skin nutrition concept, it will be an ideal method that the rejuvenator can be timely supplied when the bitumens are aging. For the first time, this self-nourishing concept is mentioned in this work to describe this biomimetic behavior. It means that the rejuvenator can be controlled releasing into the aged bitumen. The microcrack can be avoided to be triggered at the greatest extent possible as the bituminous material keeps a softened state. The hollow fiberbased self-healing method is better for polymeric materials. Similar to the vasculature in human body, the hollow fibers forming networks in a material can also be used to supply healing agent to damages [15]. The crack propagation will definitely pass through the networks and trigger breaks of fibers. Then the contained liquid in hollow fibers flows out and heals the cracks. This process involves a corresponding mechanism of microencapsulated rejuvenator. However, vascular systems obviously have more advantages over microencapsulated rejuvenator-based systems. The vascular systems can ensure the release of rejuvenator into cracks, because the crack has a higher probability to pass through fiber network. Moreover, the vascular systems can supply large volumes of liquid agents continuously as it has a continuous pipe structure. In addition, more liquid may potentially be used for repeated selfhealing [18]. Besides the self-healing functions, the vascular systems can indirectly provide more functions, such as strength enhancement, structural monitoring, and thermal management.

Previously, various methods of preparing polymeric hollow fibers have been reported, including electrospinning, melt-spinning, dry-spinning, and wet-dry spinning. These electrospinning hollow fibers have many shortcomings, because of lower mechanical strength, harder controlling of size, lower heat-resistant grade, and less hollow liquid content. In addition, melt-spinning process is not suitable for the fabrication of hollow fibers containing liquid, limited by the formation technology. The high melt-spinning temperature will force the liquid to volatilize. On the other hand, it is hard to form microporous structure on the hollow fibers under such a high temperature. At present, many polymeric hollow fibers can also be made by dry-wet spinning using polystyrene, polyethylene, polyvinyl alcohol, poly(methyl

methacrylate), and PVDF [19]. Among these polymers, PVDF is widely utilized to form hollow fibers with relatively mature technologies because of its outstanding performances including higher thermal adaptability, high mechanical strength, and excellent hydrophobicity [19]. The most crucial is the PVDF that has been widely used to prepare hollow fibers owing special microporous structures as water ultrafiltration. For example, Bülow et al. [20] reported that water was filtered through the hollow fiber membrane. Water was penetrated the hollow fiber membrane transversely from the hollow fiber lumen by the internal pressure method, but inorganic salts and organic functional groups in the sewage are not permeable and are trapped, eventually resulting in water being purified. Li et al. [21] used polysulfone-silicone rubber composite membrane that is a polysulfone asymmetric hollow fiber as a base membrane, hollow fiber outer surface of the vacuum coating with a layer of dense silicone rubber for mixing hydrogen gas recovery. Adopting the above mature technology, we can fabricate PVDF hollow fibers with pore structures containing oily rejuvenator by a one-step spinning method. Through the designed pore structure, the rejuvenator can be slowly released from the hollow fiber achieving a self-nourishing result. In case a microcrack is generated, the tip stress of the microcrack will pierce the hollow fiber and allow oily rejuvenator to flow out rapidly. In this material system, there are some harsh requirements for hollow fiber materials due to asphalt road construction operations. Fortunately, PVDF has remarkable properties comparing to other polymer materials, such as excellent mechanical properties, high thermal stability, excellent chemical resistance, and high hydrophobicity to meet the above requirements. The melting point of PVDF is between 140°C and 170°C. According to asphalt mixing and construction temperature division, the usual hot mix asphalt temperature is 135°C-163°C. Therefore, the thermal stability of PVDF meets the requirements of asphalt. It is reported that the tensile strength of PVDF fibers is 29.4 MPa, while the highest shear strength of asphalt mixture is less than 3.0 MPa. PVDF fibers in the asphalt matrix can resist the stirring shear force. The hydrophobicity of PVDF is consistent with its surface tensions. For example, Liu et al. [19] reported a critical surface tension of PVDF membrane of 25-28.5 dynes/cm.

Self-nourishing concept is inspired by the biological phenomenon such as the cell nourishing. The bituminous materials will age during its service life. The selfnourishing bituminous materials are the bionic materials using hollow fibers supplying small molecules of rejuvenator spontaneously. The self-nourishing process is based on a penetration process of small molecules through the micropores on hollow fibers. When the bitumen is aging, the small molecules will continuously replenish into bitumen depending on the force of concentration difference. Based on the above inspiration, the aim of this work was to prepare and characterize a novel vascular self-nourishing and self-healing hollow fibers containing oily rejuvenator for aged bitumen. PVDF hollow fibers were fabricated by a one-set wet-dry spinning method directly. Pore structures were designed in hollow fibers to achieve the selfnourishing and self-healing behaviors. In one case, rejuvenator was controlled releasing during aging of bitumen by the force of concentration gradient. In another case, the microcrack could be healed by the rejuvenator released from the broken hollow fibers. The morphologies, size, and microstructure of hollow fibers were analyzed systemically. The mechanical properties and thermal stability of hollow fibers have also been investigated to ensure the reliability of these fibers in bitumen. Moreover, the penetration and diffusion behaviors were measured and modeled to understand the relationship between the microstructure and rejuvenator movements. All these results prove the practicability by using this novel vascular self-nourishing and self-healing hollow fibers containing oily rejuvenator for aged bitumen.

## 10.2 Preparation of hollow fibers as self-healing microvascular by a one-step spinning technology

PVDF resin was obtained from Solvay Advanced Polymers (6010#, Parachute, China). N,N-Dimethylacetimide (DMAC, 98%) was purchased from Samsung Fine Chemical Co., Ltd. (Ulsan, Korea). Oily rejuvenator was used as the self-nourishing and self-healing material (0.905 g/cm<sup>3</sup>, 4.240 Pa · s, Tianjin Chem. Co., China). An aged bitumen (40/50 penetration grade) was artificially prepared by a thin-film oven-test applying a bitumen product (80/100 penetration grade, softening point  $45^{\circ}$ C, Qilu Petrochemical Industries Co., Shandong Province, China).

The preparation of PVDF hollow fibers containing oily rejuvenator by one-step dried-wet method can be divided into two steps as illustrated in Fig. 10.1: (1) In the first step, PVDF powder was dried in an electric oven under 60°C for 24 h to remove the moisture completely. According to the experimental formula, a solvent of DMAC (400 g) was precisely weighed and poured into a flask equipped with a heater and a stirrer. The solid powder (100 g, dried PVDF) was poured into the three-necked round-bottomed flask in which the solvent DMAC was uniformly mixed, then the mouth of the three-round round bottom flask was immediately sealed, and then stirred in a constant temperature water bath at a temperature of 60°C for 8 h. The mixture was completely dissolved in a uniform casting solution; the casting solution was deformed for 24 h in a vacuum oven (Fig. 10.1A). (2) The casting solution was poured into a spinning pot with a maintained temperature at 60°C. Nitrogen gas (N<sub>2</sub>) with a high pressure (0.15 MPa) extruded the metered casting solution from the spinneret. The core fluid flowed through the rotor flowmeter under a pressure of 0.2 MPa, which entered the cavity of the hollow fiber from the central hollow of the spinneret and serves as a support (Fig. 10.1B). After leaving the spinneret, the casting solution moves through the air gap between the coagulation bath and the spinneret and then entered the external coagulation baths with tap water at  $25^{\circ}C \pm 1^{\circ}C$  (Fig. 10.1C and D). The solidified fibers were wound around the winding wheel with the help of a guide wheel. Finally, the ends of the fibers were sealed with a heat sealer to avoid release of core liquid. The fibers soaked in distilled water for 48 h to remove the impurities from the surface. One hollow fiber without containing materials was labeled as sample C1. Other hollow fibers containing oily rejuvenator were fabricated under the same conditions with PVDF contents of 20%, 30%, and 35%, which are labeled as samples of C2, C3, and C4, respectively.


**Figure 10.1** Illustration of hollow fibers preparation process, (A) the PVDF solution and rejuvenator in different containers, (B) the cross section of the mold for hollow fibers, (C) the formation mechanism of hollow fibers with pores in PVDF, and (D) the coagulation baths and winding.

# 10.3 Characterization methods of hollow fibers

# 10.3.1 Fourier-transform infrared microscopy

The chemical bands of hollow fibers, pure rejuvenation, and hollow fibers containing oily rejuvenation were measured by FTIR spectroscopy (PerkinElmer Spectrometer 100). FTIR spectra were recorded in the 400-4000 wavenumber range with a resolution of 4 cm<sup>-1</sup>.

# 10.3.2 Environmental scanning electron microscopy

The cross-sectional morphologies of fibers were observed by ESEM (Hitachi S4800, Japan). The hollow fiber was adhered to one side of the sample stage using a conductive double-sided tape without cracking. The inside or outside diameters of fibers were average values of 10 measurements. The cross-sectional surfaces of hollow fibers were sputtered with gold powder, which morphologies were observed at an accelerating voltage of 20 kV.

# 10.3.3 Mechanical properties of fibers

The tensile strength values of a hollow fiber (5.0 cm) was measured by using a mechanical testing instrument (Instron 5900, USA) with 50 kN load and a drawing rate of 20 mm/min at room temperature.

# 10.3.4 Thermal stability

The thermal stability of PVDF hollow fibers was measured by thermogravimetric analysis [TGA/differential thermogravimetric analysis (DTG), SDT-2960, Dupont, USA]. The scanning rate was  $10^{\circ}$ C/min under a nitrogen (N<sub>2</sub>) protection (50 mL/min).

# 10.3.5 Contact angle

A contact-angle testing system (EasyDrop, Kruss, Germany) was applied to measure the contact angles. One droplet of deionized water  $(2.0 \,\mu\text{L})$  was dropped on the surface of a single fiber. The images were captured by a camera connected to a computer, and the contact angle values were calculated automatically via a drop-shape analysis software.

# 10.3.6 Permeability of hollow fibers

The permeability of the hollow fibers was estimated using dead-end small-scale filtration equipment according to a reported method [22]. In detail, the whole filtration experiment was operated at room temperature  $(25^{\circ}C)$  and the osmotic pressure was 0.1 MPa. Prior to the permeability experiment, a dried hollow fiber was made into a component with three fibers. Epoxy resin was used to cover the two ends of this component. Therefore, water could only penetrate through the shells of the fibers. The calculated water permeability was based on the inner surface areas. The deionized water was enforced to infiltrate from inside to the outside through the cavities of hollow fibers. The permeability flux is calculated according to Eq. (10.1):

$$P = \frac{1}{A} \frac{dV}{dt} \tag{10.1}$$

where *P* is the permeability (L/( $m^2$  h), *A* is the area of cross section ( $m^2$ ), *V* is the volume of permeated water (L), and *t* is the permeation time (h).

# 10.4 Microstructure and properties of hollow fibers

# 10.4.1 Physicochemical structure of hollow fibers

A novel self-nourishing concept is proposed for the first time in this work. Inspired of the concept of capillary bionics, hollow fibers were designed with the capability



**Figure 10.2** Illustration of self-nourishing and self-healing hollow fibers containing rejuvenator in aged bitumen: (A) original bitumen material, (B) aged bitumen material, (C) self-nourishing process based on the penetrated rejuvenator out of hollow fibers, (D) self-healing process based on the microcrack healed by the released rejuvenator from break hollow fibers, and (D) the recovery bitumen sample.

of penetrating rejuvenator into an aged bituminous material. Fig. 10.2 illustrates the rejuvenator movements from hollow fibers into bitumen in detail. With the aging of bitumen (Fig. 10.2A and B), rejuvenator acts as a supplementary to restore the component proportion of asphaltene/maltene in bitumen. It reconstitutes the binder's chemical composition with a relatively higher ratio of maltene component [11]. When the bituminous material has been aged, the rejuvenator permeates out of hollow fibers derived by the force of gradient difference (Fig. 10.2C). The pore structure determines the permeation occasion and permeation rate. With a supplement of rejuvenator, the bituminous material has a steady stream of rejuvenator supplements. In another case, when microcracks are generated, the rejuvenator will also be released into bitumen with a similar self-healing approach of microencapsulate



**Figure 10.3** Photographs of hollow fibers: (A) the one-step spanning method of hollow fibers containing bituminous rejuvenator, (B) the spinneret of hollow fibers containing liquid rejuvenator, and (C) the long continuous hollow fibers with (left)/without (right) oily rejuvenator.

rejuvenator (Fig. 10.2D). Because hollow fibers can contain more rejuvenator than microcapsules, its self-healing efficiency (SHE) will be higher than that of micro-capsules [15,18]. Even though a hollow fiber does not break in crack propagation, the fiber will act as a mechanical strength-reinforced fiber. Under the above two mechanisms of action, the bitumen can maintain a longer service time without fracture (Fig. 10.2E).

Fig. 10.3A shows the spinning hollow fiber wrapping around the guide wheel. The continuous hollow fiber implies that the hollow fiber containing rejuvenator has been successfully prepared by the one-step wet-spinning method using PVDF resin. The spinneret can be used to control the size of diameter and thickness of shell of hollow fiber, playing a significant role (Fig. 10.3B). Its outer diameter is 0.12 mm. Fig. 10.3C exhibits two types of successfully prepared hollow fibers. The left (black) one is hollow fibers containing oily rejuvenator, and the right (white) one is the hollow fibers without core material. It can be preliminarily deduced from the colors that the rejuvenator has been contained by hollow fibers using one-step



**Figure 10.4** ESEM cross-sectional morphologies of hollow fibers (C2): (A) the hollow fiber with smooth inside and outside surfaces and (B) the measurement of the external diameter and thickness of hollow fibers.

Sample	Inter diameter (µm)	Shell thickness (µm)	Core-shell ratio	Mean pore length (μm)	Mean pore size (µm)
C1	1307	103	12.8	19.0	5.1
C2 C3	850 625	240	3.5 2.8	51.6 46.0	5.8 8.6
C3 C4	900	207	4.3	32.0	3.8

 Table 10.1 Physical parameters of hollow fibers.

spinning process. Fig. 10.4 shows the typical ESEM cross-sectional morphologies of hollow fibers (C2). The contained rejuvenator has been washed way using alcohol. The smooth internal and external surfaces of hollow fibers can be observed directly (Fig. 10.4A). No damage or break has been found on fiber shell. It plays a strategic importance for the force distribution of fibers, when they are embedded in a matrix material. The external diameter and shell thickness rules are marked in Fig. 10.4B. The structures on both sides of the hollow fiber shell are symmetrical with the same morphology, and the pore diameter has the property of consistency.

Table 10.1 lists the physical parameters of hollow fiber samples (C1, C2, C3, and C4), including inter diameter, shell thickness, core-shell ratio, mean pore length, and mean pore size. First, the measurements show that the C1, C2, C3, and C4 fibers have the inter diameter values of 1307, 850, 605, and 900  $\mu$ m, respectively. Their shell thickness values are 103, 240, 224, and 207  $\mu$ m, respectively. The data indicates that the thickness of the hollow fiber shell obtained from the coagulation bath using oily rejuvenator bath is larger than that of the hollow fiber shell using water bath. For the rejuvenator bath fibers, C2 has the maximum value of 240  $\mu$ m and C1 has the minimum value of 103. Moreover, the thickness of the hollow fibers decreases with the growing content of PVDF in these fibers (C2, C3, and C4). Second, the hollow fibers are assumed to be absolutely filled with oily

rejuvenator without blank space. The core-shell ratio is defined as the weight ratio of contained material and shell material of hollow fibers, which is a parameter indicating the containing capability of core material in hollow fibers. The core-shell ratios are 12.8, 3.5, 2.8, and 4.3 for C1, C2, C3, and C4, respectively. A larger core-shell ratio value means more rejuvenator has been contained under the same fabrication process. In addition, the hollow fibers with a larger core-shell ratio may release more rejuvenator with higher capability of self-nourishing and self-healing. Third, the mean pore length and mean pore size are both parameters reflecting the asymmetrical porous structure in shells of hollow fibers. Because the shell thickness values decrease with the increasing PVDF contents the length and aperture of the finger-like pores decrease at the same time. It means that the pore structure can be regulated by controlling the contents of hollow fibers. For example, asymmetric hollow fibers were prepared to enhance the increase the permeation rate of water through ethanol as an internal coagulation bath, resulting in a membrane without skin structure in internal surface of hollow fiber shell [23].

FTIR spectra were analyzed to determine the chemical structure of substances of hollow fibers (Fig. 10.5). The oily rejuvenator applied in this work is a mixture of *n*-alkanes. Spectrum (a) represents its FTIR spectra with four obvious absorption peaks at 749, 1377, 2854, and 2918 cm<sup>-1</sup>. C–F bonds in PVDF display peaks



Figure 10.5 FTIR spectra of hollow fiber materials: (a) the rejuvenator, (b-d) 20%, 30%, and 35% PVDF content hollow fibers with rejuvenator, and (e) 20% PVDF hollow fibers without rejuvenator.

at 1402, 1273, 840, and 763 cm<sup>-1</sup> [spectrum (b)]. Rejuvenator has the  $-CH_2$  asymmetric stretching vibration absorption peak (2854 cm<sup>-1</sup>) and the  $-CH_3$  asymmetric stretching vibration (2918 cm<sup>-1</sup>). The special peaks do not appear in PVDF spectra. Therefore, they can be chosen as a main criterion to discriminate the existence of oily rejuvenator in PVDF hollow fibers. FTIR spectra (b–d) belong to the hollow fibers (C2, C3, and C4) with PVDF content of 20%, 30%, 35%, respectively. The original containing rejuvenator was washed away by alcohol. By comparing the FTIR spectra of hollow fibers, it can be demonstrated that the oily rejuvenator still existed in PVDF hollow fibers. It implies that the rejuvenator molecules do not completely removed because they have penetrated inside of the PVDF shells through the porous structure. Spectrum (e) shows the characteristic peaks of the hollow fibers (C1, 20% PVDF) without rejuvenator. Apparently, it does not have the special bands of rejuvenator.

#### 10.4.2 Microstructure of hollow fibers

Fig. 10.6 shows ESEM cross-sectional morphologies of different hollow fiber locations. The cross section has a rough ring shape (Fig. 10.6A). There are many similar elongated gullies or voids on the surface of this ring, which are perpendicular to both inner and outer surfaces. More details can be observed in an enlarged image (Fig. 10.6B). The cross-section zone is divided into three layers with typical asymmetric structures distinguished by color areas, namely the dense skin layer (red color), the microporous layer (yellow color), and the disordered aggregate pore layer (green color), respectively. The skin microstructure was formed based on the solvent (DMAC) in the phase conversion diffusing from the uniform casting



**Figure 10.6** ESEM cross-sectional morphologies of hollow fibers: (A) the overall outline of cross-section of hollow fiber, (B) local morphologies of hollow fiber shell material, and (C) enlarged finger pore of the hollow fiber shell materials.



Figure 10.7 Strain-stress curves of various hollow fibers.

solution into the outer coagulation solution (the nonsolvent water). Meanwhile, a double diffusion below the skin layer occurred, in which the water diffused into the casting solution in the opposite direction of the DMAC solvent, promoting the micropore layer growth. The penetration of oily rejuvenator would be enhanced via the formation of microporosity. Oily rejuvenator (referred to as soft nonsolvent) acts as an internal coagulation bath in the formation of aggregate pore layer comprised of almost identical spherical particles with a crystallization process. The crystallization has occurred prior to liquid-liquid demixing during the formation of fiber shells, which is similar to Young et al.'s [24] report about a spherulitic PVDF membrane structure prepared with 1-octanol (as soft nonsolvent) as a coagulation bath. The mean size of the micropore is about 9  $\mu$ m and the mean length of is 46.0  $\mu$ m (Fig. 10.7C). There are some wrinkles on the inner surface of the micropore.

#### 10.4.3 Tensile strength of fibers

The mechanical strength of hollow fibers was evaluated by a tensile strength test analysis. Fig. 10.7 shows the strain-stress curves of hollow fiber samples (C1, C2, C3, and C4). In general, the stress of hollow fiber is gradually enhanced with the increase of strain. However, the hollow fibers containing oily rejuvenator (C2, C3, and C4) present tremendously lower mechanical stress than that of hollow fiber without rejuvenator (C1). In addition, the strain value shrunk from 341.2% (C1) to 36.9% (C2). It has been reported that the PVDF flat membranes prepared by DMAC solvent had a tensile strength of 13.2 N and an elongation at break of 4.72% [25]. Hollow fiber samples (C2, C3, and C4) have the tensile strain values of 0.034, 0.039, and 0.076 MPa, respectively, which are slightly improved with

increase of PVDF concentration. Hwang et al. [26] found that the PVDF hollow fiber had a relative higher tensile strength of 14.8 MPa/cm<sup>2</sup>. The lower tensile strength of hollow fibers may be more suitable to be embedded in bitumen. The fibers can be punctured easily when microcracks are generated or extended, leading to a quick release of oily rejuvenator [18].

## 10.4.4 Thermal stability of fibers

Fig. 10.8A shows the TGA curves of pure rejuvenator and hollow fiber samples (C1, C2, C3, and C4). It can be seen that the oily rejuvenator decomposed rapidly from 306°C to 387°C. At last, only about 0.08% mass has been remained. While the C1 hollow fiber begins to lose weight at 63°C (only 1.34 % of original mass lost), it belongs to the evaporation of the solvents (DMAC) in the hollow fiber. Then the C1 sample starts to lose weight sharply at 453.2°C, and finally about 26% of the original mass still remains at about 700°C. When the temperature increases to 900°C, the PVDF begins to decompose. For the hollow fiber samples (C2, C3, and C4), their TGA curves have a two-step decomposition process because of the continuing oily rejuvenator. With the increasing of temperature, the rejuvenator in hollow fibers enveloped firstly. For C1, C2, and C3, their first-step descending curves are similar to descending curve of pure oily rejuvenation, and their second descending curves are fitting the trend of C1 curve. It proves from another angle



**Figure 10.8** TGA/DTG curves of hollow fibers (C1, C2, C3, and C4): (A) TGA curves and (B) DTG curves.



**Figure 10.9** ESEM morphologies of hollow fibers (C2)/bituminous material composites treated with various temperatures: (A) 180°C, (B) 200°C, (C) 220°C, (D) 240°C, and (E) 260°C.

that oily rejuvenator has been holding successfully in hollow fibers. Fig. 10.8B shows the DTG curves of above hollow fiber samples (C1, C2, C3, and C4), and the losing weight rates of different hollow fibers. Under the high temperature of  $400^{\circ}$ C, C4 sample loses weight faster than C1, C2, and C3. C1 has the lowest losing weight rate because no rejuvenator evaporated firstly. In addition, it can be found that C2 sample envelopes its oily rejuvenator rapidly than C3 and C4. The reason can be attributed to its larger mean pore length, which allows rejuvenator to evaporate smoothly with less resistance.

Besides the TGA/DTG tests, the ESEM cross-sectional morphologies of hollow fibers/bituminous material composites were observed under various temperatures to estimate the temperature tolerance grades of hollow fibers (Fig. 10.9). As shown in Fig. 10.9A, several C1 fibers have been mixed in a bituminous material. The fibers still exists in matrix with a hollow structure; the shell structure of fibers are not destroyed under temperature of 180°C. The green color marks the bituminous material zone, and the yellow color marks the shell of fiber. No interface debonding is found, which indicates that the hollow fiber has a tight adhesion with bituminous material. Fig. 10.9B–E presents the ESEM cross-sectional morphologies of hollow fibers/bituminous material composites under 200°C, 220°C, 240°C, and 260°C, respectively. With the increase of temperature, the fibers still keep the integrity and stability in bituminous material. At the same time, the interface structures are not damaged. These conclusions imply that the hollow fibers can survive under the application temperature of bituminous material without a premature damage or break.

### 10.4.5 Contact angle of fibers

The hydrophilicity and hydrophobicity of hollow fibers are important for the selfnourishing because it is the force of rejuvenator penetration from inside to outside



**Figure 10.10** Contact angles of water on hollow fiber surfaces: (A) water contact angles and (B) oily rejuvenator contact angles.

of PVDF material. The self-nourishing process is based on the penetration of oily rejuvenator molecules through the micropores on hollow fibers. The penetration driving force is the concentration difference of rejuvenator molecules between inside and outside of hollow fibers. Rejuvenator is a lipophilic substance. Therefore, the PVDF hollow fibers must also have an oil-lipophilic property, ensuring that the rejuvenator molecules pass through the micropores. The contact angle of water (Fig. 10.10A) and oily rejuvenator (Fig. 10.10B) were measured on the outer surface of fiber samples (C1, C2, C3, and C4). Both C2 (84.5 degrees) and C3 (90 degrees) have a larger water contact angle values than C1 (67.9 degrees) (Fig. 10.10A). Although the water contact angle of C4 (87.6 degrees) has a little decrease than that of C2 and C3, it is still larger than C1. It indicates that the containing rejuvenator has significantly enhanced the hydrophobicity of the hollow fibers. The reason can be attributed to the oily rejuvenator remaining on the surface penetrating from inside hollow fibers. In Fig. 10.10B, the fiber samples (C1, C2, C3, and C4) have the oily rejuvenator contact angle values of 42.2, 20.6, 13.9, and 12.5 degrees, respectively. Pure PVDF (C1) has a characteristic of lipophilicity. The existence of rejuvenator also greatly improved the lipophilicity for C2, C3, and C4. This distinguishing feature also supports the idea that the oily rejuvenator can smoothly penetrate into aged bitumen and complete the self-nourishing process automatically.

#### 10.4.6 Rejuvenator penetration behaviors

In order to confirm the penetration behaviors of rejuvenator, the cross-sectional microstructure of hollow fibers were observed at various penetration times. Fig. 10.11A-C shows the ESEM cross-sectional morphologies of hollow fibers (C3) after rejuvenator penetration at days 0, 2, and 4. At the original state, the hollow fiber has a complete channel structure without fillings (Fig. 10.11A). After 2 and 4 days of penetration, the channel structure can be found with filled with



**Figure 10.11** ESEM cross-sectional morphologies of hollow fibers (C3) during rejuvenator penetration: (A-C) the hollow fibers after rejuvenator penetration at day 0, day 2, and day 4 and (D) a typical amplified cross-sectional morphologies of hollow fiber after rejuvenator penetration at day 6.

rejuvenator (Fig. 10.11B and C). The rejuvenator even can be identified overflowing from the cross section of hollow fiber (Fig. 10.11C). Fig. 10.11D shows a typical amplified cross-sectional morphology of hollow fiber after 6 days of rejuvenator penetration. More details about the rejuvenator penetration can be seen from the microstructure of the hollow fiber. After a period of penetration, oily rejuvenator passed through the holes of the hollow fibers. Some rejuvenator still remains in hollow fiber during penetration. All the above microstructure information implies that the rejuvenator can successfully penetrate the hollow fibers based on the forces of the hydrophobicity.

Fig. 10.12A displays the water permeation-time data of hollow fibers (C1, C2, C3, and C4), influenced by PVDF concentration. The reason for using water is that it can pass easily through the pore structure. The results can be applied to evaluate conveniently the microstructure of hollow fibers. The hollow fibers (C2, C3, and C4) all have a linear growth trend of penetration volumes as the extension of time. The slopes of C4 and C2 are greater than that of C3, indicating that permeation rate of C3 is slower than that of C4 and C2. The same result is also shown in Fig. 10.12B. The values of C2 and C4 permeate fluxes are 5.67 and 5.13 L/(m<sup>2</sup> · h), which are both larger than the value of C3 permeate flux (2.96 L/(m<sup>2</sup> · h)). Interestingly, no permeation behaviors have been observed in C1 under the same osmotic pressure. Because both inner and outer surfaces of the hollow fiber coagulated in water baths, a dense layer is formed, preventing the pass-through of water. It can be noted that the self-nourishing must be slow with a continuous addition of rejuvenator molecules into the aged bituminous material. In contrast, the C3 sample shows the best results.



**Figure 10.12** Penetration behaviors of hollow fibers: (A) time-dependent total volume of permeated water and (B) the permeate flux of hollow fibers for various PVDF concentrations.

# 10.5 Diffusing behavior of rejuvenator in bitumen

A thin bituminous material (40/50) film was spread on a slide glass with a thickness of about 1.0 mm. A hollow fiber was buried in the middle of this bitumen film. The rejuvenator diffusion behaviors into the aged bituminous film were studied by using a fluorescence microscope (Olympus BX41-12P02, Japan).

A modified attenuated total reflection Fourier-transform infrared spectroscopy (FTIR-ATR) method was applied to measure the diffusion behaviors of oily rejuvenator in a bitumen sample. The testing sample consisted of a bitumen layer (thickness, <2 mm) and single horizontally placed hollow fiber (Fig. 10.13B). The above sample was tightly pasted on top of a ATR prism (zinc selenide) (Fig. 10.13B). To keep a uniform temperature of this sample, an insulating cap was used. About 0.01 wt.% diphenyl sulfone (DPS) was mixed into rejuvenator. An absorption band (843 cm<sup>-1</sup>) (Fig. 10.2C) was specified as an index marker band to analyze the *D* values [7]. First, the total IR light (internal reflectance) was exploited by the nonabsorbing ATR prism. The measurements were carried out at various times under the same condition. The absorbance at 843 cm<sup>-1</sup> was calculated using a logarithmic time scale of the integrated peak area (Fig. 10.13D). Eq. (10.2) shows the Fick's law, which is generally applied to describe the *D* values,

$$\frac{\partial c}{\partial t} = D \cdot \frac{\partial^2 c}{\partial p^2} \tag{10.2}$$

$$D_T = D_0 e^{(s_1/T) + s_2} \tag{10.3}$$

where t is the time, p is the position, and c is the concentration.  $D_T$  is described using  $D_0$  and temperature T in Eq. (10.3), where  $s_1$  and  $s_2$  are constants.



**Figure 10.13** The illustration of FTIR-ATR method testing the diffusion behavior of rejuvenator in bitumen: (A) a cross section of test sample of hollow fiber/bitumen, (B) the FTIR-ATR testing prism, (C) the fairly specific absorption band for DPS at 843 cm<sup>-1</sup> selected as a marker band to calculate the diffusion coefficient, the integrated peak area determined as absorbance, and (D) a logarithmic scale curve of time-absorbance, the sloping line in the horizontal direction determined by the diffusion coefficient.

Both self-nourishing and self-healing processes in this material system greatly depend on the oily rejuvenator diffusion behaviors in aged bitumen. In order to measure the diffusion behaviors, it is necessary to first understand the fiber state or fiber distribution in bitumen. Hollow fibers containing rejuvenator (C3, 2.0 cm) were mixed into an aged bituminous material (40/50 penetration grade), forming a cylindrical fiber/matrix composite (Fig. 10.14A). Liquid N<sub>2</sub> was dropped on the top of this composite sample to trigger a crack. The cross section of break fibers can be clearly seen in the fracture surfaces of this composite sample, indicated by arrows (Fig. 10.14B). The fibers dispersed in bitumen separately without accumulation or entanglement phenomenon. Both vertical and horizontal fibers are appeared in the fracture surfaces. In an amplified image of another composite sample (Fig. 10.14C), we can even figure out the break details of fibers in the noted circles. Moreover, the bituminous sample is considered as isotropic material because of the random distribution of the fibers. The length of fibers will apparently determine this distribution. Too short length will decrease the mass of rejuvenator and too long length will cause the entanglement of the fibers. The appropriate length will be optimized in future works.

The rejuvenator diffusion behaviors can be directly observed using a fluorescence microscope. The green color in fluorescence microscopy images can help us



**Figure 10.14** Photographs of bitumen/hollow fibers (C3, 2.0 cm) composites: (A) a composite sample fractured by liquid nitrogen, (B) the break hollow fibers in interfaces of the crack, and (C) a magnified photograph of a composite sample with isotropic distributing hollow fibers.

to describe the diffusion zone of oily rejuvenator in bitumen [14]. In Fig. 10.15A, the rejuvenator can been seen flowing out from one end of a hollow fiber. This method has been proved to be effective in previous works of self-healing bitumen using microcapsules containing rejuvenator [25]. After sealed two ends, a segment hollow fiber with a length of 2.0 cm was buried in the middle of an bituminous material spreading on a slide glass (Fig. 10.15B). The thickness of this bituminous material film was about 1.0 mm, which met the requirements of observing the rejuvenator movements. The diffusion observation was carried out in a 40/50 penetration grade bituminous material. After 60 days, only about 100  $\mu$ m width rejuvenator area was formed around the hollow fiber (Fig. 10.15C). Even though



**Figure 10.15** Fluorescence microscopy morphologies of one fiber in bitumen on a glass sheet: (A) the rejuvenator flowing out from one end of the fiber, (B) the sealed ends fibers in bitumen on a glass sheet, (C) the fiber in bituminous material (80/100) for 60 days, (D-I) the fiber in bituminous material (40/50) for 1 day, 2 days, 3 days, 4 days, 5 days, and 6 days, respectively, and (J) the fiber in bituminous material (40/50) for 10 days.

the rejuvenator has the penetration capability through the hollow fiber, it cannot large-scale diffuse in large scale unaged bitumen without concentration gradient. The concentration gradient is the force to drive the movement of rejuvenator molecule in bitumen. The process known as diffusion means that molecules have a tendency to move away from areas where there are high concentrations and to move toward less concentrated regions. Therefore, no significant diffusion behavior can be found without a larger concentration gradient. Fig. 10.15D-J shows diffusion behaviors of a hollow fiber in an aged bituminous material (40/50) for 1-6 days. From the areas and shades of green color around the fiber, the diffusion can be proved definitely. Especially in Fig. 10.15G and H, a discontinuous phenomenon of rejuvenator in hollow fiber appears after several days, which confirms the penetration and diffusion behaviors of rejuvenator. Due to the inhomogeneity of the material, the diffusion area does not have a uniform green color. Wavy diffusion areas can be found because of the various speeds of rejuvenator at different points. As shown in Fig. 10.15J, the green color is extremely uniform after 10 days of diffusion. The small molecules will soften the aged bituminous material and recover the original property of the aged bituminous material. At the same time, the softened bituminous material has a higher self-healing capability by viscous fluidity and prevents further propagation of microcracks.

The self-nourishing and self-healing processes in vascular system are a periodic continuous movement of liquid rejuvenator, including steps of penetration, release, capillary, and diffusion. Diffusion coefficient (D) is defined as a ratio between the molar flux and the gradient of concentration. Diffusivity may fit Fick's law or other equations involving physical and chemical parameters. In this study, the microstructure of hollow fibers and temperature are considered as two main factors influencing the D values. It must be mentioned that the aging degree of bitumen greatly influences the diffusion behaviors of rejuvenator [14]. In order to simplify the complexity, only one type of aged bituminous material (40/50) was selected as a diffusion matrix to test the basic diffusion phenomenon and rules. The preliminary measurements will give a broad range of values, which will greatly help us to understand the movement rules of rejuvenator molecules in bitumen. At the same time, these values are guides for designing and optimizing the microstructure of hollow fibers in future works. It is critical factor to evaluate the feasibility of rejuvenator in aged bitumen. Because of the rejuvenator containing in hollow fibers, it is difficult to estimate the diffusion phenomenon inside of bitumen by usual methods. In this study, FTIR-ATR approach [27] was adopted to measure the D values of rejuvenator in the aged bituminous material. A nonabsorbing prism was used to reflect infrared light, and the total internal infrared light was detected. The DPS marker band (843 cm<sup>-1</sup>) was used to calculate the D values through the integrated peak area.

Fig. 10.16 shows the D values of rejuvenator from hollow fibers (C1, C2, and C3) in a 40/50 bituminous material under the temperature range of  $-20^{\circ}$ C to  $40^{\circ}$ C. This temperature range is the average temperature in the north of China. In the above fiber/matrix composite system, the D values are definitely affected by three factors: the pore size of hollow fibers, the inner size of hollow fibers, and the temperature. These hollow fiber samples (C1, C2, and C3) have different pore sizes of 5.8, 8.6, and 3.8  $\mu$ m, and their inner sizes are 850, 625, and 900  $\mu$ m, respectively (Table 10.1). Under the temperature of zero degree, nearly no diffusion behaviors were tested. One reason is that the oily rejuvenator has a solidified solid, high viscosity limits the movement of molecules. Another reason is that the molecules block the movement channels in fibers. The D values for each sample show a sharp increase with increasing temperature. This trend is consistent with the rule of selfhealing bitumen using microencapsulated rejuvenator. Higher temperature supplies more energy for movements of rejuvenator molecules. C3 has the largest D value under the same temperature. In the same aged degree bituminous material under the same temperature, this result is only attributed to the pore structure on hollow fibers. In other words, the diffusion behaviors are determined by the total crosssectional areas of penetrating channels  $(P_A)$  on hollow fibers, which is calculated by multiplying the number of pores per unit area  $(N_{pore})$  and the mean pore size  $(S_{\text{pore}})$  as given in Eq. (10.4), where  $\sigma$  is a parameter.

$$P_A = \sigma N_{\text{pore}} \cdot S_{\text{pore}} \tag{10.4}$$



**Figure 10.16** Diffusion coefficient of rejuvenator from hollow fibers (C1, C2, and C3) in the bituminous material under the temperature range of  $20^{\circ}\text{C}-40^{\circ}\text{C}$ .

Not similar to the microencapsulated self-healing bitumen results with a linear growth trend reported in previous works, the D values for each sample has a nonlinear exponential relation. Usually, Fick's law can be used to mathematically describe the diffusion behaviors and this simplified theory can describe many diffusion behaviors with less influence of chemical structure. However, the vascular self-nourishing and self-healing hollow fibers containing oily rejuvenator have more complex diffusion process. Rejuvenator must penetrate the hollow fibers and then diffuse into an aged bituminous material. Therefore, the D values are controlled by both processes of penetration and diffusion. A preliminary curve fitting has been given as shown in Fig. 10.15, and a well-fitted mathematical model is shown in Eq. (10.5).

$$D = ae^{-e^{(b(T-T_c))}}$$
(10.5)

where *a*, *b*, and *c* are constants. Comparing to Fick's law, this model is similar to a double superposition of diffusions. Although this rule may not be an accurate calculation, it still be a guide to design the microstructure of hollow fibers containing rejuvenator in our future works. Moreover, the relationship between  $P_A$  and D will be analyzed to control the speeds of self-nourishing and self-healing behaviors.

Fibers has been applied in bitumen to form composites. Najd et al. [28] believed that it was not necessary to add more bitumen to the asphalt mixture when it is added to the glass fibers because of the fiber's ability to adsorb the bitumen. In addition, with the addition of glass fiber mixture, the strength of asphalt mixture have been effectively improved. Song [29] reported the work about using the

polyester fiber-composite to modify asphalt mixture research, in which it showed that the asphalt mixture added polyester fiber had enhanced water-resistant ability and low-temperature cracking resistance. However, the above study showed that these fibers are solid fibers, but only as a reinforcing material in order to enhance the mechanical strength of asphalt mixture. However, it is impossible to repair the crack generated by the aged asphalt matrix material. The feature of this study was to prepare hollow fibers containing oily rejuvenator that can both pierce the hollow fiber and release the oily rejuvenator when it is needed, as well as possible fiber reinforcement. In addition, hollow fibers were infiltrated from oily rejuvenator to repair aged asphalt matrix materials and to extend the service time of asphalt pavements.

# 10.6 State of hollow fibers in bitumen

Comparing to the self-healing system of microencapsulated rejuvenator, the vascular self-healing system obviously possesses more advantages [20-22]. One advantage is that the vascular self-healing can ensure the release behavior of the rejuvenator from hollow fibers into cracks, as the crack has definitely passed through the fiber network in asphalt binders. Another advantage is that the vascular self-healing system can supply enough volume of liquid rejuvenator continuously into aged bitumen as hollow fibers have a continuous pipe structure with larger volume. It was found that enough liquid rejuvenator could even initiate repeated self-healing process [17,22].

The technology of microencapsulated rejuvenator has attracted widespread attention from researchers in the self-healing asphalt field. Microencapsulated rejuvenator product has been applied in real application as a commercial product [15-18,21]. At present, the vascular self-healing for asphalt using hollow fibers containing rejuvenator is still in its embryonic form of research [22]. Although hollow fibers have been prepared satisfying the application conditions in bitumen, more research contents are waiting for an intensive study in the future as shown in Fig. 10.17. Before a real application of vascular self-healing in asphalt, hollow fibers should be mixed in pure bitumen to inspect the states of hollow fibers in bitumen, including the integrality, distribution, shape, thermal stability, interface stability, and break-release behaviors. Obviously, it is a premise to next steps of research, considering the self-healing behavior and the real application in asphalt. The in-depth understanding of the hollow fibers states in bitumen will, at the same time, supplies more information about the relationship between fibers and matrix material. It will in turn guide the optimization of hollow fiber's preparation.

The long fibers were cut to short ones with a length of 3.0 cm. Both ends of a short one were sealed by hot-sealing scissors. The temperature of hot-sealing scissors is 70°C. The hollow fiber can be sealed rapidly and easily without leakage of oily rejuvenator. Then they were regularly arranged in a cuboid silicone mold without lid. The molten bitumen was poured into the mold repeatedly to obtain a bituminous composite material with multi-layer uniformly distributed hollow fibers.



Figure 10.17 The research road-map of hollow fibers for self-healing asphalt and the issues requiring attention in the states of hollow fibers in bitumen.

The sample of hollow fiber containing oily rejuvenator was cut to short ones (1 mm), which were observed by microscopy (BX43, Olympus, Japan) with a digital camera (DP26, Olympus, Japan) and the obtained images were copied with cell Sens digital imaging software (Olympus 1.9, Japan).

Cylindrical hollow fiber/bitumen composite samples (diameter 1 cm, height 2 cm) were placed in a high-temperature vacuum oven defoaming for 1 h and cooled to room temperature. X-ray computed tomography (micro-XCT) was used to obtain two-dimensional images of the samples. Moreover, the two-dimensional images of the samples were reconstructed to form three-dimensional (3D) XCT images using 3D-modeling analysis software (Avizo 9.0).

An repetitive heating-cooling test was designed to measure the resistance of hollow fibers in a violent temperature changes. A single hollow fiber containing oily rejuvenator was put on a slide glass coating with a thin layer of pure bitumen. Then, it was placed in a  $180^{\circ}$ C electric heating blast oven for 0.5 h. The sample was then removed and left to cool at room temperature for 1.5 h. The above heating-cooling operation was repeated 20 cycles.

Pure bitumen was set in an electric heating blast oven and heated to  $100^{\circ}$ C for about 10 min. Then, a small amount of pure bitumen was poured into a cuboid silicone mold without lid and some hollow fibers were laid, and then the above operation was repeated twice. Finally, the mold was cooled to room temperature under room temperature conditions and transferred to the refrigerator at a temperature of 0°C for 6 h. In that environment, the pure bitumen block sample was removed from the mold and the bitumen block was broken vertically in the middle of the bitumen block. The break point was observed to find the break and release behaviors of rejuvenator from hollow fiber.

A hollow fiber with a length of 3.0 cm was embedded in a thin aged bituminous film (40/50) with a thickness of 5.0 mm spreading on a glass sheet. Fluorescence microscopy (Olympus BX41-12P02, Japan) was used to observe the penetration process of oily rejuvenator. Images were taken at various times under temperature of  $0^{\circ}$ C to avoid the influence of the temperature sensitivity of bitumen.

### 10.6.1 Distribution and integrality of hollow fibers in bitumen

The distribution of hollow fibers plays an important role for self-healing in asphalt. It can be imaged that the size and length of hollow fibers will greatly influence their distribution in asphalt. Longer fibers may increase the difficulty of the asphalt mixture. At the same time, shorter fibers may contain insufficient amounts of rejuvenator, which will also greatly reduce the efficiency of self-healing behavior. Excessively short fibers (<2.0 mm) can be considered as microcapsules, which is not a real vascular self-healing hollow fiber. It means that the size and length of hollow fibers must be optimized to meet the structure and construction requirements of asphalt. However, the prerequisite for conducting this optimization study is to ensure that the hollow fibers can be distributed in pure bitumen without physical and chemical resistances. Thereby, the states of the distribution of self-healing hollow fiber in bitumen are measured by applying a XCT through an in-situ observation. XCT is a microscope that uses X-ray scanning to acquire the internal structure of a material. Some of the hollow fibers containing oily rejuvenator were placed in a plastic hollow cylindrical mold (diameter 1 cm, height 2 cm), and the molten pure bitumen was poured into the mold. Furthermore, it was placed in a hightemperature vacuum oven defaming for 1 h and finally cooled to room temperature before XCT test.

By comparing X-ray attenuation intensity before and after scanning, the contour information can be obtained, and the contour information is processed to obtain scanned images of the specimen. CT images reflect the extent of absorption of X-rays to the sample with different gray levels. Shadow indicates low absorption region, namely low-density areas; the white represents high absorbency zone, that is, high-density areas. Fig. 10.18 shows XCT images of hollow fibers distributing in bitumen with both vertical distribution and horizontal distribution states. In Fig. 10.18A, the cross section of hollow fibers in bitumen can be discerned clearly with a circular hole shape. Several circulars have a degree of deformation because of the extrusion and thermal action. Hollow fibers can distribute in bitumen homogeneously without entanglement and adhesion phenomena. Moreover, the shell structure and the micropore structure of hollow fibers can even be seen directly. Fig. 10.18B shows a XCT image of horizontal distribution of hollow fibers in bitumen. Due to the different positions of hollow fibers in bitumen sample, they display various lengths. From the longest one on the right side, it can be deduced that the hollow fiber has an integrality state in bitumen without rupture and decomposition.



**Figure 10.18** XCT slides of hollow fibers distributing in bitumen: (A) vertical distribution of hollow fibers and (B) horizontal distribution of hollow fibers.



**Figure 10.19** 3D-XCT reconstruction structures of hollow fibers distributing in bitumen: (A) vertical distribution of hollow fibers and (B) horizontal distribution of hollow fibers.

In order to further characterize the distribution and integrality of hollow fibers in bitumen, the XCT images were reconstructed to form 3D-XCT structures. The hollow fibers were rendered blue by the software of Avizo 9.0. Fig. 10.19A1-A3 show the 3D-XCT reconstruction structure of hollow fibers distributing in bitumen

with a vertical distribution state. These fibers distribute in bitumen without entanglement and adhesion in a relatively parallel state (Fig. 10.19A1 and A2). Each fiber has a certain angle of inclination. Especially in Fig. 10.19A3, it can be observed that all fibers have a continuous structure without break in a perfect integrality state. This conclusion is consistent with the above analysis in Fig. 10.6. Fig. 10.19B1–B3 shows the 3D-XCT reconstruction structure of horizontal distributed hollow fibers in bitumen. Various phases of composite material are differentiated and colored based on the principle of different gray values (result from different density of phases) under the X-ray [24]. Therefore, only a material with the same density can be selected. The deformation of hollow fibers is also detected in Fig. 10.19B1 because of the extrusion or thermal action. As the micropore structure in hollow fibers, the 3D-XCT reconstruction image (Fig. 10.19B2) appears as particle materials. In Fig. 10.19B3, the integrality is demonstrated again by the continuous 3D-XCT structure of hollow fibers.

#### 10.6.2 Thermal stability of hollow fibers in bitumen

Thermal stability is an extremely significant parameter for hollow fibers. The normal temperature of pure bitumen is maintained at about 180°C during the process of transportation, agitation, and construction [15]. Therefore, the hollow fibers must survive under this temperature. Fig. 10.20 shows the TGA/DTG curves of hollow fiber without oily rejuvenator. The TGA curve of PVDF hollow fiber has essentially no weight loss before 453.2°C. The fastest decomposition temperature is 463°C



Figure 10.20 TGA/DTG curves of PVDF hollow fiber without rejuvenator.

(the peak temperature corresponding to the DTG curve). At the temperature of 778.3°C, the PVDF hollow fibers do not lose weight anymore. The same result is also found in the DTG curve. The final residual weight of the PVDF hollow fiber is about 27.8% belonging to the carbide and fluoride materials.

The repeated heating-cooling test between  $25^{\circ}$ C and  $180^{\circ}$ C was conducted to analyze the durability and reliability of the hollow fiber in pure bitumen. Similar repeated heating-cooling test during  $25^{\circ}$ C $-180^{\circ}$ C was successfully applied to evaluate the thermal stability of microcapsules containing rejuvenator [14–16]. Fig. 10.21A1–A5) shows the images of hollow fibers containing rejuvenator in bitumen after various thermal treatment cycles (1, 2, 18, 19, and 20). Fig. 10.21B illustrates the 20 thermal treatment cycles; one cycle includes an increasingdecreasing temperature process in 1.5 h. Each cycle has a time interval of 1.5 h. The hollow fibers containing oily rejuvenator in bitumen still keep their shape without deformation and decomposition during high-temperature heat treatment. This result illustrates that the hollow fiber containing repair agent has excellent durability and good reliability.



**Figure 10.21** Thermal stability of hollow fibers containing rejuvenator in bitumen after 20 thermal treatment cycles, with each cycle including an increasing-decreasing temperature process for 1.5 h and every two cycles owing a time interval of 1.5 h between every two adjacent cycles:  $A_1-A_5$ : the images of hollow fibers in bitumen after 1, 2, 18, 19, and 20 cycles and (B) the increasing-decreasing temperature process during 20 cycles.

### 10.6.3 Interface stability of hollow fibers/bitumen composites

It is an important issue that the hollow fibers have a tight bonding with bituminous material. The debonding phenomenon should be avoided because it may greatly reduce the mechanical properties of composites [25]. In addition, the debonding will also interrupt the propagation of microcracks, and then the break of hollow fibers will not be triggered. That means the SHE may be seriously reduced. Usually, thermal action is the main factor causing debonding to fiber/polymer composites [26]. Therefore, a violent process of temperature change was designed to simulate the actual situation in this work. Fig. 10.22 shows the scanning electron microscopy (SEM) cross-sectional morphologies of hollow fibers/bitumen composites before and after 100 thermal treatment cycles. One cycle includes an increasing-decreasing temperature process in 1.5 h. Each cycle has a time interval of 1.5 h. It can be seen in the original state (Fig. 10.22A) that the PVDF hollow fibers have a good bonding structure with bitumen. The interface structure is tight without gaps. PVDF hollow fibers had a good bonding with epoxy because of the large number of hydrogen bonds of PVDF molecular chains [27]. Similarly, these hydrogen bonds on hollow fibers can form molecular linkages with bituminous



**Figure 10.22** SEM cross sectional morphologies of hollow fibers/bitumen composites: (A) the original interface morphology of the bitumen sample, (B) the bitumen sample after 100 thermal treatment cycles, one cycle including an increasing-decreasing temperature process in 1.5 h, every two cycles owing a time interval of 1.5 h between every two adjacent cycles, (C) an amplified interface morphology of a hollow fiber and bitumen after the thermal treatment.

molecules. This conclusion has also been found in the interface structure between bitumen and other polymers [28]. After the violent process of temperature change (Fig. 10.22B), no debonding appears between the hollow fibers and bitumen. Hollow fibers keep a complete structure without decomposition andrupture, which is consistent with the conclusion of thermal stability. Two hollow fibers in close position even do not have interaction, causing debonding or other microstructural changes. In a local magnified SEM morphology after the temperature change (Fig. 10.22C), the interface area of a hollow fiber and bitumen can be easily distinguished well. The red arrow points the interface boundary of hollow fiber/bitumen composite. No debonding appears under such a temperature treatment due to the tight molecular linkages, especially the abovementioned hydrogen bonds.

#### 10.6.4 Break and release behaviors of hollow fibers in bitumen

The vascular self-healing behaviors of bitumen are based on the released oily rejuvenator from hollow fibers. It means that the hollow fibers in bitumen should break when they encounter a crack. Therefore, the fibers can not be too strong or too resilient to resist the tip stress of cracks. Then oily rejuvenator can flow out without resistance. Fig. 10.23 shows the photographs of break interfaces of a cylindrical bitumen/hollow fibers sample (height 5 cm, diameter 3 cm). Liquid N<sub>2</sub> was dropped on the composite, and then a crack was triggered (Fig. 10.23). The composite sample was opened along this crack; the interface structure expressed more state details of hollow fibers in bitumen (Fig. 10.23B). A crack with wedge shape is observed in a red dotted frame. The break points of hollow fibers are dispersed in the bitumen



**Figure 10.23** Photographs of break interfaces of a cylindrical bitumen/hollow fibers sample (height 5 cm, diameter 3 cm): (A and B) the break interface of the sample, (C) an amplify zone of the crack with break hollow fibers, and (D) the rejuvenator droplet on the break interface spilling out from hollow fibers.

homogeneously. In Fig. 10.23C, the break points of hollow fibers can be seen more clearly in an amplify zone of this crack. The fracture points of hollow fibers are flat without burrs. It implies that the tip stress can easily cut through several hollow fibers. This phenomenon has also been reported in the self-healing bitumen using microcapsules containing rejuvenator [18]. Although it has been proved that the hollow fibers can be cut by the tip stress of crack, the propagation of crack still needs to be investigated in-depth. The length, width, and tip stress of propagation of crack should be considered to enhance the SHE. A model should also be developed in future work to predict the self-healing behaviors including the factors of temperature, the aging degree of bitumen, the content of hollow fibers, the mass of rejuvenator, the mechanical properties of hollow fibers, and arrangement mode of hollow fibers in bitumen. Another enlarged zone of blue-dotted frame (Fig. 10.11B) is shown in Fig. 10.23D with oily rejuvenator on the surface of this composite material. The rejuvenator droplets were spilled out from hollow fibers when the composite sample was opened along the crack. It indicates that the hollow fibers contain a sufficient amount of liquid material. The rejuvenator can smoothly flow out from the hollow fibers without a large resistance to flow.

It is also an important issue to observe the process of the rejuvenator outflow to understand its speed and form, which may finally determine the SHE of bitumen. The reason is that the self-healing speed is greatly determined by the amount of rejuvenator filling in the crack [17]. However, this observation is hard to perform because of the measurement difficulty of rejuvenator inside a bitumen sample without artificial destruction. To overcome this problem, a thin film of bitumen (5 mm) with hollow fibers was broken (Fig. 10.24A) and placed on a glass sheet to observe the outflow of rejuvenator. As the arrows pointing, the hollow fibers were parallel arranged in bitumen no more than two layers to ensure the outflow of rejuvenator from an exact end of a hollow fiber (Fig. 10.24B). The thin bituminous film was placed on a glass sheet and broken by liquid N2 immediately. A microscopy with a digital camera (Leica MD 2700M, USA) was used to capture the outflow images of liquid rejuvenator. Fig. 10.24C-H shows the outflow morphologies of rejuvenator from hollow fibers at various times of 2, 4, 6, 8, 10, and 12 min, respectively. At the beginning of a break for this sample (Fig. 10.24C-E), the rejuvenator can even spatter out quickly due to the full state of hollow fibers containing rejuvenator. After about 6 min, more rejuvenator accumulates at the exit of hollow fibers (Fig. 10.24F-H). Under the capillary action of 10 min, oily rejuvenator has a steady outflow from hollow fibers. It can be deduced that when the outflow is inside of aged bitumen, the outflow speed may more quickly attributing to because of the concentration difference of small molecules [2,17].

Besides the surface microcrack of bitumen, the microcrack may also appear inside the bitumen because of a pressure deformation or a shear deformation [27]. The temperature changes also can trigger inside microcracks especially in cold areas. Normally, the inside microcrack is hard to be observed. The inside microcrack need to be healed as soon as possible; otherwise, it may evolve into a larger crack for aged bitumen [17]. For the vascular self-healing system, the hollow fibers at the same time should be broken by the inside microcrack and release rejuvenator immediately. Therefore, it is essential to observe the states of hollow fibers suffered



**Figure 10.24** Photographs of break interface of a thin film of bitumen (5 mm) with hollow fibers composite sample: (A and B) the break points of the bitumen sample with parallel arranged hollow fibers, (C-H) the microscopy morphologies of outflow of rejuvenator from hollow fibers at various times of 2, 4, 6, 8, 10, and 12 min.

an inside fracture. A cylindrical shaped (diameter 1 cm) aged bitumen (40/50 penetration grade) sample under 0°C with hollow fibers was used to simulate the practical application. It was placed on a universal testing machine (YH-9000A, Yuhong Optoelectronic Equipment, Nanjing, China) suffering a pressure deformation (50 N) and a shear deformation (50 N) successively. This operation is arbitrary and designed to make an inside microcrack of bitumen sample. Fig. 10.25 shows the typical XCT images of hollow fibers in aged bitumen under 0°C with a vertical distribution (Fig. 10.25A) and horizontal distribution (Fig. 10.25A). In Fig. 10.25C, typical tearing failure points can be found obviously for two hollow fibers. The broken surface is not neat, which indicates that the fibers are not cut directly by a microcrack. The fibers have experienced a deformation rupture under the combined actions of external force and tip stress of microcrack. Another type of inside fracture of hollow fibers may be splitting under an external action as shown in Fig. 10.25D. Hollow fibers has neat fracture surface. The above observations imply that the hollow fibers can adapt to the external forces with a rapid stimulus



**Figure 10.25** XCT images of hollow fibers in aged bitumen under 0°C: (A) a bitumen sample with vertical distribution hollow fibers, (B) a bitumen sample with horizontal distribution hollow fibers, (C) the tearing failure points of hollow fibers, and (D) the transverse failure point of hollow fibers.

response, quickly triggering rupture and repairing the microcracks in time. There is no doubt that further research will need to be carried out about the relationship between the mechanical property of hollow fibers and external force. The knowledge will in turn guide the microstructure of hollow fibers to enhance the selfhealing capability of bitumen.

# 10.6.5 Penetration and diffusion behaviors of rejuvenator

In our previous work [22], a novel concept of "self-nourishing" has been at the first time mentioned to describe this biomimetic behavior of hollow fibers containing



**Figure 10.26** Movement tracks of oily rejuvenator out of hollow fibers: (A) the outflow of rejuvenator from a break point of hollow fiber, (B and C) the permeation of rejuvenator through the micropores on a hollow fiber, and (D) the diffusion behavior of rejuvenator into aged bitumen.

rejuvenator based on the micropores. In some cases, the self-healing may not occur while no cracks have been triggered during the aging process of bitumen. Predictably but disappointingly, the hollow fibers method is only remedial measure when cracks have already appeared [22]. Inspired by the skin nutrition concept of human body, it is a perfect idea that the oily rejuvenator can be timely supplied during the aging process of the bitumen. Similar to the vascular in human body, the hollow fibers forming networks in a bitumen material can also be used to supply rejuvenator molecules [20,22]. Based on our initial work of investigating the movement morphology of oily rejuvenator permeated in bitumen [17], more details were observed to describe the penetration behaviors of rejuvenator through the micropore on hollow fibers. The fluorescence microscopy images were used to analyze the diffusion behaviors of rejuvenator directly based on the green color of the diffusion zone [2,17]. Fig. 10.26A–D illustrates a typical whole penetration-diffusion process

of a hollow fibers in a thin aged bitumen film (40/50, thickness 5 mm). The ends of this hollow fiber were sealed by hot scissors. Some squeezed out oily rejuvenator is found in Fig. 10.26A, which means that the hollow fiber is full of rejuvenator. After 1 day, the penetration track is observed as the arrows pointing the movement direction of rejuvenator (Fig. 10.26B). After 5 days (Fig. 10.26C), more rejuvenator in red color enriches on one side of the hollow fiber. This phenomenon is consistent with the previous conclusion [22]. The rejuvenator then diffused into aged bitumen based on the gradient concentration difference between the marginal area of hollow fiber and the inside of aged bitumen. Interestingly, a gradient diffusion track of oily rejuvenator in aged bitumen is found in aged bitumen after 10 days. In Fig. 10.26D, the area in a bracket is a typical gradient diffusion zone. This gradient diffusion has an obvious hierarchical structure through distinguishing colors. The arrows point the direction of this gradient diffusion. After a period of time, the consumed rejuvenator recycled the aged bitumen. An empty area in hollow fiber in white color is found, belonging to the zone without rejuvenator.

# 10.7 Self-healing capability of bitumen using hollow fibers

Besides the self-healing realization methods, the testing methods are also important for bituminous material. Several self-healing evaluation methods have been summarized about the evaluation of self-healing capability for pure bitumen including dynamic shear rheometer method, beam on elastic foundation method, and direct tension testing method [15]. The dynamic shear rheometer method is carried out to test shear modulus using two pieces of bitumen together under a parallel plate, which is complexly affected by many factors [16,17]. With the advance of research, the self-healing bitumen can be considered as a healing process of a single open microcrack. The self-healing process is simplified as a recovery of mechanical property for bitumen [8,9]. The primary power of self-healing of bitumen is the viscoelastic character of bituminous molecule. The molecule diffusion and entanglement help the microcrack closure definitely [9]. The third method is designed based on the above consideration applying a beam on elastic foundation (BOEF) setup. A bitumen beam is able to control the self-healing process of a cracked surface autonomously including crack closure and crack-healing at various rest periods and temperatures [10,18]. In details, a notched bitumen beam is supported by a rubber foundation, which absorbs most of the deformation of the beam and closes the crack without a load [8]. Under a vertical pressure, the microcrack is triggered following the notch. Self-healing process occurs for a period of time under a certain temperature. Then the load strength is tested again for this bitumen beam. The self-healing capability is defined as the recovery degree of tensile loads. This method has been successfully applied to evaluate the SHE of bitumen/microcapsule composite samples [10].

However, the above BOEF method is not suitable for the microvascular selfhealing system of bitumen. It can be imaged that when a crack is generated, the fibers in the crack surface do not all break simultaneously. The consistent fibers will greatly influence the self-healing capability. Moreover, the oily rejuvenator cannot all flow out and reduce the efficiency the self-healing. It means that the test method must ensure a synchronous rapture in the crack during a mechanical experiment evaluation of the microvascular self-healing capability of bitumen applying hollow fibers with oily rejuvenator. Another repetitive tension test has been carried out to evaluate the multi-self-healing character of bituminous material mixing with microencapsulated rejuvenator [11]. The mechanical characteristics of the bitumen samples mixing with microcapsules were analyzed by a direct tensile test. The results indicated that the bitumen samples had a multirecovery capability, which led the aged bitumen to recover their original state with rejuvenation [11]. It was also discovered that self-healing capability of bitumen sample was ruined with more self-healing cycles. Microcracks need more time to be healed in a higher aged degree of bitumen. Moreover, temperature also has a significant influence on selfhealing capability of bituminous material. Interestingly, a bitumen sample does not recover to its original loading state after an immediate reloading because of its nature of viscoelasticity.

Inspired by the above repetitive tensile method, the objective of this work was to evaluate mechanically the microvascular SHE of bitumen mixing with PVDF hollow fibers with oily rejuvenator. These results have some limitations because pure bitumen binder does not exist in a real pavement. It is normally combined with filler and additives forming mastic. To reduce the complexity of the material structure, the self-healing behavior of pure bitumen sample was characterized by its recovery efficiency based on repetitive tensile tests. The self-healing mechanisms were investigated through observation of the morphologies of microstructure. A simple mechanism was explained based on the SHE considering four factors of fiber content, fiber direction, self-healing temperature, and self-healing time, which guide the control of fiber composite structure and the usage conditions of hollow fibers in bitumen.

#### 10.7.1 Vascular self-healing efficiency evaluation method

Fig. 10.27A shows the size and shape of the samples for tensile strength test. A bitumen sample is in the middle of two copper molds. The bitumen samples were fabricated in a silicone rubber mold, which geometry ensures the tensile direction in the center of the sample [19]. To keep both sides of one bitumen sample has the same shape, it was covered with another silicone rubber mold. The sample with molds was kepted in a chamber with temperature of 0°C for 24 h. Then the bitumen sample was obtained through a demold process. The tensile strength tests were carried out using a tensile machine with a sensor ( $\pm 2.5\%$  RH,  $\pm 1^{\circ}$ C, 0.0001 N; Aigu, NK-ZP 10-1000 N, Fuma Electric Equipment Co., Shenzhen, China) using a displacement speed of 100 mm/min at 0°C in a thermostat. Under a direct tension, the sample was ultimately fractured into two parts in middle. Then the two parts



**Figure 10.27** Illustration of the tension test method to evaluate the self-healing capability of bitumen with hollow fibers: (A) the original sample marked with size before a tension, (B) the sample with a crack after a tension, (C) the setup of tension in a thermostat, and (D) the data output and calculation.

were immediately spliced together. The spliced sample was once again heated for a period of 24 h under different temperatures of 0°C, 5°C, 10°C, 15°C, 20°C, 25°C, and 30°C. It was the self-healing process for the sample. This sample was moved into the chamber for a recondition with 24 h under 0°C (Fig. 10.27C). Another fracture process was repeated for the demolded bitumen sample under 0°C with a displacement speed of 100 mm/min. The sample suffered the above fracture-healing-refracture cycles repetitively, the computer recorded data automatically (Fig. 10.27D). A video camera on top of the thermostat recorded the image details during the tension tests. The SHE is calculated by using Eq. (10.6):

$$SHE = \frac{L_f}{L_0} \times 100\%$$
 (10.6)

where  $L_0$  is the original tension strength of bitumen sample and  $L_f$  is the tension strength of bitumen sample after *n* times healing cycles (f = 1, 2, 3...).

### 10.7.2 Distribution of hollow fibers in bitumen samples

The distribution of hollow fibers plays an important role for self-healing in asphalt. It can be imaged that the size and length of hollow fibers will greatly influence their distribution in asphalt. Longer fibers may increase the difficulty of asphalt mixture. At the same time, shorter fibers may contain insufficient amounts of rejuvenator, which will also greatly reduce the efficiency of self-healing behaviors. Excessively short fibers (<2.0 mm) can be considered as microcapsules, which is not a real vascular self-healing hollow fiber. It means that the size and length of hollow fibers must be optimized to meet the structure and construction requirements of asphalt.

However, the prerequisite for conducting this optimization study is to ensure that the hollow fibers can be distributed in pure bitumen without physical and chemical resistances. Thereby, the states of the distribution of self-healing hollow fiber in bitumen were measured by applying a XCT through an in-situ observation. XCT is a microscope that uses X-ray scanning to acquire the internal structure of a material. Some of the hollow fibers with oily rejuvenator were placed in a plastic hollow cylindrical mold (diameter 1 cm, height 2 cm), and the molten pure bitumen was poured into the mold. Furthermore, it was put into one high-temperature vacuum oven defaming for 1 h and finally cooled to room temperature before XCT test.

The contour information is obtained by comparing X-ray attenuation intensities. Shadow indicates the low-density areas with low absorption, the white represents the high-density areas with high absorbency. Then the contour information is helped to achieve CT scanning images of material reflecting the extent of absorption of X-rays with different gray levels. Fig. 10.28 shows XCT images of hollow fibers distributing in bitumen with both vertical distribution and horizontal distribution states. In Fig. 10.28A, the cross section of hollow fibers in bitumen can be discerned clearly with a circular hole shape. Several circulars have a deformation because of the extrusion and thermal action. Hollow fibers can distribute in bitumen homogeneously without entanglement and adhesion phenomena. Moreover, the shell structure and the micropore structure of hollow fibers can even been seen directly. Fig. 10.28B shows another XCT image of horizontal distribution of hollow fibers in bitumen. Due to the different positions of hollow fibers in bitumen sample, they have a various lengths. From the longest one on the right side, it can be deduced that the hollow fiber has a integrality state in bitumen without rapture and decomposition.

In order to further characterize the distribution and integrality of hollow fibers in bitumen, the XCT images were reconstructed to form 3D-XCT structures. The hollow fibers were rendered blue by a software of Avizo 9.0. Fig. 10.28C and D shows the 3D-XCT reconstruction structure of hollow fibers distributing in bitumen with a vertical distribution state. These fibers distribute in bitumen without entanglement and adhesion in a relative parallel state (Fig. 10.28C). Each fiber has a certain angle of inclination). Especially in Fig. 10.28D, it can be observed that all fibers have a continuous structure without break in a perfect integrality state. This conclusion is consistent with the above analysis.



**Figure 10.28** XCT results of hollow fibers in bitumen: (A and B) two typical XCT morphologies of hollow fibers in bitumen and (C and D) 3D-reconstruction morphologies of hollow fibers in bitumen.

# 10.7.3 Self-healing capability influenced by fiber contents

In this work, the SHE was calculated based on the repetitive tensile tests through the crack closure and crack-healing at various rest periods and temperatures. Fig. 10.29 shows more image details of this repetitive tensile method under a low temperature (0°C). A bitumen sample has a elongation under a tension strength (Fig. 10.29A1) and then it breaks beyond the tensile limit state (Fig. 10.29A2). The break point is in the middle of this sample (Fig. 10.29B1) and the break interface is smooth under low temperature (0°C) (Fig. 10.29B2). To test the SHE, the two parts of the broken sample are spliced together for a period of time under a set temperature (Fig. 10.29B3). After a healing process, it is stretched again to a break state (Fig. 10.29B4). The whole tension-healing-tension process can be repeated several times to evaluate the multiself-healing capability of bitumen samples [19].



**Figure 10.29** Photographs of the bitumen samples with/without hollow fibers containing rejuvenator during the tension tests under a low temperature  $(0^{\circ}C)$ ,  $(A_1)$  a bitumen sample during a original tension with an elongation,  $(A_2)$  a tension break of this bitumen sample,  $(A_1)$  the break point of a bitumen sample,  $(B_2)$  the break interface of this bitumen sample,  $(B_3)$  the two parts of the break sample spliced together for a self-healing process,  $(B_3)$  the healed sample suffered a second tension,  $(C_1 \text{ and } C_2)$  the bitumen with one fiber,  $(D_1 \text{ and } D_2)$  the bitumen with one fiber, and  $(E_1 \text{ and } E_2)$  the bitumen with one fiber.

As a fiber composite, the fibers contents in a bitumen sample definitely influence the break strength and SHE values. In order to understand the self-healing behaviors of bitumen with different fiber numbers [one fiber (Fig.  $10.29C_1$  and  $C_2$ ), two fibers (Fig.  $10.29D_1$  and  $D_2$ ), and three fibers (Fig.  $10.29E_1$  and  $E_2$ ), several samples were made and tested their self-healing capability. All fibers are arranged parallel to the stretching direction. It was found that the all the fibers in a bitumen sample broke at the same time with the break of matrix material. No fiber was pulled out from bitumen material. The fracture interface of bitumen has a relatively flat structure under low temperature.

Cracking is one of the main distresses responsible for the service life reduction of asphalt pavements. Therefore, it is important to for the service life prediction, it is important to understand the crack healing behaviors. The fracture-healingrefracture tests in this work is helpful to investigate the self-healing capability during a loading-healing-reloading process. Su et al. [11] has reported that the selfhealing behaviors are greatly dependent on the healing time, temperature, duration


Figure 10.30 Tensile load-time curves of bitumen samples with one fiber or without hollow fiber under  $0^{\circ}$ C.

of healing, crack phase, material modifications, and aging degree of bitumen. Normally, two procedures are applied to evaluate the self-healing capability under different crack phases. As the material is temperature-sensitive, a low temperature of zero degree was applied as the tensile temperature in this work. The simplified process is helpful for the parallel comparison of fracture values. Fig. 10.30 shows the load-time curves of bitumen samples without/with one fiber under 0°C during two procedures including original tension and first reloading after one healing process. Curve (1) belongs to the sample without fiber during the original tension. It has a fracture at point A with a value of about 40 N. After a self-healing process, it was suffered a tension again [curve (2)] with a fracture point B (11 N). Therefore, it can be deduced that the SHE of this sample is about 27.5% (11/40). Another bitumen sample with one fiber was tested according the above steps. It has a fracture at point C in curve (3). The maximum value is about 70 N, which is larger than that for the pure bitumen sample. The reason is that the fiber has played a role in enhancing the tensile strength of the bituminous matrix [13,14]. During its the tension after a healing process [curve (4)], a sudden downward trend appeared (point C). This phenomena may belong to the re-separation of the fiber in bitumen. During a healing process, the two ends of the broken fibers may contact and adhere together. Under a load, the reconnected fiber will be first separated again. Meanwhile, the bituminous material bears tension keeping a continuous structure. With the increase of the load, the bitumen then fractured at the maximum point D (45 N). This sample has a SHE of about 64% (45/70), which is larger than the that of the pure bitumen sample. It is considered that the self-healing of a open crack is a viscosity-driven process including two phases of crack closure and strength



**Figure 10.31** Tensile load values influenced by fiber contents (0-4 fibers, all fibers paralleling to tensile direction) under 0°C during two healing cycles.

recovery. The completion of the crack closure process does not imply a full recovery of the tensile strength in this method. Moreover, the details of the fiber fracture and connection behaviors still need more investigation in future works.

Besides the SHE of bitumen sample with one fiber, it is necessary to test the SHE data of bitumen samples with more fibers. Definitely, multi-fibers will greatly influence the healing behaviors of bituminous material. Fig. 10.31 illustrates the tensile load values of bitumen samples influenced by fiber contents (0-4 fibers)under 0°C during two self-healing cycles. All fibers in bituminous matrix are paralleling to the tensile direction. It can be seen that the original tension load is nearly the same value of 122-135 N. Under a low temperature, the fracture of bitumen may be a brittle rapture with less impact of fibers. After the first self-healing process for 24 h, the pure bitumen sample has the minimum tensile load value of 51 N. The other four samples with fibers (1-4 fibers) has the tensile load values of 65, 66, 73 and 74 N. From the above data, it can be deduced that the fibers additive has greatly enhanced the recovery degree of mechanical properties. In other words, the fibers definitely help to improve the self-healing capability of bitumen samples. The tensile load values of samples have also been tested after the second selfhealing process with a healing time of 24 h. Comparing to first healing process, a similar result is found as the second healing process. The pure bitumen sample has tensile load of 41 N, which is less than the value after its first healing. Other samples with fibers have a decrease trend of tensile load values. This phenomenon indicates that the multi-self-healing efficiency will be greatly reduced because the rejuvenator may have a largest diffusion behavior in the first self-healing process. This conclusion has also been drawn in the case of microencapsulated rejuvenator applied in bitumen [22]. It was found that with increasing number of self-healing cycles, the recovery percentage decreases, which means that its self-healing ability is being lost [9]. When its strength is lower than a limit point, the aged bitumen cannot recover its properties anymore and the self-healing ability disappears. Moreover, the recovery time also increases with increasing self-healing cycles. This indicates that the microcrack needs more time to heal with increasingly aged bitumen [9,10]. Another reason is that the fibers can not enhance the tensile strength of bitumen composite samples with a rapture state after the first healing process. The rapture fibers can not return to the connection state during the self-healing of bitumen. It means the fibers do not afford help to resist the tensile strength of bitumen samples.

#### 10.7.4 Self-healing efficiency influenced by fiber orientation

The application of fiber-reinforced organic or inorganic composites in civil engineering is increasing as a high-performance structural element or reinforcing material for rehabilitation purposes. Its high strength-to-weight ratio and excellent durability make the composite an ideal material for construction. Considering the orthotropic nature, fiber orientation has been found to have a significant influence on the material properties of laminated or pultruded composites. Polymer fibers typically have uniaxial tensile strengths. Therefore, for materials with small off-axis angles, the majority of the load is taken along the fiber orientations. In contrast, for high off-axis angles (higher than 30 degrees), the composite presents similar behavior to that of just the resin (matrix)-the fiber has little effect. Therefore, due to the higher strength of the fiber, a considerably higher strength or stiffness of glassfiber reinforced plastic (GFRP) composite can be achieved in small off-axis fiber orientations compared with high off-axis loading directions. In some relevant previous works, it has been found that the ratio between pultruded GFRP strengths at 0 and 90 degrees is around 70% [23]. Bitumen binder is a thin film between the aggregates forming the asphalt. Fibers definitely disperse in these thin films. To estimate the SHE of bitumen samples with different fiber orientations, various fiber orientations have been proposed to understand the mechanical behavior. The present work is to shed some new lights on the relationship between the uniaxial and flexural post-cracking behavior with special reference to fiber distribution and orientation in the standard test specimen.

Fig. 10.32 shows the SHE values of bitumen samples with one fiber under 0°C during their four healing cycles. The fiber origination ( $\theta$ ) is defined as the angle between the hollow fiber and tensile direction. When  $\theta$  is equal to 15 degrees, the SHE values have a similar data between 70%-75% in the first healing cycle. The fiber orientation nearly do not affect the self-healing process. The reason may be attributed to the oily rejuvenator movement out of fiber without obstruction. With the increasing  $\theta$  values, the SHE is dramatically decreased. For example, the sample has a fiber orientation of  $\theta = 30$  degrees, its SHE values are 73.5%, 73%, 67.0%, and 64.1% during 1-4 healing cycles. Meanwhile, the sample has the SHE values are 72.3%, 72%, 66.1%, and 60.2% during 1-4 healing cycles with a fiber



**Figure 10.32** Self-healing efficiency of bitumen samples with one hollow fiber influenced by the angles (hollow fibers and tension direction) during four healing cycles under  $0^{\circ}$ C.

orientation of  $\theta = 45$  degrees. This trend has also been found in above analysis (Fig. 10.8). However, it was found that the sample only has a little decrease in SHE values during the first and second self-healing cycles with the fiber orientation between 15 and 45 degrees. This phenomenon means that the fiber orientation nearly do not influence the healing behaviors during the original two healing cycles. In the beginner healing process, the rejuvenator will have nearly the same diffusion behaviors. However, larger fiber orientation will definitely block the flow of oily rejuvenator. The rejuvenator may have less opportunity to diffusion into the rapture interface of bitumen sample. Another reason is that with the increasing fiber orientation, the rejuvenator cannot directly flow out into the rapture interface because of the existence of the orientation. More details of this phenomenon will be investigated under a molecular lever in our future work.

### 10.7.5 Self-healing efficiency influenced by temperature and time

It should be noted thatbituminous material has a nature of viscoelastic property. The self-healing process, namely the crack closure and the strength gain, is a viscoelastic feature. The strength gain relies on the wetting and diffusion of bituminous molecules and then leads to the mechanical acceleration. The polymer molecules movement greatly depends on the time and temperature factors. Normally, the self-healing model can be describers using the time-temperature superposition principle as shown in Eqs. (10.7 and 10.8) [9,10].

$$SHE = \left[1 + \left(\frac{m}{t \alpha_T}\right)^{\frac{\log 2}{n}}\right]^{\frac{n}{\log 2}} \cdot 100$$
(10.7)

$$\log \alpha_T(T) = \frac{\Delta E_a}{2.303R} \left( \frac{1}{T} - \frac{1}{T_0} \right) \tag{10.8}$$

where  $\alpha_T$  is the time-temperature superposition shift factor, *m* and *n* are model parameters,  $\Delta E_a$  is the apparent activation energy (unit: J/mol), and *R* is the universal gas constant (8.314 J/(mol K). Based on this model, Tabakovi et al. [15] demonstrated the model parameters of pure bitumen. Su et al. [11,14] investigated the time-temperature dependency of self-healing bitumen materials using microcapsules containing rejuvenator.

Fig. 10.33A shows the self-healing efficiency values of bitumen samples with one hollow fiber influenced by temperature (5°C, 10°C, 15°C, 20°C, 25°C, and 30°C) during three healing cycles. Fig. 10.33B illustrates a typical photograph of bitumen sample during a tension under 30°C. We can see the viscoelastic property of bitumen sample from the tensile deformation. Because the bituminous material is high temperature-sensitive, the test temperature range is  $0^{\circ}C-30^{\circ}C$  in this work. From the SHE values, it can be concluded that increasing the temperature can dramatically enhance the self-healing effects for each healing cycle. The reason is that higher temperature can accelerate the capability of molecule movement both for



**Figure 10.33** Self-healing efficiency of bitumen samples with one hollow fiber influenced by temperature (5°C, 10°C, 15°C, 20°C, 25°C, and 30°C) during three healing cycles: (A) the data of self-healing efficiency and (B) a photograph of bitumen sample during a tension under  $30^{\circ}$ C.

bitumen molecule and rejuvenator molecule. In other words, higher energy can reduce the resistance to the molecular motion. Similarly, this phenomenon has been explained in detail in previous works [13,25]. The third healing cycle has the lowest SHE values, which is consistent with the above conclusion. The multi-self-healing process will reduce the healing capability of bituminous materials. After repeated healing actions under the same temperature, the molecules have reached a new equilibrium state. Without external energy, the self-healing process will have an increasing threshold level.

Besides the temperature, time is another factor that greatly influences the selfhealing property of bituminous material. The reason is that the molecules of viscoelastic material have enough time to regulate the molecular states. In detail, the bituminous material has an enough time to occur the wetting through entanglement for molecular chains [9]. At the same time, the rejuvenator molecules also have a longer time to diffuse in aged bitumen, recovering the original characteristics of bitumen [10]. To verify this conclusion, a simplified experimental process was designed in this work. Fig. 10.34 shows the SHE of bitumen samples with one hollow fiber influenced by time (1, 2, 3, 4, 5, and 6 days) for three healing cycles under 0°C. For the pure bitumen sample, its SHE values has a similar same value of 50% in the beginning two days. Then the value decreases to about 45% in the next four days. Without external help of rejuvenator, the pure aged bitumen does not have an enhanced self-healing capability [9,10,15]. For each bitumen sample



**Figure 10.34** Self-healing efficiency of bitumen samples with one hollow fiber influenced by time (1, 2, 3, 4, 5, and 6 days) for three healing cycles under 0°C.

with hollow fiber, the SHE value has increased with the time prolongation in the same self-healing cycle. For example, the SHE values of bitumen sample in the second healing cycle in 6 days are 65%, 70%, 77%, 78%, 78%, and 80%. After 6-days of healing process, the bitumen can nearly recover to its original state before the first healing process. It indicates that the time prolongation can dramatically enhance the self-healing results of bitumen.

# 10.8 Conclusion

In this study, hollow fibers containing oily rejuvenator were successfully fabricated by a one-step wet-spinning method using PVDF resin. The states of self-healing hollow fibers containing rejuvenator in pure bitumen were experimentally observed. These hollow fibers containing oily rejuvenator were prepared by a one-step wetspinning technology applying PVDF as shell materials. Hollow fibers were mixed in bitumen forming composite samples. A repetitive tensile method was applied to evaluate the self-healing behaviors autonomously including crack closure and crack-healing at various rest periods and temperatures. From the mentioned preliminary results, the following conclusions can be concluded

- 1. The SEM morphologies for inside microstructure of hollow fibers imply that the surface of hollow fibers were smooth without flaw. The micropores in shell of fibers are channels for rejuvenator to release out of hollow fibers.
- 2. Several bitumen composite samples were made with sealed ends and fixed-length hollow fibers. XCT results show that these fibers distributed in bitumen with an integrality state. Hollow fibers can resist the thermal actions of temperature changes in bitumen. Hollow fibers can survive in bitumen safely without debonding.
- **3.** 3D XCT images show the details of both vertical and horizontal distribution of hollow fibers in bitumen. The break of hollow fibers can be triggered by both interface break and external forces.
- **4.** The penetration and diffusion behaviors of rejuvenator were observed based on the movement tracks of oily rejuvenator using a fluorescence microscopy. The rejuvenator permeated through the micropores on a hollow fiber and diffusion into aged bitumen during 5 days.
- **5.** The PVDF hollow fibers containing oily rejuvenator were prepared by a one-step wetspinning method. The fixed-length hollow fibers with sealed ends can be mixed in bitumen forming homogeneous composite samples. No debonding phenomenon was found between the hollow fibers and the bitumen. This tight interface structure provides a prerequisite for mechanical testing.
- **6.** A repetitive tensile method was successfully designed to measure the self-healing behaviors of bitumen samples through comparing the SHE values at various rest periods and temperatures.
- **7.** Both the content and orientation of hollow fibers in bitumen influenced the self-healing behaviors. Hollow fibers cannot enhance the tensile strength of bitumen composite samples with a rapture state after the first healing process, because they do not afford help to resist the tensile strength of bitumen samples. Larger intersection angle between the

tensile strength and the hollow fiber ruins the smooth flow of rejuvenator into crack, leading to a poor self-healing effect.

**8.** Raising temperature and prolonging time have the same enhancing effect on the SHE following the time-temperature superposition principle. Because of the nature of viscoelastic behavior, self-healing is considered as a process of crack closure and the strength gain with the help of viscoelastic feature under a certain temperature for a period of time.

# 10.9 Future work and outlook

In this chapter, self-healing biomimetic microvascular composite containing oily rejuvenator were successfully fabricated and characterized. To promote the wide application of this product, several recomendations are offered. The cost of this self-healing biomimetic microvascular product should be decreased as much as possible so that it can attract more pavement engineering users. Large-scale production can dramatically reduce the cost. A model should be developed to optimize the application of hollow fibers in asphalt including the length, diameter, and amount in various bitumen materials. Fig. 10.35 shows a photograph of hollow fiber



Figure 10.35 Photograph of hollow fiber containing rejuvenator in asphalt.

containing rejuvenator in asphalt. A real application should be carried out to optimize the application technology. As a novel chemical product, its needs a continuous optimization in real applications.

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# Self-healing pavements using microcapsules containing rejuvenator: from idea to real application



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## 11.1 Introduction

Asphalt concrete is one of the most common types of pavement surface materials used in the world. It is a porous material made at a very high temperature of about  $180^{\circ}$ C that consists of mixture of asphalt binder (bitumen), aggregate particles, and air voids. After some years of usage, the stiffness of asphalt concrete increases, and its relaxation capacity decreases. The binder becomes more brittle, then microcracks develop in it and cracking of the interface between aggregates and binder occurs [1]. It is prone to go fragile and stiff due to exposing to heat, oxygen, and ultraviolet light during storage, mixing, transport, and laying down, as well as during service life [2–4].

To restore the asphalt original properties, one of the most effective methods is to use a rejuvenator, which most important goal is to resort the asphaltenes/maltenes ratio [5]. Rejuvenating agents consist of lubricating and extender oils, which contain a high proportion of maltene constituents. In general, rejuvenator should be highly aromatic; both hardening susceptibility and temperature susceptibility are improved. However, there is a big problem for a rejuvenator to be successfully applied. It is hard to penetrate the pavement surface. Shen et al. [6] reported the usage results of three rejuvenators, and it was found that none penetrate into the asphalt concrete more than 2 cm. The rejuvenator, at the same time, will cause a high reduction of surface friction of pavement and contaminate the environment. Therefore, encapsulation rejuvenator inside-usage in asphalt is an alternative approach [7]. Pours sand was applied to encapsulate rejuvenator using epoxy resin [8]. The advantages of these capsules are strong enough to resist the mixing process, the high temperature, and all the years in road until the capsules are necessary. However, there are still some limitations restricting these capsules' application. The main reason is that it is hard for rejuvenator flowing out from the porous sands when the shell is broken because the rejuvenator has high viscidity consisting of lubricating and extender oils. The capillary action of porous structure also limits the rejuvenator to release out.

We are now in a position to start exploring novel microcapsules containing rejuvenator applied in asphalt by chemical method. The microcapsules must have the thermal stability resisting the melting temperature of asphalt. These microcapsules break due to the constant fatigue loads and the higher stiffness of the binder that occurs when it oxidizes; then the rejuvenator is released to restore the asphalt. To produce microcapsules containing rejuvenator by chemical method, the factors of cost, complexity, and capacity must be considered for construction industry. Moreover, these core/shell structures of microcapsules need to meet the specific requirements such as size distribution, thermal stability, nonbiodegradable and mechanical properties, because these influence their service performance [9]. The most common chemical methods for the microencapsulation are interfacial polymerization, in situ polymerization, emulsion polymerization, layer-by-layer deposition, spray, and coacervation [10]. A survey of literature shows that the melamineformaldehyde (MF)-shell microcapsules fabricated by in situ polymerization may be an option to deal with the above problems [11]. MF-shell microcapsules have been successfully applied to encapsulate flavor, pharmacist, phase change materials, and self-healing materials [12-14]. Usually, MF resin is adsorbed and cured on surfaces of core particles through in situ polymerization with the help of a polymeric surfactant. MF resin is easy to obtain at low cost. The MF-shell microcapsules have relatively higher thermal stability and mechanical properties [15]. In our previous study [16], we have reported that the MF resin was applied to fabricate the microcapsules with compact shell structure. Through controlling the synthetic details, the microcapsules can own the ability to resist the presser with a larger yield point [17]. Recently, it was proved that the methylation reaction on MF-shell material can enhance the thermal stability and mechanical properties of microcapsules [18]. In addition, the grafted shell will improve the interface stability between microcapsules and matrix [19].

Studies have reported the relationship between the shell morphologies and compactability of MF shells [20-22]. In these previous studies, the method of controlling permeability is to decrease the shell defects, such as methods of adding accessory ingredients, modeling the degree of cross-linking, and changing the molecular structure of shell polymer. Several new techniques for compact encapsulation have been developed, such as increasing the molecular weight of polymer, the expansion ability, and the mechanical strength [23-25]. Besides all the abovementioned technologies, MF shells are all fabricated by in situ polymerization with a process of one-step dropping monomers for suitable thickness and structure of shells. The mechanism of one-step dropping monomers means a one-step coacervation (OSC) of prepolymers [26]. These microcapsules cannot be satisfied with the nonpermeable need of some special core materials such as phase change materials (PCMs), liquid crystal, and biologically active materials, which are not expected to be released and contaminated by outside environment. It is suspected that the MFshell microcapsules containing rejuvenator can resist the temperature of about 180°C-200°C mixing with melting asphalt. Therefore, it is a very important approach to fabricate novel structure microcapsules with higher compact shells. Interestingly, a two-step coacervation polymerization was developed to increase the thermal stability and compactability of MF-shell microcapsules [27,28].

At present, the development of simple, cheap, robust, and environmentalfriendly method for encapsulating suitable size rejuvenator is crucial for chemical engineering and construction engineering. In view of the above, the objective of this work was to fabricate microcapsules containing rejuvenator by in situ polymerization method using methanol-modified melamine-formaldehyde (MMF) as shell material. Styrene-maleic anhydride (SMA) copolymer solid was used as a nonionic dispersant. To enhance rigidity and toughness of MMF shells, a two-step coacervation structure was synthesized through a twice polymerization method. The chemical structure, surface morphologies, average diameter, and thermal stability properties of these MMF-shell microPCMs were investigated systemically. The breakage of the shell will occur based on the mismatch of thermal expansion of the core and shell materials at high temperatures [29]. It was expected that the rejuvenator would be protected with less cracks and lower permeability during asphalt pavement.

# 11.2 Design the microcapsules containing rejuvenator

The self-healing process based on microcapsule is one of the newest smart ideas, which has been proved very successful in some polymer self-healing materials [30]. However, it is limited by two big puzzles applying in asphalt, that is, microcapsule shells have higher thermal stability and higher mechanical strength to resist the melting temperature and mixing pressure of asphalt in practical applications. Microcapsules must keep shape and compatibility at 180°C-200°C during asphalt application. To overcome this problem, pours sand was applied to microcapsules [31]. Although the mechanical properties were improved, the rejuvenator was not easy to release from pours structure. On the contrary, microcapsule shells will not break with higher mechanical strength, such as inorganic shells or flexible organic shells. The result is that the macrocrack may be triggered and lead to fracture without leaking of rejuvenator. Therefore, the shells usually are polymeric materials with appropriate strength and toughness.

To produce microcapsules containing rejuvenator by chemical method, the factors of cost, complexity, and capacity must be considered in construction industry. Moreover, these core/shell structures of microcapsules need to meet the specific requirements such as size distribution, encapsulation ratios, and nonbiodegradable property, because these influence their service performance [32]. As asphalt thin films between aggregates are less than 50  $\mu$ m, size of microcapsules containing rejuvenators should be smaller than 50  $\mu$ m to avoid being squeezed or pulverized during asphalt forming. Previous work has demonstrated that the mechanical strength of microcapsules is size-dependent [33]. Therefore, it was important to find appropriate fabrication parameters to suit the size demand.

The most common chemical methods for the microencapsulation are interfacial polymerization, in situ polymerization, emulsion polymerization, layer-by-layer deposition, spraying, and coacervation [34]. A survey of literature shows that the MF-shell microcapsules fabricated by in situ polymerization may be an option to deal with the above problems [35]. In situ polymerization is a direct polymerization, which is carried out on the oily particle surfaces. MF is a hard, thermosetting plastic material made from melamine and formaldehyde by polymerization. It is easy to obtain at a low cost. It can act as flame-resistant material. Its degradation involves methylene and/or methylene ether bridges and possibly methylol groups. Below about 250°C, only these groups appear to be involved and fairly high initial rates of weight loss are followed by decreased rates of loss [36]. The MF-shell microcapsules also have relatively higher thermal stability and mechanical properties [18]. Through controlling the synthetic details, the microcapsules can own the ability to resist the presser with a larger yield point [37]. Recently, it has been proved that the methylation reaction on melamine-formaldehyde (MMF) shell material can enhance the thermal stability and mechanical properties of microcapsules [38]. In addition, the grafted shell will improve the interface stability between microcapsules and matrix [11]. Interestingly, we also found that the mechanical strength and thermal stability of shells can be controlled by regulating the shell structure parameters (thickness and density) [18].

There are many methods and techniques to fabricate microcapsules using two or more reactive components forming core-shell structure. MF-shell microcapsules are usually designed by a method called as in situ polymerization with a certain of size, thermal stability, and mechanical property [12,39,40]. A majority of these methods for from the encapsulating walls is to provide minute discrete droplets containing the intended fill material dispersed within a continuous phase that contains at least one of the reactive components. The microstructure of shells formed from materials present only in the discontinuous phase, greatly depending on the polymerization kinetics. The parameters of temperature, pH value, and shell material concentration affect the shell formation. Thus, the shell-forming materials dispersed into the discontinuous phase polymerize and migrate outward to the interface between the discontinuous and continuous phases, resulting in the formation of various microcapsule shells. All results indicate that we can control the fabrication process in in situ polymerization to design the shell structure of microcapsules. Fig. 11.1 illustrates the potential relationship between polymerization conditions and the properties of microcapsules. For example, Zhang et al. [11] investigated the effects of stirring rate, contents of emulsifier, and contents of core material on diameters, morphology, and thermal stabilities of the MF-shell microcapsules. Fan et al. [40] found that surface morphology of the microcapsule depended mainly on the final pH value and stirring rate. We also reported that the dropping speed of shell materials had a great influence on the shell structure of MF-shell microstructure [16]. Therefore, it is positive that we can optimize the polymerization parameters to achieve the microcapsules containing rejuvenator for asphalt. Microcapsules containing rejuvenator must be of optimal size and strong enough to remain intact during manufacture and further processing such as drying, pumping, and mixing in



**Figure 11.1** Illustration of the potential relationship between fabrication conditions and the properties of microcapsules by in situ polymerization.

melting asphalt. However, there are little systematic experiments about the controlling of MF-shell properties.

# 11.2.1 Experimental methods

#### 11.2.1.1 Materials

The shell material was a commercial prepolymer of MF modified by methanol (solid content was 78.0%) purchased from Aonisite Chemical Trade Co., Ltd. (Tianjin, China). The rejuvenator was a commercial oily product. SMA copolymer (Scripset520, Hercules, USA) was applied as dispersant. Nonylphenol ethoxylate [NP-10,  $C_9H_{19}C_6H_4(OCH_2CH_2)_{10}OH$ ] is used as a nonionic surface-active agent in this study. A small percentage of the anhydride groups have established connection with a low molecular weight alcohol and it is fine, off-white, free-flowing powder with a faint, aromatic odor.

#### 11.2.1.2 Fabrication microcapsule containing rejuvenator

Microcapsulation by coacervation procedure has been described in our previous work [38]. It includes three main steps: (1) phase separation of the coating polymer solution: SMA (10.0 g) and NP-10 (0.2 g) were added to 100 mL water at 50°C and allowed mixing for 2 h. And then a solution of NaOH (10%) was added dropwise

adjusting its pH value to 4-5. The above surfactant solution and rejuvenator were emulsified mechanically under a vigorous stirring rate of 500-4000 r/min for 10 min using a high-speed disperse machine. (2) Adsorption of the coacervation around the core particles: the encapsulation was carried out in a 500 mL three-neck round-bottomed flask equipped with a condensator and a tetrafluoroethylene mechanical stirrer. The above emulsion was transferred in the bottle, which was dipped in a steady temperature flume. MMF prepolymer (32 g) was added dropwise with various dropping speeds under a stirring speed of 500 r/min. After 1 h, the temperature was increased to  $80^{\circ}$ C. (3) Solidification of the microcapsules: after polymerization for 2 h, the temperature was decreased slowly at a rate of  $2^{\circ}$ C/min to atmospheric temperature. At last, the resultant microcapsules were filtered and washed with pure water and dried in a vacuum oven.

#### 11.2.1.3 Optical morphology

An optical microscope was used to check the fabrication process of microcapsules in emulsion. About 1 mL of the colloidal solution was extracted and spread on a clean glass slide  $(1 \times 3 \text{ cm})$ . Various images were taken in the different stages of microcapsule forming process.

#### 11.2.1.4 Environmental scanning electron microscopy

The dried microcapsules were adhered on a double-sided adhesive tape without cracking the shells. The surface morphologies were observed by using a Philips XL30 environmental scanning electron microscope (ESEM) at an accelerated voltage of 20 kV.

#### 11.2.1.5 Average size of microcapsules

For each microcapsule sample, the average diameter is the mean value of 50 microcapsules measured from the ESEM morphology image.

#### 11.2.1.6 Shell thickness and density of microcapsules

About 2 g MMF-shell microcapsules were mixed in 5 g epoxy resin. After the composite was dried at room temperature, it was carefully cut to obtain the cross section. The thickness of shells can be achieved from the SEM images of cross section of microcapsules [21]. At least 20 shells of each sample microcapsule were measured and the average data were recorded. About 10 g of dried microcapsules were ground with a completed shell break. The mixture material was then put in water to separate the shell and core materials. The shell material was then filtrated and dried. The density of shell ( $\rho$ ) was measured by testing cumulate density of powder following Eq. (11.1):

$$\rho = \frac{W}{V} \tag{11.1}$$

where W is the weight of shell and V is the volume of the shell. V was tested using a gas displacement technique.

#### 11.2.1.7 Thermogravimetric analysis

For the microcapsules to survive the mixing process of the asphalt, they must resist the temperature in a range of  $160^{\circ}\text{C}-180^{\circ}\text{C}$ . These temperatures can produce molecular scissions or intermediate compounds in the rejuvenator such as ketones or alcohol, as well as mass loss due to the evaporation of these products. The thermal stability characterization of microcapsules was performed on a Dupont SDT-2960 Thermogravimetric analysis (TGA) analyzer at a scanning rate of 5°C/min under a flow of 40 mL/min N<sub>2</sub>. In order to obtain thermal stability curves, mass of microcapsules and mass of rejuvenators were measured.

### 11.2.1.8 Measurement of the mechanical strength of single microcapsule

The nanoindenter (TI 950 TriboIndenter, Hysitron) was applied to measure the mechanical properties of the microcapsules (hardness and Young's modulus) of the microcapsules. Nanoindenter is a mechanical properties microprobe that is designed to provide data collection capabilities in tandem with the versatile TestWorks interface. The microcapsules were dispersed into alcohol, and then carefully separated on a smooth glass slide. Another glass slide with thin-layer strong glue was used to conglutinate the separated microcapsules. A cone tip (3 µm radius) was used to measure hardness and Young's modulus as shown in Fig. 11.2A and B. At the beginning of tests, the microscope was turned on to find a single microcapsule. Then the tip was slowly shifted to the surface of the single microcapsules. The load placed on the indenter tip is increased as the tip penetrates further into the specimen and soon reaches a user-defined value. At this point, the load may be held constant for a period or removed. The force and displacement resolutions were 50 nN and 0.01 nm, respectively; the holding time of indentation was 10 s. The surface approach velocity was 10 nm/s and the maximum displacement was 2000 nm. A record of these mechanical values can be plotted on a graph to create a load-displacement curve. The area of the residual indentation in the sample is measured and the hardness (H) is defined as the maximum load  $(P_{max})$  divided by the residual indentation area  $(A_r)$ , or

$$H = \frac{P_{max}}{A_r} \tag{11.2}$$

The hardness is given by the above equation, relating the maximum load to the indentation area. The area can be measured after the indentation by in situ atomic force microscopy.



**Figure 11.2** Mechanical properties of single microcapsule are measured by nanoindentation: (A) a tip is slowly shifted to the surface of the single microcapsules, (B) the load placed on the indenter tip is increased as the tip penetrates further into the specimen and soon reaches an user-defined value, and (C) typical continuous load—displacement curve.

A set of continuous load-displacement curves are presented in Fig. 11.2C. Some important quantities are the peak load and displacement,  $P_{\text{max}}$  and  $h_{max}$ , the residual depth after unloading,  $h_f$ , and the slope of the initial portion of the unloading curve (S = dP/dh). S has the dimensions of force per unit distance, and so it is also known as the elastic stiffness of the contact. Young's modulus (E) was calculated from the slope of the linear portion, dP/dh upon unloading [41],

$$\frac{1}{E_r} = \frac{1 - \nu}{E} + \frac{1 - \nu_0}{E_0} \tag{11.3}$$

$$E_r = \left(\frac{\Pi}{A_r}\right)^{1/2} dp/dh \tag{11.4}$$

where  $E_r$  is the reduced modules, E and v are Young's modulus and Poisson's ratio for the materials and indenter, respectively.

#### 11.2.2 Results and discussion

# 11.2.2.1 The effect of core material emulsion stirring speed on the average size of microcapsules

SMA was applied as a nonionic dispersant by in situ polymerization method in this study. SMA molecules can hydrolyze in water by NaOH and form carboxyl (-COOH) groups. These hydrophilic polar groups, alternatively arranging along the SMA backbone chains, thus associate with water molecules and trimly cover the oil droplets surface with hydrophobic groups oriented into oil droplets and

hydrophilic groups out of oil droplets [11]. This orientation of molecular groups results in a relatively strong electron negative field on the surface of oily particles. Anionic polyelectrolyte-hydrolyzed SMA has anionic carboxyl groups that can interact with positively charged below the  $\zeta$  potential. And then the MMF prepolymer will be adsorbed by static on oil particles and polymerization occurred.

We used 2.0 wt.% SAM to disperse the rejuvenator in this study. Optical microphotographs of microPCMs were taken to illuminate the encapsulation details. Fig. 11.3A and B show morphologies of microcapsules with a core/shell ratio of 2/ 1 fabricated under 3000 r/min emulsion stirring rate in emulsion state and dried state at room temperature. It can be seen that the organic core material is dispersed into particles in water. Being encapsulated by shell material, the core particles are ultimately separated through the regulation of hydrolyzed SMA molecules. These microcapsules are regular globe shape with smooth surfaces. Fig. 11.4 shows the average diameters of microcapsules (core/shell ratios of 3/1 and 1/3) under various emulsion stirring rates in the range of 1000-6000 r/min. With the decreasing of stirring rates, the average diameters decreased sharply from 23.5 to 5.0 µm. Higher stirring rates will smash the oily rejuvenator into smaller droplets. This is in agreement with the results reported in our previous study [11]. The stirring rate only determines the core particle size; the microcapsule size is at the same time influenced by the shell thickness. As the formation of a shell is a coacervation process, the shell thickness is dominated by the amount of shell material. Less core/shell ratio leads to a higher shell thickness value. This result accords with our previous



**Figure 11.3** Morphologies of microcapsules with a core/shell ratio of 2/1 fabricated under 3000 r/min emulsion stirring rate in (a) emulsion state and (b) dried state at room temperature.



**Figure 11.4** The average diameters of microcapsules (core/shell ratios of 3/1 and 1/3) under various emulsion stirring rates in the range of 1000–6000 r/min.

study and indicates that the average diameter is mainly determined by emulsion stirring rates [16].

It can be confirmed that the rejuvenator applied in this study as core material will not influence this rule. In addition, it means that the MMF cross-linked with a compact structure forming thin shells. From the data, it can also be concluded that the core/shell ratios nearly do not affect the average size of microcapsules. In other words, the average size is mainly decided by the core material disperse speed. The average diameter is an important parameter for microcapsules containing rejuvenator influencing their application possible in asphalt. Too small size will limit the encapsulated content of rejuvenator. In another case, large microcapsules may be broken in mixing with asphalt concrete.

#### 11.2.2.2 Shell thickness and density of microcapsules of microcapsules

Thickness and density of shells are also critical properties for the thermal stability and compactness of microcapsules. It is not suitable that the rejuvenator leaks or penetrates outside, losing the shell protection. Fig. 11.5 shows the SEM morphology of shell thickness of microcapsules. Interestingly, it can be seen in Fig. 11.5A that the shells had been broken after the microcapsules under 300°C for 30 min. The



**Figure 11.5** SEM morphology of shell thickness of microcapsules: (A) the broken shells of microcapsules under 300°C for 30 min, (B) typical intersecting surface morphology of microcapsules/epoxy composite, and (C) a shell cut not across the equator of global shape microcapsule.

microcapsules had cracks on shells under high temperature. Then the core material had been released out without the protection of shells. Because the shell materials still keep the weight and pieces state, we can measure their thickness and density. Fig. 11.5B shows a typical intersecting surface morphology of microcapsules/epoxy composite. It must be noted that the shell may not be cut across the equator of global shape microcapsule as shown in Fig. 11.5C, and the thickness must be an average value of at least five microcapsules for each sample.

Table 11.1 lists the data of shell thickness and density of microcapsules prepared by core stirring rates of 1000-3000 r/min. The microcapsule samples, coded as M-1-M-12, were all dispersed with 2.0% SMA with core/shell ratios of 2/1, 1/1, 1/2, and 1/3, respectively. For example, microcapsule samples (M-10, M-11, and M-12) have the thickness data of  $2.78 \pm 0.52$ ,  $2.88 \pm 0.40$ , and  $3.27 \pm 0.49 \,\mu\text{m}$ . It means more shell material makes the shell thicker structure. This conclusion is similar to the previous result in analyzing of the average size of microcapsules. The microcapsule sample M-12 has the largest density of  $1.14 \pm 0.26$  g/cm<sup>3</sup>. The reason is that more shell material may decrease the shell structural defects such as holes and caves. The prepolymer can regulate their molecules and crosslinked with enough reaction time on core material leading to a higher cross-linking density of shell material. Normally, MF-cured resin has the density of about 1.50 g/cm<sup>3</sup> [11]. As microcapsule formation is a coacervation process, the MMF shell may be looser than ideal state. From the thickness data, we can also prove that the shell is a tiny membrane, which will not greatly affect the average size of microcapsules.

Sample	Core/shell ratio	Stirring rate (r/min)	Shell thickness (µm)	Shell density (g/cm <sup>3</sup> )
M-1	2/1	1000	$1.68 \pm 0.21$	$0.90\pm0.35$
M-2		2000	$1.63 \pm 0.45$	$0.95 \pm 0.44$
M-3		3000	$1.71 \pm 0.62$	$0.94\pm0.52$
M-4	1/1	1000	$1.69 \pm 0.41$	$0.88 \pm 0.46$
M-5		2000	$1.70 \pm 0.47$	$1.07\pm0.47$
M-6		3000	$1.85 \pm 0.32$	$1.08\pm0.58$
M-7	1/2	1000	$2.70 \pm 0.42$	$1.05\pm0.54$
M-8		2000	$2.84\pm0.52$	$1.09\pm0.65$
M-9		3000	$2.70 \pm 0.44$	$1.00\pm0.34$
M-10	1/3	1000	$2.78\pm0.52$	$1.04\pm0.45$
M-11		2000	$2.88 \pm 0.40$	$1.02\pm0.37$
M-12		3000	$3.27 \pm 0.49$	$1.14\pm0.26$

Table 11.1 Shell thickness and density of microcapsules containing rejuvenator.



**Figure 11.6** Thermal stability of microcapsules containing rejuvenator tested by TGA: (A) the TGA plot of pure rejuvenator shows a marked weight loss between 337°C and 469°C, (B) TGA curves of microcapsules with various core/shell ratios (2/1, 1/1, 1/2, and 1/3) under core material emulsion stirring speed of 3000 r/min, and (C) the typical TGA curves of microcapsules (with core/shell ratio of 1/1, core material stirring rate of 4000 r/min) fabricated with various shell material dropping speeds of 5.0, 2.5, 1.0, and 0.5 mL/min.

#### 11.2.2.3 Thermal stability of microcapsules containing rejuvenator

TGA has been widely applied to investigate the encapsulation effect and shell compactness of microcapsules [42]. The microcapsules were composited with increasing temperature according to presenting residual weight (wt.%). The curves may reflect thermal stability and structure of polymeric shell. In Fig. 11.6A, the TGA plot of pure rejuvenator shows a marked weight loss between 337°C and 469°C. This is due to the evaporation of oil substance under high temperature. For the pure MMF prepolymer, a degradation temperature of 192°C is reported in a previous study [16]. Cross-linked MMF resin had a rapid weight loss by thermal decomposition. The final thermal degradation of MMF prepolymer is observed at about 265°C.

Fig. 11.6B shows the TGA curves of microcapsules with various core/shell ratios (2/1, 1/1, 1/2, and 1/3) under core material emulsion stirring speed of 3000 r/min. All the samples were fabricated by adding the shell prepolymer with the dropping speed of 1.0 mL/min. It is clear that all microcapsules containing rejuvenator lost weight above the temperature of 220°C. Nearly about 10% weight is lost under 100°C for all microcapsule samples because of the evaporation of water and small molecules. The degradation temperature of microcapsules is higher than pure MMF prepolymer, which means that MMF prepolymer already cross-linked to a network structure. In addition, the degradation temperature is higher than the melting temperature of 180°C. It indicates that the cured MMF resin will not be thermal decomposed mixing with melting asphalt. Other significant phenomena are observed that core/shell ratios had a great influence on the original decomposing temperature of microcapsules. More shell materials increased the thermal degradation temperature of microcapsules. The initial decomposing temperature of microcapsules with a core/shell ratio of 1/3 is about 280°C higher than that of microcapsules with core/ shell ratios of 2/1, 1/1, and 1/2. This result attributes to two reasons. One is that more shell material will make the shell with an increased thickness. With the temperature increasing, the shell will first crack or be broken under high temperature before decomposition. Another reason is that the thicker shell will keep compact shape without defects. The rejuvenator had been protected well in shells. It implies at the same time that the rejuvenator had successfully been encapsulated by MMF resin.

Besides core/shell ratios, the shell material dropping rate is another factor that greatly affects the microstructure of microcapsules [16]. Moreover, it has been proved that the shell compactability may be increased with the decreasing of particle size of microcapsules [43]. To simplify the complexity of this problem, we selected the microcapsule samples all fabricated under the stirring speed of 4000 r/ min to investigate the relationship between the shell material dropping rate and thermal stability of shells. Fig. 11.6C shows the typical TGA curves of microcapsules (with core/shell ratio of 1/1, core material stirring rate of 4000 r/min) fabricated with various shell material dropping speeds of 5.0, 2.5, 1.0, and 0.5 mL/min. As can be seen, when the shell material dropping speed increased in fabrication, the obtained microcapsules had worse thermal stability. The microcapsules had the highest beginning decompose temperature of about 300°C prepared by shell material dropping speed of 0.5 mL/min. This might be due to that the prepolymer concentration in solution determined by the dropping rate will affect the formation speed of shells. Under more rapidly dropping rate, the shell will be formed faster with enough shell material, which brings disfigurements such as microcrack, microcavity, and capillary on shells. These disfigurements may lead the core material to release out easily in a thermal decomposition process. On the contrary, shells will form slowly under the situation of low prepolymer concentration in solution. The prepolymer molecules will adhere on core particles compactly. This result indicates that lower fabrication speed will be a positive approach to fabricate microcapsules containing rejuvenator with better thermal stability.



**Figure 11.7** SEM morphology of microcapsules fabricated under core material stirring rate of 3000 r/min (core/shell ratio, 2/1).

To verify the thermal stability of microcapsules in asphalt, a microcapsule sample was mixed with melting asphalt under various temperatures. The shapes of microcapsules in asphalt were observed by an optical microscope. The microcapsules had an average size of about  $16\mu$ m fabricated under core material stirring rate of 3000 r/min (core/shell ratio of 2/1) as shown in Fig. 11.7. In Fig. 11.8A, it is proved that microcapsules had survived in asphalt under temperature 140°C. With the increasing of temperature to 160°C (Fig. 11.8B) and 180°C (Fig. 11.8C), the microcapsules still keep the global shape without crack and thermal decomposition. These results indicated that these microcapsules can resist the thermal effect of asphalt in the application.

#### 11.2.2.4 Effect of shell material dropping speed on the morphologies of microcapsules

In particular, we pay more interest to develop the methods of increasing the thermal stability of microcapsules containing rejuvenator. TGA tests confirmed that lower shell material dropping speed can make the microcapsules with higher compactability. Furthermore, it is remarkably important to understand the true encapsulation mechanism. More information will be achieved through research on the morphologies of shell. The formation of MMF-shell microcapsules is attributed to precipitation of higher molecular weight prepolymer in the aqueous solution and their aggregation and deposition on the oil-droplets surface result in the rough, porous outer layer of the MMF shell. The smooth nonporous microcapsule shell is believed to be the perfect result of deposition of a low molecular weight prepolymer on the core material. It has been reported that the pH value, stirring rate, and shell material concentration were the main factors influencing the shell morphology [12]. In this



180°C

**Figure 11.8** Optical morphologies of microcapsule/asphalt composites under temperatures of (A) 100°C, (B) 140°C, and (C) 180°C.

study, we focus on the relationship between shell material dropping speed and the morphologies of microcapsules.

Fig. 11.9A–D shows the SEM surface morphologies of microcapsules containing rejuvenator (core/shell ratio, 1/1; stirring rate, 3000 r/min) fabricated under the shell prepolymer dropping rate of 5.0, 2.5, 1.0, and 0.5 mL/min, respectively. In Fig. 11.9A, most of the microcapsules have a rough morphology and a little polymer occupies the interspaces of microcapsules. It can be attributed to the unencapsulated core material and the uncovered shell material. Especially, the surfaces have many protrusions, which may have occurred by incomplete cross-linking or high-speed chemical reaction. With the decreasing of prepolymer dropping rate, the surface morphology of microcapsules is becoming more smoother with litter adhesion material as shown in Fig. 11.9B. There was a part of polymerized shell materials existing between microcapsules without forming shells. In Fig. 11.9C and D, microcapsules have a global shape with no concavo-convex and wrinkle in shell surfaces, and little polymer is pilling between piled microcapsules. These results



**Figure 11.9** SEM surface morphologies of microcapsules containing rejuvenator (core/shell ratio, 1/1; stirring rate, 3000 r/min) fabricated under the shell prepolymer dropping rates of 5.0, 2.5, 1.0, and 0.5 mL/min: (A) microcapsules have a rough morphology and a little polymer occupies the interspaces of microcapsules, (B) with the decreasing of prepolymer dropping rate, the surface morphology of microcapsules is becoming more smoother with litter adhesion material, and (C and D) the microcapsules have a global shape with no concavo-convex and wrinkle embedded in shell surfaces.

reflect that this method has decreased the roughage through prepolymer molecules regulation with enough time. The morphology of microcapsules is the main factor for penetration property of shell. Rejuvenator is protected by MF shell without influences of outside material and environment. This would make the core materials safely without being strained, and the microcapsules would have a long life in practice.

# 11.2.2.5 Mechanical properties of microcapsules

Material micromechanical properties can be determined by indentation techniques, in which a rigid body of defined geometry is pressed into the surface of the tested materials. Nanoindentation has already been applied to measure the mechanical properties of microcapsules containing phase change materials [44] and self-healing agents [45]. Fig. 11.10 shows that the typical load-displacement curves of single microcapsule upon loading increases bilinearly until hold, and then decreases



**Figure 11.10** The typical load—displacement curve of single microcapsule upon loading increases bilinearly until hold, and then decreases sharply upon unloading. Microcapsule samples were fabricated under the core material disperse rate of 3000 r/min with core/shell ratios of 3/1, 2/1, 1/1, 1/2, and 1/3.

sharply upon unloading. All microcapsule samples were fabricated under the core material disperse rate of 3000 r/min with a core/shell ratios of 1/1, 1/2 and 1/3. All curves have a similar shape because they represent both elastic (recovery) and plastic (inelastic) deformations behaviors [44]. It was found that the microcapsules with thinner shell had the lower load data. During unloading, the portion represents the elastic behavior with elastic displacement being recovered. Comparing these curves, it was clear that microcapsule samples had different abilities to recover the original shape. In theory, creep in polymer materials under constant load is controlled by the available free volume for polymer chains and molecular units move. Thinner shell may give the cross-linked MMF polymer more volume to realize movement of molecular units. A survey of literature shows that there is a big difference for load-displacement behavior of microcapsules. For example, Pan [41] reported that MF microcapsules were compressed to rupture under the load of 2.5 mN; meanwhile, Santos et al. [46] found that the MF microcapsules showed elastic deformation under the load of 10.5 mN. The reason may be attributed to the difference in molecular weight and shell microstructure (thickness, density, and porosity) of microcapsules.



**Figure 11.11** Hardness of single microcapsule: the microcapsules were prepared under core material disperse rates of 3000–6000 r/min (0.5 mL/min prepolymer dropping rate) with core/shell ratios of 2/1, 1/1, 1/2, and 1/3.

To obtain furthermore micromechanical information, the hardness and Young's modulus of microcapsules were measured three times for each single microcapsule sample in this study. Because of normal distribution of size, the microcapsules were randomly selected with nearly the average size. As has been proven before, the original core material disperse rates are the main factor determining the average size of microcapsule; shell thickness depends on core/shell ratios. It must be mentioned that research has deduced that the strong glue imposed a negligible influence on the core/shell structure nanoindentation test data [47]. In Fig. 11.11, the hardness of single microcapsule has two impact parameters: core material disperse rates (size) and core/shell ratios (shell thickness). The microcapsules were prepared under core material disperse rates of 3000-6000 r/min (0.5 mL/min prepolymer dropping rate) with core/shell ratios of 2/1, 1/1, 1/2, and 1/3. For each sample, the hardness has a slightly increasing trend with the increasing of size. Moreover, higher shell proportion of microcapsules leads the shell to a higher hardness, which means that the thicker shell has higher ability to resist deformation. This conclusion is similar to the reported results about some living cells [46]. Interestingly, it has also been reported about the dependence of failure strength on microcapsule diameter previously [15].



**Figure 11.12** Young's modulus for single microcapsule samples: the microcapsules were prepared under core material disperse rates of 3000–6000 r/min (0.5 mL/min prepolymer dropping rate) with core/shell ratios of 2/1, 1/1, 1/2, and 1/3.

Fig. 11.12 shows the mean values of Young's modulus for single microcapsule samples affected by size and shell thickness. It indicates that larger microcapsule has higher modulus for each sample. It should be pointed out that this trend is similar to the reported results [30]. The Young's modulus of microcapsule with core/ shell ratio of 2/1 is in the range of 1.15-1.75 GPa; meanwhile, Young's modulus of microcapsule with core/shell ratio of 1/3 is in the range of 1.9-2.7 GPa. It means that more shell material can make the shell with higher thickness leading to higher stiffness of elastic microcapsules. Previous study proved that the MF-shell microcapsules have a "yield point" [11]. Prior to the yield point, a single microcapsule will deform elastically and will return to its original shape when the applied stress is removed. Once the yield point is passed, some fraction of the deformation may be permanent and nonreversible. In this study, all the tests did not carry out over the yield points of the microcapsules by nanoindentation. It can be imaged that the micromechanical properties of microcapsules will depend on the shell thickness and microstructure and molecular weight. The characteristics of core material may be at the same time influence the load-displacement behavior of microcapsules. During the deformation compression, the volume and Poisson's ratio of polymer microcapsules may change continuously with the deformation because of the spherical geometry.

# 11.3 The mechanism of self-healing of microcapsules

An increase in the application of a higher percentage of the preservation and renovation of asphalt pavement is achievable using a rejuvenator. It is the only method that can restore the original properties of the pavements [31]. The most important goal of utilizing rejuvenator products is to restore the asphaltenes/maltenes ratio. Rejuvenating agents have the capability of reconstituting the binder's chemical composition and consist of lubricating and extender oils containing a high proportion of maltene constituents [48]. However, a rejuvenator is not successfully applied because it is hard to penetrate the pavement surface. Shen et al. [49] reported the results using three rejuvenators. It was found that none could penetrate into the asphalt concrete more than 2 cm. When applying these materials, the road must be closed for some time after their application. The rejuvenator, at the same time, will cause a high reduction of surface friction of pavement for vehicles. Moreover, an important aspect of these rejuvenators is that they may be dangerous to the environment. Encapsulation rejuvenator inside-usage in asphalt may be an alternative approach. García et al. [7] reported a method to prepare capsules containing rejuvenator by using epoxy resin as coating and pours sand as skeleton. We reported a method to fabricate microcapsules containing rejuvenator utilizing methanol MMF resin as shell material [50]. These microcapsules had satisfactory thermal stability in bitumen and reliable mechanical properties resisting the mixing process and temperature changes [51]. It has been proved that this product was environmentalfriendly powder encapsulating suitable size rejuvenator for chemical engineering and construction engineering [52].

Self-healing materials based on microcapsules have been studied widely, which have structurally incorporated the ability to repair damage caused by mechanical usage over time. A monomer is encapsulated and embedded within the matrix materials. When the crack gets to the microcapsule, the capsule breaks and the monomer bleeds into the crack, where it can polymerize and mend the crack [53]. Besides the similar structure of microcapsules and self-healing materials for bitumen/microcapsules materials, we still need to consider their special self-healing mechanism. Microcapsules in bitumen can be broken by microcracks, and then the released rejuvenator seals the microcracks and permeates the surrounding bitumen. The capillaries and penetration behavior will determine the self-healing efficiency of aged bitumen [54]. With the help of capillarity, rejuvenator flows into narrow microcracks without the assistance of, and in opposition to, external forces. First, the core/shell structure of microcapsules is taken into account for the specific requirements such as size distribution, encapsulation ratios, and nonbiodegradable property, because these influence their service performance. As bitumen acts as thin layers between aggregates that are usually less than 50 µm, size of microcapsules containing rejuvenators should be smaller than 50 µm to avoid being squeezed or pulverized during asphalt forming. Second, the shell thickness must be controlled to make sure that the microcapsules have excellent thermal stability. It has been found that the thicker shell can enhance the mechanical properties of microcapsules, the microcracks may not able to break these microcapsule [55]. It must be prevented that the microcracks propagation will go round the shells.

The aim of this paper is to investigate the self-healing mechanism of aged bitumen using microcapsules containing rejuvenator. Various microcapsule samples were fabricated to optimize the core/shell structures. In order to observe the selfhealing behaviors, liquid nitrogen was used to generate microcracks in aged bitumen. The effects of mean size and shell thickness of microcapsules were evaluated to understand the self-healing behaviors. At the same time, the capillarity and penetration behaviors were analyzed to evaluate the rejuvenator movement. Properties of virgin and rejuvenated bitumen were compared to evaluate the performance recovery of aged bitumen.

#### 11.3.1 Characterization of microcapsules containing rejuvenator

SMA was applied as a nonionic dispersant by in situ polymerization method in this study. SMA molecules can hydrolyze in water by NaOH and form carboxyl (–COOH) groups. These hydrophilic polar groups, alternatively arranging along the SMA backbone chains, thus associate with water molecules and trimly cover the oil-droplets surface with hydrophobic groups oriented into oil droplets and hydrophilic groups out of oil droplets [11]. Optical microphotographs of microcapsules were taken to illuminate the encapsulation details. Fig. 11.13A shows the morphology of rejuvenator dispersed by ESEM under an emulsion rate of 3000 r/min. It was observed that the organic core material is dispersed into particles in water. After being encapsulated by shell material as shown in Fig. 11.13B, the microcapsules are ultimately separated through the regulation of hydrolyzed SMA molecules with core/shell ratio of 2/1. Fig. 11.13C shows the final dried microcapsules with regular globe shape with smooth surfaces.

The mean size is an important parameter for microcapsules containing rejuvenator influencing their application possibility in asphalt. The too tiny size will limit the encapsulated content of rejuvenator. In another case, large microcapsules may be broken in mixing with asphalt concrete. Fig. 11.14 shows the ESEM morphologies of microcapsules (core/shell ratio of 3/1) with different mean sizes fabricated



**Figure 11.13** Optical morphologies of microcapsules containing rejuvenator fabrication process: (A) rejuvenator dispersed by ESEM under a 3000 r/min emulsion stirring rate, (B) microcapsules with core/shell ratio of 2/1, and (C) dried microcapsules with regular globe shape with smooth surfaces.



Figure 11.14 ESEM morphologies of microcapsules containing rejuvenator fabricated under emulsion stirring rates of (A) 1000 r/min, (B) 2000 r/min, and (C) 3000 r/min.



Figure 11.15 Shell thicknesses of microcapsules fabricated under various stirring rates.

under emulsion stirring rates of 1000, 2000, and 3000 r/min. It can be seen that the mean sizes decreased sharply with the decreasing of stirring rates because higher stirring rates will smash the oily rejuvenator into smaller droplets. The rejuvenator applied in this study as core material still obeys this rule. Fig. 11.15 shows shell thickness data of microcapsules. The results suggest that the shell thickness is not greatly influenced by the stirring rates. As the formation of a shell is a coacervation process, the shell thickness is dominated by the amount of shell material. Less core/ shell ratio leads to a higher shell thickness value. It can be concluded that the core/ shell ratios nearly do not affect the average size of microcapsules. In other words, the mean size is primarily decided by the core material disperse speed. Therefore, a

typical microcapsule sample (fabricated with 3000 r/min and core/shell ratio of 2/1) was applied to investigate the self-healing mechanism of aged bitumen.

#### 11.3.2 Thermal analysis of microcapsules

TGA has been extensively applied to investigate the encapsulation effect and shell compactness of microcapsules. The microcapsule sample was composted with increasing temperature according to present residual weight (wt.%). It has been reported in our previous work that this rejuvenator has a marked weight loss between 337°C and 469°C owing to the evaporation of oil substance under elevated temperature. Fig. 11.16A shows the typical TGA curve of microcapsule sample fabricated under emulsion rate of 3000 r/min and core/shell ratio of 2/1. The TGA curve may reflect thermal stability and structure of polymeric shell. Fig. 11.16B and C show SEM morphologies of microcapsules under temperatures of 100°C, 300°C and 400°C. It can be seen that the microcapsules still keep the global shape under 300°C without break. Nearly about 10% weight is lost less than 100°C for all microcapsule samples because of the evaporation of water and small molecules. The decomposite temperature is at about 396°C (Fig. 11.16D). This degradation temperature is higher than the melting temperature of bitumen at 180°C. It indicates that the cured MMF resin will not be thermally decomposed during mixing with melting asphalt. With the increasing of temperature, the shell will first crack or be broken under high temperature before decomposition.



**Figure 11.16** Thermal stability of microcapsules: (A) the TGA plot of pure rejuvenator and (B–D) SEM morphologies of microcapsules under various temperatures.



Figure 11.17 Optical morphologies of microcapsules in melting bitumen under different temperatures for 30 min, (A) 140°C, (B) 160°C, (C) 180°C and (D) 200°C.

To verify the thermal stability of microcapsules in bitumen, the microcapsules were mixed with melting bitumen under various temperatures for 30 min. Then the shape of microcapsules was observed by using an optical microscope. The microcapsules had an average size of about 16  $\mu$ m fabricated under core material stirring rate of 3000 r/min (core/shell ratio of 2/1). In Fig. 11.17A, it can be seen that microcapsules survived in bitumen under the temperature of 140°C. With the increasing of temperature to 160°C (Fig. 11.17B), 180°C (Fig. 11.17C) and 200°C (Fig. 11.17D), the microcapsules still kept their global shape without crack and thermal decomposition. These results indicate that microcapsules can resist the thermal effect of bitumen in application.

#### 11.3.3 Capillarity behaviors of rejuvenator in microcracks

In order to figure out more details of microcracks generation and the capillarity behaviors of rejuvenator, a bitumen/microcapsules composite (5/95, w/w) was observed continuously by using a fluorescence microscope under the temperature of 0°C. The microcapsules were fabricated under core material stirring rate of 3000 r/ min with core/shell ratio of 2/1. As shown in Fig. 11.18A–C, a microcrack was generated by liquid N<sub>2</sub> with a width of about 10–15  $\mu$ m. It propagated successfully



**Figure 11.18** The capillarity behaviors of rejuvenator in self-healing bitumen by microcapsules: (A) a microcrack was generated by liquid  $N_2$  with the width of about  $10-15 \,\mu$ m, (B and C) the microcrack propagated and pierced the shells of microcapsules, (D and E) the liquid of rejuvenator leaked out from microcapsules and flowed into the microcapsules, and (F) movement trace and direction of rejuvenator during the capillarity.

and pierced the shells of microcapsules. We can distinguish rejuvenator and bitumen through the colors in fluorescence microscope images. Rejuvenator has green color and bitumen is yellow. As can be seen in Fig. 11.18D and E, liquid rejuvenator leaks out from microcapsules and flows into the microcrack. During a period of time, rejuvenator fills the microcrack and then spread the whole microcrack through capillarity. Especially in Fig. 11.18F, we can clearly recognize the movement traces and direction of rejuvenator during the capillarity. To confirm the existence of the capillarity, we compared the states of microcrack in 60 min under 0°C. Fig. 11.19A shows the original state of a microcrack in bitumen/microcapsules sample with a width of 20  $\mu$ m. The crack was an arc curve in the sample. Interestingly, the crack color changes to green during 60 min (Fig. 11.19B) because it has been filled with rejuvenator coming out from broken microcapsules.

Besides the confirmation of the capillarity, we also want to evaluate the extent and rate of rejuvenator through capillarity in microcracks. It is well known that the height L of a liquid column of capillarity is given by Eq. (11.5),

$$L = \frac{2\gamma \cos\theta}{\rho g r} \tag{11.5}$$

where  $\gamma$  is the liquid-air surface tension,  $\theta$  is the contact angle,  $\rho$  is the density of liquid, g is the local gravitational field strength, and r is the radius of tube (crack). However, this rule cannot guide capillarity in self-healing bitumen. Because the microcapsules were homogeneously distributed in bitumen, the rejuvenator could


**Figure 11.19** Fluorescence microscope morphologies and capillarity behavior of rejuvenator in a microcrack. The states of a microcrack in bitumen/microcapsules sample with a width of  $20 \,\mu\text{m}$  before (A) and after (B) 60 min. The microcrack has been filled with rejuvenator out of microcapsules.

quickly disperse from both sides of a crack. Therefore, we pay more attention to the parameter of time (t), which means the period of rejuvenator filling the whole microcrack. When a dry porous material such as a brick or a paper is brought into contact with a liquid, it will start absorbing the liquid at a rate that decreases over time. For a bar of material with cross-sectional area A that is wetted on one end, the cumulative volume V of absorbed liquid after a time t is

$$V = AS\sqrt{t} \tag{11.6}$$

where S is the sorptivity of the material. As rejuvenator is oily liquid, it may take a longer time for it to disperse due to its high viscosity. Another issue needs to be considered is the volume of microcapsules in bitumen. With higher volume ratio of microcapsules ( $V_m$ ), the microcrack will, at the same time, have a higher probability to meet microcapsules. From the summary of the above mentioned laws, a conclusion can be drawn that the capillarity time (T) for self-healing microcapsules is determined by three aspects: the properties of liquid (M, related to viscosity and contact angle), the volume of cracks (V', related to width and length), and the volume ratios of microcapsules in the medium. T can be written as an equation:

$$T = f(M, V', V_m)$$
 (11.7)

To simplify this complex problem, bitumen/microcapsules samples were prepared with various  $V_m$  values to measure the capillarity speeds of rejuvenator. The



**Figure 11.20** The capillarity speeds of rejuvenator in bitumen microcracks under temperature of 0°C: (A and B) the illustrations of the lateral movement and longitudinal movement, (C) the relationship between the lateral movement time and the volume of microcapsules (5, 10, 15, 25, and 30 vol.%) in microcracks (width: 10, 20, and 30  $\mu$ m), and (D) the relationship between the longitudinal movement time and the volume of microcapsules (5, 10, 15, 25, and 30 vol.%) in microcracks (length: 100, 200, and 300  $\mu$ m).

movement time of rejuvenator is measured to determine the speed of the capillarity using the scale of fluorescence microscope.

We investigated both the speeds of the lateral movement (Fig. 11.20A) and longitudinal movement (Fig. 11.20B) in microcracks under temperature of 0°C. The width and length data in Fig. 11.8 were measured by a fluorescence microscope with scale. In Fig. 11.20A and B, the arrows point the directions and tracks of rejuvenator movement. Fig. 11.20C shows the relationship between the lateral movement time and the volume of microcapsules (5, 10, 15, 25, and 30 vol.%) in microcracks (width: 10, 20, and 30  $\mu$ m). The time data were carefully recorded by observing the rejuvenator movements under a fluorescence microscope with a scale. First, it can be known that rejuvenator needs more time to complete a lateral movement in wider microcracks. For example, the times are 67, 54, and 46 min for samples with 5 vol.% microcapsules in microcracks with widths of 30, 20, and 10  $\mu$ m, respectively. Second, higher volume ratio of microcapsules leads the rejuvenator to take less time for a lateral movement. The reason may be that more rejuvenator has released out into microcracks and the releasing area is improved. Fig. 11.20D shows the relationship between the longitudinal movement time and the volume of microcapsules (5, 10, 15, 25, and 30 vol.%) in microcracks (length 100, 200, and 300  $\mu$ m). Higher volume ratio of microcapsules also leads the rejuvenator to take less time to complete a longitudinal movement. At the same time, it has been seen that a longer microcrack makes the rejuvenator to take more time to complete a long-distance movement. However, higher volume ratio of microcapsules (>25 vol.%) will weaken this trend. We can attribute this phenomenon to the mass increase of rejuvenator in a microcrack.

### 11.3.4 Observation of rejuvenator diffusion in aged bitumen

Mass transfer by molecular diffusion is one of the basic mechanisms in many branches of science. Molecular diffusion is a transport property, which monitors the rate of mass transfer of species in a medium [56]. In addition, it was reported that the diffusion behaviors of bitumen have a great relationship with temperature, viscosity of the diffusion medium, diffusant size, and polarity [57]. In this study, we only observe the diffusion processes in order to design and plan microcapsules-based bitumen recovery processes, because it is required for determining the rate of mass transfer between the rejuvenator and aged bitumen at given conditions. The diffusion coefficient between liquid—solid phases is mainly determined by liquid molecular structure, solid structure, temperature, and pressure. Fig. 11.21 shows the fluorescence microscope morphologies of rejuvenator leaked out from microcapsules diffusion in aged bitumen at 0°C for 24 h. A microcrack was generated in this aged bitumen sample previously. It can be confirmed in Fig. 11.21A that the microcapsules were broken by the microcrack and the rejuvenator flowed out. The



**Figure 11.21** Fluorescence microscope morphologies of rejuvenator diffusion in aged bitumen at room temperature for 24 h.

diffusion areas were also observed after a period of 24 h. Interestingly, we even can identify the diffusion direction of rejuvenator coming out of a microcapsule into the aged bitumen as seen in Fig. 11.21B. A gradient diffusion layer exists between the rejuvenator and the matrix. Normally, diffusion behavior of binary materials is reflected in its phase diagram. The composition of the phases is dependent on the phase structures and the temperature, the phase structures being the border between the one- and two-phase regions. Therefore, it can be concluded that various conditions including the aging degree of bitumen and the diffusion temperature. The diffusion coefficient will be discussed in our subsequent research.

### 11.3.5 Properties of virgin and rejuvenated bitumen

In addition to an analysis of rejuvenator diffusion, we wished to evaluate the results of rejuvenating aged bitumen. Efficiency of the rejuvenator depends on its viscosity and the quantity added to the aged bitumen and thus we decided to conduct a preliminary investigation to explore the effects of blending known quantities of rejuvenator with penetration grade bitumen on binder rheology. To this purpose, a series of microcapsules/bitumen samples were first heated at 200°C for 12 h and then stored for 60 days. The microcapsules applied in this test had an encapsulation ratio of 85% with an average size of approximately 20  $\mu$ m and had been fabricated under conditions of stirring rate of 3000 r/min and core/shell ratio of 1/2. Based on the previous investigation results, it was predicted that the majority of the encapsulated rejuvenator would leak out of the spheres and penetrate into the aged bitumen. The subsequent rejuvenating effect was assessed by comparing the properties of aged bitumen samples under the same conditions.

In this study, the original bitumen (80/100) was found to have penetration, softening point, and viscosity values of 86 d-mm, 46.7°C, and 325 mPa s, respectively. In contrast, the aged bitumen (40/50) had values of 43 d-mm, 53.5°C and 578 mPa s, respectively. The aged bitumen was subsequently rejuvenated with microcapsules at 2%-10% by weight of bitumen. Table 11.2 lists the penetration, softening point, and viscosity of the aged bitumen before and after rejuvenation with varying amounts of microcapsules. These data demonstrate that the penetration value increased as the amount of microcapsules in the aged bitumen increased.

This result is attributed to a reduction in the ratio of asphaltenes to maltenes. The addition of approximately 10% microcapsules returns the aged 40/50 bitumen to a condition similar to that of the original bitumen. Additionally, the softening point value decreased with the addition of microcapsules. In the aged bitumen, increases in the level of high molecular weight asphaltenes tend to produce a harder material with lower temperature susceptibility and thus increase the softening point. The addition of the rejuvenator, however, is evidently capable of mitigating this effect. Lastly, although the aged bitumen had a high viscosity value, the addition of 10% microcapsules returned the viscosity to 390 mPa s. Therefore the same workability is expected from the rejuvenated bitumen.

	Original bitumen	Aged bitumen (40/50)	Rejuvenated aged bitumen with microcapsules (wt.%)						
	(80/100)		2	4	6	8	10	12	
Penetration (d-mm, 25°C)	86	43	50	63	64	70	76	80	
Softening point (°C)	46.7	53.5	51.0	50.3	49.2	47.1	46.6	45.4	
Viscosity (135°C) (mPa s)	325	578	573	552	500	470	406	390	

**Table 11.2** Properties of virgin and rejuvenated bitumen (penetration value, softening point value, and viscosity value).

## 11.4 The mechanism of multi-self-healing of microcapsules

Multi-self-healing behavior can thus refer to a repeated wetting and diffusion process. The diffusion rejuvenator softens the aged bitumen such that the bitumen has a reduced probability of cracking again. For this mechanism of self-healing, it is well known that the microcrack trigger depends on the degree of bitumen softness and the breaking of a microcapsule depends on the point stress value of the microcrack. Microcapsules may therefore break at different times during the bitumen aging history. The remaining microcapsules near the microcrack can then leak rejuvenator later when another microcrack is generated with an even higher crack-tip strain. It has previously been reported that microcapsules containing rejuvenator can be fabricated with various shell thicknesses and size distributions [51]. Interestingly, nanoindentation tests have revealed that a single microcapsule can undergo elastic—plastic deformation [55]. The size and shell thickness are both important influencing the factors of the micromechanical properties of the microcapsules. Therefore, we may realize multi-self-healing by adjusting the shell thickness and size distribution of microcapsules containing rejuvenator.

Besides investigating the potential performance and suitable conditions when applying microcapsules, we still need to establish an approach of demonstrating that the microcapsules actually provide a recovery action when applied to multiself-healing bitumen. The self-healing of bitumen is a complex process and strongly depends on the rest time between two load pulses, temperature, crack phase, and material type. The healing phenomenon of bituminous materials involves viscoelastic healing and viscous healing [58]. Moreover, viscoelastic healing is related to the delayed elastic recovery behavior of bituminous materials, while the viscous healing is related to the viscosity of bituminous materials. It was also observed that viscoelastic healing is much faster than viscous healing [58]. The viscoelastic healing greatly depends on the temperature. Higher temperature may accelerate the healing process. In many cold regions and countries, thermal or low-temperature cracking (microcracks) is one of the primary modes of asphalt pavement failure. However, it remains difficult to identify the behavior fully because environmental and geometrical factors, such as stiffness, consistency, and temperature susceptibility of the bitumen, affect low-temperature cracking.

To simplify the complex problem, we employed a method referred to as the beam on elastic foundation (BOEF) method to investigate the cracking and healing behaviors of bitumen [59]. Qiu [58] also noted that the BOEF method is a practical method of investigating the recovery of strength and crack opening displacement (COD). In the BOEF method, a notched bitumen beam is glued onto a low-modulus rubber foundation, and a symmetric monotonic load is applied with loading-unloading-healing-reloading cycles. The rubber foundation is used to avoid permanent deformation and to ensure a controllable healing process. The asphalt beam is glued onto the rubber foundation to simulate full contact and full friction. When no glue is used, partial slipping can occur between the beam and rubber. The rubber elastic foundation can absorb most of the deformation. In addition, the elastic foundation can help close the crack during unloading and thus support the healing of the bitumen.

### 11.4.1 Observation of rejuvenator movement in bitumen

A thin bitumen/microcapsule sample was spread onto a glass sheet. It was carefully poured with a drop of liquid nitrogen (N<sub>2</sub>) at one end. Microcracks were quickly generated in this sample because of the low-temperature brittleness. Capillarity and diffusion behaviors of the rejuvenator in the aged bitumen were observed by a fluorescence microscope (CKX41-F32FL, OLYMPUS) using the light characteristics such as reflection, diffraction, and refraction. The scale of the fluorescence microscope was applied to measure the extent of capillarity and diffusion. As bitumen is a temperature-sensitive material, the observation was in an environment of  $0^{\circ}$ C.

#### 11.4.2 BOEF setup

An improved BOEF setup was used in this study to evaluate the crack propagation and self-healing behaviors of pure bitumen. Fig. 11.22A illustrates the BOEF sketch map with design size to explain its working principle. The testing beam comprised two rectangular aluminum blocks and a bitumen center. The bitumen had a rectangular shape notch ( $5 \times 4.3$  mm, width  $\times$  length) fabricated by means of a silicon mold under a temperature of 0°C as shown in Fig. 11.22B. The two-end aluminum blocks in the beam were then glued on a rubber foundation in order to simulate full contact and full friction. Two rolling devices were under the aluminum blocks.

A hard wood support was used, connected to the rubber (shore hardness of 40 degrees, Poisson's ratio of 0.5) and steel base by glue. The load was applied in the



**Figure 11.22** Beam on elastic foundation (BOEF) setup applied to measure the cracking and healing behaviors of bitumen: (A) illustration of the BOEF, (B) the notch on the axis line of bitumen sample, (C) the size of aluminum block (length  $\times$  height), (D) bitumen size (length  $\times$  height), (E) the size of aluminum block (width  $\times$  height), and (F) the size of the notch in bitumen.

middle of the bitumen through an iron stick perpendicular to the beam. Then a crack was generated from the notch. The load-displacement curves were recorded. In order to easily observe the crack, one side of the bitumen was daubed with white paint. When the load was removed away, the crack in the bitumen closed because of the elastic rubber foundation. The beam was placed under a controlled temperature for 24 h. After the healing period, the beam was reloaded at a temperature of 0°C under the same conditions. Fig. 11.22C-F shows the size of the BOEF setup and bitumen with a notch. The aim of the test was to analyze the influence of rejuvenator diffusion on mechanical properties of aged bitumen. The key feature of the BOEF is its use of a notched bitumen beam glued on a low-modulus rubber foundation; a symmetric monotonic load was applied with loading-unloading-healing-reloading cycles.

### 11.4.3 Multi-self-healing tests

A monotonic BOEF test with loading-unloading procedure was used to analyze the self-healing properties of bitumen. Within the BOEF test setup, a symmetric mono-tonic load was applied with a loading-unloading-healing-reloading cycle. A crack

was generated under pressure at 0°C. When the load level had returned to zero, the external load applicator was removed. Then the BOEF beam was placed into a silicon mold, to avoid deformation of bitumen, at a controlled temperature for 24 h to allow the self-healing process. After healing, the bitumen beam was again subjected to a controlled reloading at 0°C to generate a crack.

## 11.4.4 Mixture of microcapsules and bitumen

Besides the stability of microcapsules, the thermal stability of the microcapsules in bitumen also needs to be investigated. In Fig. 11.23, microcapsules (Type D) were dispersed homogeneously in bitumen (5.0 wt.%) keeping their original global shape. They had survived in the melting bitumen without destruction and thermal



**Figure 11.23** Type D microcapsules dispersed homogenously in bitumen (5.0 wt.%) keeping the original global shape under temperature of (A) 200°C, (B) 100°C, and (C) 20°C.

decomposition. In Fig. 11.23A, microcapsules are shown, resisting a temperature of  $200^{\circ}$ C for 10 min in melting bitumen. Fig. 11.23B and C shows the morphologies of bitumen samples at  $100^{\circ}$ C and  $20^{\circ}$ C. Nearly all microcapsules had their original shape without thermal decomposition. This phenomenon is consistent with the above thermal stability conclusions.

Another issue we need to consider is the interface condition between microcapsules and bitumen. The addition of particulate fillers into a polymeric matrix has a significant influence on its mechanical properties. Fillers may lead to fracture toughness increase by multiple phenomena including, but not limited to, crack pinning, crack bridging, microcracking, and crack deflection [60]. In addition, it has been reported that microcracks and interface separation may occur during fabrication of microcapsules/matrix composites [61]. The expansion and shrinkage of the microcapsules and polymeric matrix may occur due to the different expansion coefficients. All these phenomena may cause microcracks or fractures in the matrix during heat absorption or resealing, damaging the thin microcapsule shell. As a result, the encapsulated rejuvenator will lose its protective shell. Fig. 11.24 shows the cross-sectional ESEM morphology of the interface bonding between microcapsules (wt.20%) and bitumen. In this image, some microcapsules were piled together because of higher weight ratio of microcapsules in bitumen. Even so, the interphase can be distinguished between the shells and bitumen. The microcapsules still keep their globular shape with compact shells. At the same time, the interphase is tight without leaks or cracks. The interface stability may be attributed to MMF molecules possessing a relatively flexible nature, allowing it to adjust its molecular arrangement decreasing microstructure defects such as cracking, debonding, and gap formation [61].



Figure 11.24 ESEM morphology of the interface bonding between microcapsules and bitumen.

### 11.4.5 Observation of the self-healing process

Before investigating the multi-self-healing behavior, we must confirm that the healing of microcapsule/bitumen composites is coming from the microcapsules and not from the natural ability of bitumen. A microcrack therefore was generated by liquid  $N_2$  with a width of less than 10  $\mu$ m to help simulate the self-healing process. The microcrack propagated successfully and pierced microcapsules along the development track and the shells of microcapsules were broken by the microcrack as shown in Fig. 11.25A-C. Then liquid rejuvenator leaked out from microcapsules and flowed into the microcrack. After a period of time, rejuvenator filled the microcrack and then spread along the whole microcrack through capillarity. Especially in Fig. 11.26C, we can clearly recognize the movement traces and direction of rejuvenator induced by capillarity. Because the microcapsules were homogeneously distributed in the bitumen, the rejuvenator could quickly disperse from both sides of a crack. Mass transfer by molecular diffusion is one of the basic mechanisms in many branches of science. Molecular diffusion is a transport property, which controls the rate of mass transfer of species in a medium. However, the diffusion behavior of bitumen has a significant relationship with temperature, viscosity of the diffusion medium, diffusant size, and polarity [60]. In Fig. 11.26D, the diffusion areas were also observed. We can even identify the diffusion direction of rejuvenator coming out of a microcapsule into the aged bitumen. In our future work, the diffusion kinetic theory will be investigated, which is determined by the rate of mass transfer between the rejuvenator and aged bitumen under given conditions. Moreover, the diffusion coefficient between liquid-solid phases will be calculated considering the factors of liquid molecular structure, solid structure, temperature, and pressure.



**Figure 11.25** Morphologies of microcrack propagation in bitumen: (A) the fracture surface of microcrack with broken shell of microcapsules; (B and C) microcrack propagated successfully and pierced microcapsules along the development track.



**Figure 11.26** Fluorescence microscope morphologies of rejuvenator movement in aged bitumen: (A) microcrack in microcapsule/bitumen composite, (B) the shells of microcapsules broken by microcracking, and (C and D) the movement traces and direction of rejuvenator induced by capillarity.

### 11.4.6 Mechanical tests of multi-self-healing behaviors

It has been reported that the self-healing behavior of bitumen is greatly dependent on the size of crack [58]. COD is an important index in fracture mechanics. Numerical methods and experimental methods are often used to find COD values of cracked structures [58,59]. Based on this knowledge, four COD levels (0.2, 0.4, 0.6, and 0.8 mm) had been selected in previous work, which were applied and the bitumen subjected to a healing period afterward; the crack generation process was carried out in a BOEF test at 0°C with a COD speed ( $S_{COD}$ ) of 0.02 mm/s [59]. It was found that the crack length grew with the development of the COD, and no visible crack could be observed at COD values less than 0.2 mm. To simplify the above tests, an example of the development of the crack at a COD value of 0.8 mm was applied in this study as shown in Fig. 11.27A and B. In order to determine the microcrack propagation length and speed, a camera recorded the scene on video with a scale. In Fig. 11.27C, it can be seen that the crack is visible with a length of about 8 mm and the microcapsules have been broken by this microcrack. The following phenomena were observed: (1) in the beginning of the crack generation phase, the BOEF beam was bending with viscoelastic behavior; (2) in the crack



**Figure 11.27** Beam on elastic foundation test to measure the cracking and healing behaviors of bitumen: (A) the crack beginning propagation, (B) the development of the crack at a COD value of 0.2 mm, and (C) optical morphology of microcapsules broken by a microcrack.

propagation phase, the crack length increased quickly; and (3) in the unloading process, the crack closed with nearly zero COD value due to the elasticity of the rubber foundation of the BOEF setup.

We have reported that the self-healing capability from the monotonic reloading response is dependent on the healing time and healing temperature [59]. In order to analyze the multi-self-healing ability, a test with loading-unloading-loading cycles was designed to prove the multirecovery of the mechanical properties. Fig. 11.28A shows the load-displacement curves of the BOEF test at 0°C for bitumen samples containing 2.0 wt.% microcapsules. Curves 1235 are the self-healing first cycle, second cycle, third cycle, and fourth cycle, and ④ is the load-strain curve of pure aged bitumen without microcapsules. For each sample, there was an interval of 10 days between the self-healing cycles. For the 123 self-healing cycles, the maximum load was beyond the load value of pure aged bitumen (4). With increasing number of self-healing cycles (123) from one to three times, the maximum load required decreased. The elastic recovery performance and workability are almost equivalent. This indicates that the aged bitumen nearly recovered its mechanical properties. The reason is that the rejuvenator coming out of the microcapsules wetted and penetrated into both sides of microcrack and improved the bonding ability of two surfaces. When the sample had a fourth cycle (5), it could not recover its original load. This may be due to there not being enough rejuvenator



**Figure 11.28** Load-strain curves of BOEF test at 0°C, bitumen samples containing 2.0 wt. % microcapsule healed at (A) 0°C and (B) 25°C for 24 h bitumen samples; self-healing cycles: 1 first cycle, 2 second cycle, 3 third cycle, 4 aged bitumen, and 5 fourth cycle.

between the two sides of the microcrack, which decreased the bonding strength of the interface. Fig. 11.28B shows the load-strain curves of bitumen samples containing 2.0 wt.% microcapsules at 0°C, which had been healed at 25°C for 24 h. Curves (1)(2)(3)(5) are the self-healing cycles, and (4) is the load-strain curve of pure aged bitumen. For self-healing cycles ((1)(2)(3)), each load is larger than the pure aged bitumen, the same trend appears at a temperature of 0°C. However, the slope of the curves decreased faster than the curves in Fig. 11.10A. This means that under higher healing temperature, the rejuvenator between the two sides of the microcrack has a higher permeation rate. Less rejuvenator or low concentration of rejuvenator in microcrack area will also decrease the bonding strength of interface.

The success of crack closure is strongly dependent on how efficiently the crack tip is shielded from the applied conditions. To analyze the relationship between self-healing time and temperature, the COD values were tested at 0°C as shown in Fig. 11.29. All samples had a 0.8 mm initial COD value and the healing cycles were carried out at 0°C or 25°C. The sample with healing cycles at 25°C had a relatively faster speed of crack healing than that at 0°C. This is attributed to the rejuvenator having a larger diffusion rate into bitumen at higher temperature. The reloading was applied after healing for sufficient time or at sufficiently high temperature, and the reloading curve shows an increased reloading strength because of the viscous healing process consisting of flow, wetting, and diffusion [58]. It must be mentioned that if the loading is applied immediately, no viscoelastic healing will occur. The healing time was 45, 23, 14, 10, and 7 h at 0°C during healing cycles 1-5. This indicates that more self-healing cycles made the bitumen sample to take more time to close the crack under the same temperature.



**Figure 11.29** COD-time curves of bitumen samples (COD: 0.2 to 0.8 mm) of multi-selfhealing process under temperature of 0°C and 25°C.

### 11.4.7 Properties of virgin and rejuvenated bitumen

Based on the investigation results, it is predicted that the majority of the encapsulated rejuvenator can leak out of the spheres and penetrate into the aged bitumen [52,62]. The action of subsequent rejuvenation can be assessed by comparing the properties of virgin and rejuvenated bitumen samples under the same conditions [58]. Efficiency of the rejuvenator depends on its viscosity and the quantity added to the aged bitumen. Table 11.3 lists the penetration, softening point, and viscosity of the original bitumen and aged bitumen before and after being rejuvenated with fourtime self-healing cycles. The original bitumen (80/100) has penetration, softening point, and viscosity values of 86 d-mm, 46.7°C, and 325 mPa s, respectively. In contrast, the aged bitumen (40/50) has values of 43 d-mm,  $53.5^{\circ}C$  and 578 mPa s, respectively. A series of bitumen samples with 2.0 wt.% microcapsules were heated to 200°C for 12 h and then stored for 60 days in each cycle. Then the aged bitumen was subsequently rejuvenated. It can be seen that the properties of aged bitumen have been partly returned to its original state after the healing cycles. In other words, the aged 40/50 bitumen returned to a condition similar to its original state through the rejuvenator, reducing the ratio of asphaltenes to maltenes. In the aged bitumen, with the increase of molecular weight, asphaltenes produces a harder material with lower temperature susceptibility and thus increases the softening point. The addition of the rejuvenator, however, is evidently capable of mitigating this effect. Usually, material properties degrade over time due to the initiation of damage on a microscopic scale, which tends to grow and will ultimately lead to failure of the original material structure. According to these analyses, a conclusion is drawn that the multiself-healing ability can partly repair the damage of bitumen during service life.

### 11.4.8 Mechanism analysis of multi-self-healing

Researchers have already given attention to characterizing the self-healing phenomenon at various scales from bituminous binders to asphalt in the fields of

	Original	Aged bitumen (40/50)	Multi-self-healing cycles						
	bitumen (80/100)		1	2	4	6	8	10	
Penetration (d-mm, 25°C)	86	43	50	60	66	71	72	75	
Softening point (°C)	46.7	53.5	51.0	50.2	49.7	49.1	46.1	47.0	
Viscosity (135°C) (mPa s)	325	578	573	510	450	340	330	300	

 Table 11.3 Properties of virgin and rejuvenated bitumen (penetration value, softening point value, and viscosity value).

mechanical experimentation and materials modeling [58]. The stiffness and strength of bitumen materials decrease when they are exposed to sunshine and load repetition. It has been realized that the damage includes the steps of microcrack initiation, propagation, and macro-cracking during cyclic loading. The recovery of material stiffness, the extension of fatigue life, and the recovery of strength were for the first time experimentally tested in the 1960s [63]. From then on, the self-healing phenomenon of bitumen was defined as the recovery properties and diminishing cracks. A literature review shows that two models can be applied to the self-healing properties of bituminous materials. One is the mechanical healing model, which is a regression model to explain the healing in macroscopic continuum damage [64]. Another model is called the physicochemical theory, which is based at a molecular level to explain the healing process. As microcapsules containing rejuvenator have been considered as a positive product to instigate recovery of the aged bitumen [55,59], it is necessary to give a mechanism analysis of multi-self-healing using the above two models. It is helpful to understand the self-healing process or regulate the self-healing efficiency.

As bituminous material is a viscoelastic polymeric material, it has a timetemperature dependent behavior. From the analysis in Figs. 11.10 and 11.11, it has been confirmed that higher temperature improves the healing speed. In order to simplify the understanding of the relationship between time and healing behavior, only the healing percentage under 0°C was investigated. Fig. 11.30 shows the mechanical property recovery of multi-self-healing microcapsules/bitumen composites tested by the BOEF procedure including aging and healing based on the above results (Fig. 11.28 and Table 11.3). It must be mentioned that this illustration does not show real data on bitumen properties, but a property tend according to the above analysis. The self-healing percentage is evaluated by the refracture strength of the healed sample divided by fracture strength of the original sample as shown in Eq. (11.8),

$$SH = \frac{S_{ref}}{S_f} \tag{11.8}$$

where *SH* is the self-healing ability as a percentage of the original fracture strength,  $S_f$  is the strength of the original fracture of sample, and  $S_{ref}$  is the strength of the refracture of the sample. Comparing the various rounds of self-healing cycle with loading and unloading as shown in Fig. 11.30, the following observation can be derived:

- In the first healing cycle, the aged bitumen has a recovery with the help of microcapsules with rejuvenator. The rejuvenator has made the aged bitumen softer. Even though, its property still cannot return 100% to its original state. This means that the microcapsules only partly healed the bitumen.
- With increasing number of self-healing cycles, the recovery percentage decreases. This means that its self-healing ability is being lost.
- The microcapsules/bitumen sample has a multi-self-healing ability. When its strength is lower than a limit point, the aged bitumen cannot recover its properties anymore and the self-healing ability disappears.



Figure 11.30 Mechanical property recovery of self-healing microcapsules/bitumen composite tested by the BOEF procedures including aging and healing.

• Moreover, the recovery time also increases with increasing self-healing cycles. This indicates that the microcrack needs more time to heal with increasingly aged bitumen.

These changes of self-healing ability can be explained by the Christensen–Anderson model [65],

$$\log \alpha_T = \frac{\Delta E_a}{2.303R} \left( \frac{1}{T} - \frac{1}{T_0} \right) \tag{11.9}$$

$$H(t,T) = 100 \times \left[1 + \left(\frac{m}{t \times \alpha_T}\right)^{\frac{\log 2}{n}}\right]^{\frac{d}{\log 2}}$$
(11.10)

where a, b are model parameters;  $a_T$  is a time-temperature superposition shift factor;  $\Delta E$  is apparent activation energy (unit: J/mol); and R is universal gas constant [8.314K/(mol K)]. From this model, we know that the increment is not linear with healing time under the same temperature. With increasing degree of aging, its viscoelastic temperature is also increasing. The aged bitumen needs more energy to close the crack. In this study, the crack closure is a phase change or physicochemical reaction under a certain temperature between the sides of the crack interface. The molecules cannot have a reaction or molecular entanglement under a lower temperature. On the other hand, lower energy cannot also activate crack closure because there is not enough energy to feed the healing process. Besides the temperature, time also has a great effect on the healing ability. Qiu [58] has found that after an immediate reloading, the bitumen sample usually cannot return to the original state but to a point with lower strength. In this study, all samples had enough time to rest before the next loading process. The molecules at the interface have enough time to regulate states or reactions; the rejuvenator has enough time to move (capillarity and diffusion). Through analysis of the recovery properties, we know that the crack healing can be considered as a viscosity-driven process. But the completion of crack closure does not mean total recovery of the strength of bitumen. Better healing may be achieved with a longer healing time.

Besides the regression model to explain the healing in macroscopic damage terms, we can explain the multi-self-healing behavior using molecular movement theory, which has been called the theory of physicochemical theory. Penetration value and softening point are normally used to identify the physical properties of bitumen. It has been determined that softer bitumen has higher self-healing capability because of its higher penetration value and lower softening point [66]. Moreover, it is believed that the chemical structure of the aged bitumen has a great influence on its healing ability [67]. Bitumen can be regarded as a colloidal system consisting of high molecular weight asphaltene micelles dispersed or dissolved in a lower molecular weight oily medium [68]. In this complex system, a so-called thixotropic behavior has been reported, which is similar to other colloids or rheopectic fluids. The thixotropic effect on the stiffness decreases during fatigue and recovery during rest periods for aged bitumen [69]. In addition, thixotropy is highly

related to the colloidal nature of bitumen. Thixotropy is also considered to be related to the changes in bitumen's microstructure. For example, Shan et al. [70] reported the thixotropy model to analyze fatigue and healing behaviors using breakdown and building-up coefficients of the microstructure. It has been proved that thixotropy does play a role in the healing character of bitumen. With the increasing of service time, bitumen loses its thixotropy because of disappearance of some smaller molecules. By adding oily rejuvenator, the viscosity of bitumen will increase to its initial value due to the movement of molecules. In this process, the penetration and diffusion play a role in determining the microstructure of bitumen. The diffusing ability of rejuvenator into aged bitumen can be enhanced with the increase of temperature and time; however, the diffusing of rejuvenator into aged bitumen is restricted due to the volatilization of light components and aging of rejuvenator under high temperature [71].

#### 11.4.9 Hypothesis and future work

Rejuvenator is an oily liquid used to recover the aged bitumen properties through the penetration and diffusion process. Microcapsules help the rejuvenator by providing a larger diffusion area than the normal method of applying rejuvenator on the surface of bitumen. Aged bitumen responds to the multi-self-healing ability of rejuvenator leaking out of microcapsules. Based on these facts, we propose a hypothesis describing the details of multi-self-healing in this microcapsule/bitumen system: the microcapsules can be broken at various times during the aging process of bitumen because the microcapsules have different shell thicknesses and sizes. This will provide a continuous supply of rejuvenator into aged bitumen over a long service history. With a constant change and adjustment of microstructure, viscosity, and thixotropy of bitumen, it will possess a healing ability for numerous times.

To visually explain this hypothesis and our future work, an illustration was applied to describe the whole process of multi-self-healing of a microcapsule/bitumen composite. As mentioned earlier, the self-healing process of bitumen can be considered as crack generation, crack closure and strength recovery. Fig. 11.31A shows a microcrack generation and a model of tip stress field. The cracks are all produced in a controlled way in this study to measure their size and shape. Then the microcapsules are broken by the tip stress (Fig. 11.31B). After a rest time, another crack may again generate following the same path. Due to the penetration and diffusion of rejuvenator, the bitumen near the crack has been softening. The self-healing capability still exists under a certain healing temperature and time [72]. However, the probability is low that another crack may again generate following the same path. The reason is that the viscoelastic behavior of bitumen will resist the damage in the wetting and diffusion area. It means that another crack will generate in another area without or with less rejuvenator. As microcapsules have a polydispersion for size and shell thickness, the microcapsules can break in various periods or conditions. Even when a crack generates, the microcapsules do not release all rejuvenator due to various shell strengths of microcapsules. All these results give the aged bitumen a multi-self-healing property, namely crack generation and crack



**Figure 11.31** Illustration of multi-self-healing ability for microcapsule/bitumen composite: (A) the crack-tip strength, (B) a microcrack generation, (C) a microcrack following the same trance of the last one, (D and E) a microcrack following the same trace of the last one, (F) a microcrack broken nearby microcrack in the diffusion area, and (G) the aged bitumen final lost healing ability.

closure, as shown in Fig. 11.31D–F. Finally, the aged bitumen loss its healing ability after a long service time when microcapsules have been exhausted and the rejuvenator has disappeared or been lost (Fig. 11.31G). This hypothesis is presented here to stimulate future work.

## 11.5 States of microcapsules in asphalt binder

Bitumen is a binder material of aggregates, which has a melting point of 180°C. It can be imaged that self-healing microcapsules need excellent thermal stability and mechanical properties to keep their integrity in asphalt. It was shown that the size

of microcapsules, the shell thickness, and the core/shell ratio are the main factors determining the stability of microcapsules [51]. Inorganic/organic composite shell structure can be formed to improve the thermal and mechanical properties of micro-capsules [73]. For example, microcapsules had been fabricated with a nano-CaCO<sub>3</sub>/ polymer shell structure [62]. The size of microcapsules did not greatly affect by the structure of microcapsules with nano-inorganic/organic shells. On the contrary, it was varied that the shell thickness increased due to the addition of nano-CaCO<sub>3</sub>. Moreover, microcapsule shells could resist a higher temperature and protect micro-encapsulated rejuvenator. It was noted that the addition of inorganic particles enhanced interaction between the asphalt binders and the microcapsules [74]. The self-healing mechanism of asphalt using microcapsules was investigated by mechanical tests; self-healing process contained four steps: crack generation, break of microcapsules, rejuvenator release, and rejuvenator capillarity–diffusion [75]. Oily rejuvenator flowed along microcracks with the help of capillarity and diffused into the aging bitumen [72] (Fig. 11.32).

Although many efforts have been carried out to investigate microcapsules containing rejuvenator in bitumen, the states of microcapsules in asphalt binders have not been explored. Asphalt is composed of bitumen, aggregate particles, and air voids. Self-healing process in asphalt normally occurs in the binders. After several years of use, bituminous material loses part of its viscoelastic capability. At the same time, microcracks occur and develop at the interface between binders and aggregates. Microcapsules in asphalt binders are expected to keep ideal states in a self-healing process. In view of the above, the purpose of this work was to analyze the morphology, distribution, and thermal stability of microcapsules in asphalt binders. In addition, microcracks were generated to determine the trigger rupture state of microcapsules in asphalt binders. Based on these observations, a conclusion can



Figure 11.32 Illustration of microcapsules containing rejuvenator dispersing in asphalt binder.

be deduced about the application possibility of the self-healing microcapsules in asphalt binders.

### 11.5.1 Experimental method

### 11.5.1.1 Materials

Bitumen was supplied by Qilu Petrochemical of China. The aged bitumen sample (40/50 penetration grade) was artificially produced with 80/100 penetration grade bitumen using a thin film oven method [72]. Rejuvenator (0.905 g/cm<sup>3</sup>, 4.24 Pa s) was obtained from Shanghai Chem. Co., Ltd. of China. Methanol melamine-formaldehyde (MMF) was supplied by Aonisite Chemical Trade Co., Ltd. (Tianjin, China). SMA was applied as dispersant copolymer (Scripset 520, Hercules, USA) [50]. Nano-CaCO<sub>3</sub> powder (mean size 20 nm) was purchased from Tianjin Sinago Technology Co., Ltd. of China.

### 11.5.1.2 Microcapsules fabrication process

The method of fabrication microcapsules containing rejuvenator was divided into three steps [76]: (1) SMA powder was mixed into 50°C water for 2 h, and then the solution was adjusted pH value to 10 by a 0.1 mol·L NaOH solution. Oily rejuvenator was added with a stirring rate stirring speed of 500 r/min for 10 min. (2) The above emulsion was transferred into a three-neck bottomed flask. MMF resin was added with a stirring speed of 300 r/min. At the same time, temperature was elevated to 80°C with a speed of 2°C/min. (3) The polymerization was kept for 2 h, and then the temperature was decreased to room temperature. At last, microcapsules were filtered and dried in a vacuum oven.

# 11.5.1.3 Preparation of asphalt samples mixing with microcapsules

Bitumen (40/50) was blended with microcapsules and aggregate particles using a propeller mixer at 165°C. The mixture was put into a mold that had lubricants in its inner wall. The mold was installed at the gyratory compactor (AFGC125X, PINE, United States) for 100 gyrations under a pressure of 600 kPa. Several asphalt samples were made with a diameter of 100 mm and a height of 67 mm. Table 11.4 lists the gradation of aggregates. Table 11.5 lists the weight contents of asphalt samples and microcapsules with a mean size in a range of  $20-50 \,\mu\text{m}$ .

Sieve size	13.2	9.5	4.75	2.36	1.18	0.6	0.3	0.15	0.075
(mm) Weight (%)	2.4	17.6	20.0	12.9	11.4	8.1	6.7	4.8	5.7

 Table 11.4 Gradation of asphalt sample.

Sample	Aggregates (%)	Bitumen (%)	Microcapsules/bitumen (%)
MB-3	94.85	5	3
MB-5	94.75	5	5
MB-7	94.65	5	7

Table 11.5 Weight contents of aggregate, bitumen, and microcapsules in asphalt samples.

# 11.5.1.4 Morphologies observation

The surface morphologies of microcapsules were observed by using ESEM at an accelerated voltage of 20 kV (ESEM, XL30, Philips). Self-healing process was analyzed by using a fluorescence microscope (CKX41-F32FL, Olympus). As bituminous material was temperature-sensitive, the observation was controlled in an environment under  $0^{\circ}$ C.

# 11.5.1.5 X-ray computed tomography

The distribution state of self-healing microcapsules in asphalt was tested by using an XCT. XCT can be used to determined the internal structure of a material by Xray scanning [77]. XCT is mainly composed of three parts: scanning part (X-ray tube, detector, and gantry), computer system, and image display and storage system. X-rays are generated by the X-ray tube, which can pass through a specimen from different directions and then are detected by the detector. Scanning information is stored in the computer. The image is displayed on the screen, which is processed and reconstructed by computer. Fig. 11.33 shows the X-ray scanning part of XCT instrument. By comparing X-ray attenuation intensity before and after scanning, the



Figure 11.33 A photograph of X-ray scanning part of X-ray computed tomography instrument.

contour information can be obtained, and the contour information is processed to obtain scanning images of the specimen. CT images reflect the extent of absorption of X-rays to the sample with different gray levels. Shadow indicates low absorption region, that is, low-density areas; the white represents high absorbency zone, that is, high-density areas.

### 11.5.1.6 Microcapsules stability in asphalt

Various asphalt samples were made to test the thermal stability of mixing microcapsules during a thermal absorbing-releasing process. The temperature was controlled by a chest [51,75]. This method also was successfully applied to test the thermal stability of microcapsules in epoxy [61]. The asphalt samples were heated to 50°C and kept for 10 min, and then decreased temperature to -10°C. The increasing and decreasing temperature speeds both were 2°C/min. The above increasing-decreasing treatment was repeated 60 times for each asphalt sample. After the absorbingreleasing process, a piece of asphalt peeled off and heated to 150°C. The melting bitumen was spread on a glass sheet. A fluorescence microscope was used to observe the morphologies of microcapsules in melting bitumen.

### 11.5.1.7 Microcrack generation

Asphalt material was poured into  $(160^{\circ}C)$  the middle of two aluminum plates with a thickness of 1 cm. Liquid nitrogen  $(N_2)$  was dropped on the surface of asphalt sample. Microcracks were quickly generated owing to the brittleness of asphalt under low temperature.

### 11.5.2 Results and discussion

### 11.5.2.1 Morphologies of microcapsules containing rejuvenator

In this study, self-healing microcapsules were prepared by in situ polymerization method using MMF shell. Hydrolyzed SMA was used as an amphiphilic polymeric surfactant. SMA was hydrolyzed by NaOH and absorbed at the interface of oily droplets. Rejuvenator droplets owned strong electron negative, which reduced the oil/water interfacial tension [75]. Rejuvenator droplets were formed by high-speed stirring. The oil droplets absorbed MMF prepolymer in order to balance the charge. The coacervation polymers were cross-linked and then formed shells under the effects of acid and heat.

Fig. 11.34A and B shows ESEM surface morphologies of dried microcapsules. They have a mean size of  $20-50 \mu m$ . The microcapsules keep a regular global shape. Their compact shells are smooth with little adherend. Core/shell structure can be recognized form the break microcapsules in Fig. 11.34C and D. The appearance of the core/shell structure indicates that the oily rejuvenator has been microencapsulated by polymeric shell material. These microcapsules have a mean size of about  $20 \mu m$ . Larger microcapsules may be more likely to break or crack. Polymer shells cannot maintain integrity under an ultimate mechanical strength or thermal



**Figure 11.34** ESEM morphologies of microcapsules containing rejuvenator: (A and B) microcapsules containing rejuvenator with mean size of 20 and 50  $\mu$ m, (C) shell structure of a break microcapsule, and (D) a microcapsule with a crack on shell.

stimulation. Therefore, it is important that the microcapsules keep regularity and have an appropriate mean size and shell thickness. For example, the mean size of self-healing microcapsules in asphalt needs to be less than 100  $\mu$ m, avoiding squeeze rupture [72]. It has been found that the shell thickness can be regulated by controlling the amount of shell material [50]. Based on the previous conclusions, all microcapsules in this study had the same core/shell ratio of 2/1 to simplify the complexity.

# 11.5.2.2 Morphologies and integrality states of microcapsules in asphalt binder

Fig. 11.35 shows a picture of asphalt samples mixing with various contents of microcapsules. The diameter of these samples is 100 mm and the height is 67 mm. The addition ratios of microcapsules are calculated by the content of microcapsules



Figure 11.35 A photograph of asphalt samples (MB-3, MB-5, and MB-7) mixing with various contents of microcapsules.

accounting for bitumen. The practicality of microcapsule self-healing technology can be detected by preparing asphalt concrete samples. Obviously, the self-healing degree of asphalt is influenced by the content of microcapsules.

In previous work, it had been reported that microcapsules could survive and in melting bitumen under 180°C-200°C [76]. The result confirms that the microcapsules have satisfactory properties of interface stability, thermal stability, and mechanical stability, which meet the needs of application in bitumen. At present, it is essential to investigate the states of microcapsules in asphalt binders. Fig. 11.36A shows an ESEM morphology of asphalt composed of aggregate and bitumen. The fine aggregates and bitumen are mixed well together. Asphalt binder is pointed by an arrow. Comparing with the total mass of asphalt, it can be concluded that the bitumen is used rarely and it only exists in the aggregates gap. Therefore, it can be imaged that the microcapsules will not sharply increase the cost of asphalt pavement comparing with the traditional repairing methods of pavement. Fig. 11.36B displays the binder mixed with microcapsules. From the point of view of the morphologies of asphalt binder, microcapsules do not affect the performance of asphalt. Microcapsules have a good compactability with bitumen so that interface debonding does not appear between microcapsules and asphalt binders. Fig. 11.36C shows the states of microcapsules in binder. The surface of microcapsules was completely adhered by asphalt binders. It can be obviously seen a fusion of microcapsules and bitumen. Stability of microcapsules performed very well and rupture was not produced at the shell materials. The microcapsules were not damaged by agitation in the molten asphalt.

On the other hand, it is essential to verify the integrity of microcapsules in asphalt binders under a high temperature. A piece of asphalt was peeled from an asphalt sample and heated to melt state. Then melting bitumen without aggregates



**Figure 11.36** Microstructure morphologies of microcapsules in asphalt sample (MB-7) at room temperature state: (A) ESEM morphology of asphalt with aggregate and asphalt binders, (B) ESEM morphology of microcapsules dispersing in bitumen, and (C) a fluorescence microscope morphology of microcapsules in asphalt binders.

was spread on a microscope slide. Fig. 11.37A–C shows the optical morphologies of microcapsules in melting bitumen from asphalt samples (MB-3, MB-5, and MB-7) under a high temperature of 180°C. As the arrows pointing, the microcapsules keep an intact globe shape without rupture. Even in MB-7 under 200°C, as Fig. 11.37D shows, the microcapsules still retain their integrity without premature rupture. It indicates that the microcapsules can resist high temperature and strong squeeze during the asphalt sample formation process. This is in agreement with the previous conclusions based on microcapsules in pure bitumen [76].

### 11.5.2.3 Distribution of microcapsules in asphalt binder

Agglomeration of microcapsule particles in asphalt should be avoided because it may greatly influence the self-healing effective [78]. Fig. 11.38 shows the in situ fluorescence microscope morphologies of microcapsules dispersing in asphalt samples of MB-3 and MB-7. As the arrow pointed, the microcapsules disperse in asphalt binders homogenously without adhesion. In this study, the shells have an inorganic/organic composite structure; the electrostatic interaction between particles does not increase the agglomeration appearance. Comparing Fig. 11.38A and B, the weight ratio increasing of microcapsules in asphalt binders do not also affect the dispersion of microcapsules. It can be imaged that the homogeneous dispersion of microcapsules in binders will enhance the uniformity of the material structure. Self-healing occurs in binders. With the aging of bitumen, the generated microcapsules will break microcapsules and release the oily rejuvenator. If the microcapsules did not disperse well, microcack might pierce fewer microcapsules. In other words, the



**Figure 11.37** Optical morphologies of microcapsules in melting bitumen peeled from asphalt samples (MB-3, MB-5, and MB-7) under high temperature: (A) MB-3 under 180°C, (B) MB-5 under 180°C, and (C and D) MB-7 under 180°C and 200°C.

self-healing process will be affected by inhomogeneity of the structure of the binders.

XCT was used to observe the dispersion of microcapsules in asphalt binders. As the shell has inorganic nano-CaCO<sub>3</sub> particles, the shape of microcapsules can be identified due to the density difference between bitumen and inorganic material in an XCT image. Fig. 11.39A shows a picture of the microcapsules/bitumen samples with cylindrical shape (3.0 wt.%, microcapsules). A CT scan makes the user to observe the inside of an object without rupture using computer-processed combinations of many X-ray image slices. These slices can be taken from different angles of specific areas of a scanned object. Fig. 11.39B is a typical tomographic slice of microcapsules/bitumen sample. The global shape of microcapsules can be clearly identified as the arrows pointed. Microcapsules disperse in bitumen homogenously



**Figure 11.38** In situ observation of microcapsules dispersing in asphalt binder by a fluorescence microscope: morphologies of (A) MB-3 and (B) MB-7.



**Figure 11.39** Images of microcapsules in pure bitumen: (A) a photograph of testing microcapsules/bitumen samples with cylindrical shape (3.0 wt.%, microcapsules), (B) XCT image of microcapsules dispersing in pure bitumen, and (C) an enlarged XCT image of a microcapsule in bitumen with a mean diameter of  $25 \,\mu$ m.

without a phenomenon of particle agglomeration. A local magnification, as shown in Fig. 11.39C, gives more details of the microcapsules. It can be seen that the microcapsules are covered with inorganically particles due to the density difference. The size of darker part is about 30  $\mu$ m, which is equal to the mean size of microcapsules. The darker parts are well-dispersed microcapsules. Otherwise, the darker parts cannot have a global shape and the same size of microcapsule's diameter. A CT slice corresponds to a certain thickness of asphalt. Therefore, voxels compose to a CT slice image; pixels compose to a typical digital image.

In this study, the bitumen was premixed with microcapsules. It might be imaged that the microcapsules were dispersed homogenously in asphalt binders based on previous results. However, it is not clear about the influence of aggregates on the distribution of microcapsules. XCT tests offer a good solution to solve this problem. Fig. 11.40A shows an XCT slice of microcapsules dispersing in asphalt sample (BM-7) with a microcrack. XCT data are generally taken from a sequence of image files, which can be visualized through a 3D-based image tool. Fig. 11.40B illustrates the 3D-XCT image of asphalt sample (BM-7). Based on the same observation angle, the microcrack still can be found along the same direction. As listed in Table 11.1, the aggregates size is in the range of 0.075–13.2 mm. Particles with the size of less than 0.075 mm can be identified in this image. It can be attributed to the presence of microcapsules. Moreover, the microcapsules have a full-range distribution in asphalt. This result is very important for self-healing asphalt using microcapsules containing rejuvenator because a randomly generated microcrack in asphalt has the same probability to meet microcapsules. The mechanism of selfhealing of asphalt using microcapsules has been reported that oily rejuvenator flows out from microcapsules and fills the whole microcrack with the help of capillarity [72,78]. As microcapsules are homogeneously distributed, rejuvenator quickly penetrates and diffuses through both sides of a microcrack. The homogeneous dispersion of microcapsules will also help to accelerate diffusion speed under given conditions [78].

### 11.5.2.4 Thermal stability of microcapsules in asphalt

The initial decomposition temperature of microcapsules was determined by the factors including core/shell ratio, morphology, and shell material dropping speed [51]. In this study, the same microcapsules were used to reduce the complexity of the differences in microstructure. Thermal tests also showed that the microcapsules with nano-CaCO<sub>3</sub>/polymer shells survived in  $180^{\circ}C-200^{\circ}C$  bitumen [76]. Although microcapsules can safely survive in asphalt, it is still required to prove that microcapsules can resist some under extreme heat conditions. It is a well-known and accepted fact about overheating when asphalt operates under extreme heat conditions, which in turn results in stable state of microcapsules. On the other hand, asphalt in real application of environmental conditions withstands the external temperature changes. The temperature-change cycles may cause the breakdown of microcapsules. This will significantly reduce the possibility of microcapsule rupture only when the microcracks appear.



**Figure 11.40** XCT images of microcapsules in asphalt binder (BM-7): (A) an XCT slice of microcapsules dispersing in asphalt sample and (B) a 3D image of microcapsules dispersing in asphalt sample.

First, a fast heating process was applied to simulate a thermal shock for microcapsules. An asphalt sample was placed in a temperature-controlled oven to increase the temperature from room temperature to  $200^{\circ}$ C at a rate of  $5^{\circ}$ C/min. Then a fluorescence microscope was used to observe the states of microcapsules in



**Figure 11.41** Morphologies of microcapsules in asphalt sample (MB-7) under various temperatures: (A)  $75^{\circ}$ C, (B)  $105^{\circ}$ C, (C)  $135^{\circ}$ C, (D)  $180^{\circ}$ C, and (E–G)  $225^{\circ}$ C.

asphalt binder under various temperatures. In Fig. 11.41, morphologies of microcapsules in asphalt sample (MB-7) were shown under various temperatures of  $75^{\circ}$ C,  $105^{\circ}$ C,  $135^{\circ}$ C,  $180^{\circ}$ C, and  $225^{\circ}$ C, respectively. In Fig. 11.41A–D, it can be seen that the microcapsules still keep their original shape without shell rupture under such a violent thermal shock. The reason may be that the bitumen in asphalt binders does not create enough interior stress to break microcapsules. At the same time, the shells of microcapsules have the capability of resisting to a high temperature ( $180^{\circ}$ C). With the increasing of temperature to  $220^{\circ}$ C, rejuvenator has been released from the rupture microcapsules as shown in Fig. 11.41E–G. Traces of oily rejuvenator can be clearly identified.

Second, a simulation method was applied to investigate the thermal stability of microcapsules in asphalt binders under an alternating temperature process. Asphalt sample (MB-7) was heated to  $50^{\circ}$ C and then decreased the temperature to  $-10^{\circ}$ C at a rate of  $2^{\circ}$ C/min. This process was repeated 50-100 times. After the absorbing-releasing process, a piece of sample was peeled off and heated to  $150^{\circ}$ C, then the melting bitumen was evenly coated on glass slides and observed by a fluorescence microscope. In Fig. 11.42A, the microcapsules are compact particles without rupture dispersing in bitumen. It means that they have resisted the alternating temperature process repeated for 50 times. With an alternating temperature process repeated for 70 times, as shown in Fig. 11.42B, it can be found that the microcapsules are brighter and their diameter has been enlarged. It is attributed to the rejuvenator of penetrating into bitumen. The shells at this state cannot encapsulate the



**Figure 11.42** Fluorescence microscope morphologies of microcapsules in asphalt binders. Asphalt sample (MB-7) with a thermal treatment process repeated for different times: (A) 50, (B) 70, and (C and D) 100 times. One temperature-change cycle: heated to  $50^{\circ}$ C with a rate of 2°C/min and keeping for 10 min, and then decreased the temperature to  $-10^{\circ}$ C at a rate of 2°C/min.

rejuvenator safely. After an alternating temperature process repeated for 100 times, the release trace of rejuvenator appears in Fig. 11.42C. This phenomenon has also been found in previous works [62]. When the microencapsulated rejuvenator has been released, microcapsules can help rejuvenator to diffusion in a relatively accurate location comparing to the normal method of applying rejuvenator on the surface of asphalt [72]. Fig. 11.42D shows an enlarged morphology of microcapsules in binder after a temperature process repeated for 100 times. Some microcapsules have been reputed by the thermal action. It needs to be mentioned that self-healing microcapsules may be broken at different conditions, which will supply a continuous rejuvenator into bitumen during the aging process.

### 11.5.2.5 Microcapsules' break by microcracks

Mechanical tests have been designed to measure self-healing behaviors of bitumen [59]. These tests confirmed that oily rejuvenator could flow out from microcapsules and fill the microcrack through capillarity. Then rejuvenator diffused in bitumen under the molecule concentration gradient. Besides the above stationary state, it is

interesting to find more details of microcapsules state in a microcrack. Through observing the state of microcapsules in a microcrack, the self-healing process can be more deeply understood. To stimulate a self-healing process, microcracks were generated by liquid  $N_2$  with a width of less than 30  $\mu$ m as shown in Fig. 11.43A. Microcapsules were pierced by a propagated microcrack successfully. Fig. 11.43B shows the fluorescence microscope morphology of microcapsules in a microcrack in the initial state. The white area is the microcrack. The arrows point the microcapsules in the interfaces of the microcrack. The diffusion process was observed through microcapsules and rejuvenator status. After 60 min, the same position was observed again under the same temperature. It can be seen in Fig. 11.43C, the white area has disappeared. This implied that the microcapsules were broken by microcracks and leaked the oily rejuvenator. The light green color is rejuvenator with capillarity and diffusion traces. The microcrack has been healed with the help of rejuvenator. This self-healing efficiency is almost equal to the reported results in 1 h [72]. With the help of



**Figure 11.43** Morphologies of microcapsules in a microcrack in asphalt: (A) ESEM morphology of a microcrack, (B) fluorescence microscope morphology of microcapsules in a microcrack, and (C) fluorescence microscope morphology of microcapsules in a microcrack after 60 min.

capillarity, rejuvenator filled the microcracks with a movement rate mainly determined by the volume content of microcapsules.

# 11.6 Real application of microcapsules in pavement

The world's first self-healing asphalt pavement using microcapsules containing rejuvenator has been built in Tianjin of China with a 50 m length and has completed a 3-year test from January 2014 to January 2017 (Fig. 11.44). The feasibility of this method is proved by continuous observation and testing, which provides a large amount of data support for future applications. In three years, it is found that the microcracks in this pavement have been decreased 77% comparing to the nearby pavement without of self-healing microcapsules. The world's first commercial self-healing microcapsule product is sold by Tianjin Sinogo Co. Ltd in Tianjin (China). The production capability is 1 t per day. Self-healing product is yellow powder, which needs to be packaged tightly (Fig. 11.45).



**Figure 11.44** The world's first self-healing asphalt pavement using microcapsules containing rejuvenator.



**Figure 11.45** The world's first commercial self-healing microcapsule product is sold by Tianjin Sinogo Co. Ltd. in Tianjin (China).

# 11.7 Further advice

In this chapter, the self-healing microcapsule product has been described systemically from preparation to real application. To promote the wide application of this product, several recommendations are offered.

- 1. The price of this self-healing microcapsule product should be decreased as much as possible. Therefore, it can attract more pavement engineering users. Large-scale production can dramatically reduce price.
- **2.** A model should be given to optimize the application of microcapsules including the size, shell thickness, and amount in various bitumen materials.
- **3.** More real applications should be carried out to optimize the application technology. As a powder chemical product, the skill of application needs a continuous optimization.

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# Novel magnetically induced healing in road pavements



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#### 12.1 Introduction

Roads, only known in the past for moving people and goods, are nowadays also seen as one of the possible alternatives to make our cities smarter. Although the concept of smart roads is not universally defined yet, many attempts from researchers and practitioners have contributed to developing new materials and technologies that possess "smart" capabilities.

The simple combination of bitumen and aggregate for road asphalt pavements can now be engineered to deliver particular functions; among these, asphalt healing is one of the most interesting. The possibility of recovering from cracks with minimal external intervention is definitely appealing to many road authorities who see asphalt healing as a method to reduce the number of maintenance interventions on their road network.

In particular, asphalt healing is considered suitable to heal small pavement cracks (i.e., microcracks) when still at early stage, whereas full-depth large cracks still need to be addressed by major rehabilitation/maintenance interventions. The methodology can therefore be considered advantageous in extending the asset service life as a preventive maintenance approach, investing now to save additional maintenance costs in the future.

Although still at early stage, the introduction of this technology has been trialed on real roads only in a very few places [1], mainly in Europe; most of the research effort, in fact, has been focused on studying several aspects of recovering from asphalt cracks at the laboratory scale.

Induction heating of materials has proved to be a very effective and energysaving technology in the metallurgic industry where a large amount of heat is needed in a very short time to work with metals. It is also adopted in medicine where very small conductive particles are used to generate localized heat through hyperthermia inside the body [2].

However, generating enough electromagnetic field to heat up the entire section of a road in a very short time (i.e., the induction heating machine continuously "healing" the road without any stop) can require a significant amount of electric power, hence denying the benefits generally ascribed to preventive maintenance treatments. In addition, asphalt material is not conductive in nature (e.g., both the bitumen and aggregate are nonconductive) and needs an external facilitator to trigger induction heating and consequent healing of cracks. Commonly, low-cost conductive materials are included in the asphalt mix-design to "activate" the induction process and heat up localized portions of asphalt; heat is then further transmitted to the surrounding material through thermal conductivity and causes the fractured bitumen to melt, fill the cracks, and finally heal once back at ambient temperature.

Engineer the process to use very low input power and generate high heat in the shortest possible time (heating rate) is of utmost importance to guarantee a broader adoption of the technology on real roads.

This chapter will investigate the use of ferrous and ferromagnetic materials to promote and enhance healing of bituminous material through induction heating.

#### 12.2 Principles of induction heating of ferrous and magnetic materials

Induction heating exploits AC magnetic field to induce power in an electrically conductive medium. Several physical laws are involved in explaining this complex phenomenon.

Maxwell's equations define how induced currents are generated and distributed inside the material to be heated due to the electromagnetic field, while Fourier's law can be used to evaluate the thermal field. Ohm's law and Joule-Lenz's law are also relevant to induction heating. Specific properties of the material, such as magnetic permeability and thermal capacity, also play an important role.

The detailed discussion on the general principles behind induction heating is widely available in the literature to which the reader is referred [3,4]. What follows is a short summary explaining the fundamental process that allows ferrous and magnetic particles inside the asphalt mix to be heated up by an external source—the inductor —and produce an alternating magnetic field through a coil. The object being heated is commonly referred to as the workpiece. There is no contact between the workpiece and the induction coil.

According to the fourth Maxwell's equation, if electric current flows in a conductor, it generates a magnetic field in the surrounding space (i.e., a copper wire connected to a battery). If the current varies with time, the magnetic field generated by the current also changes with time. The third Maxwell's equation defines the electromotive force generated by the time variation of the magnetic flux (i.e., the frequency of induction heating). If a conductor object (i.e., a ferrous material) is placed in the electromagnetic field, it will be subjected to induced currents according to Ohm's law. As a voltage is applied to the conductor (i.e., the electromotive force), then a current must flow between any two points of the conductor. These currents are commonly defined as eddy currents. The quantity of current flowing between two points depends, among other variables, on the resistivity of the conductor. Eddy currents generate power in the workpiece and its dissipation creates heating according to the Joule's effect and a consequent increase in temperature. The heat, or thermal energy, is finally distributed according to the thermal properties of the material (i.e., specific heat and thermal conductivity). Several materials change their properties with temperature, hence making the entire process of heating to change during the induction time. The change in resistivity, for instance, entails that more energy is required to heat steel one degree when it is hot compared to when it is cold. Similarly, the thermal conductivity decreases with an increase in temperature for some materials.

Modifying the frequency of induction heating at the source affects the penetration depth in the workpiece; low frequencies (i.e., 10-50 Hz) generally guarantee deeper penetration while high frequencies (i.e., 50-500 Hz) produce quicker heating of the surface.

In ferromagnetic materials, hysteresis losses also play a role during induction heating. The alternating electromagnetic field generates an oscillation of the magnetic dipoles of the material because the polar orientation is continuously changed at every cycle. A minor amount of heat is then generated by the friction produce during the dipoles oscillation (hysteresis).

Commonly, ferromagnetic materials exhibit hysteresis losses up to a specific temperature, known as the Curie temperature; after this point, they become nonmagnetic and hysteresis does not occur anymore. Most of the materials used for induction heating purposes of asphalt have Curie temperature exceeding  $600^{\circ}$ C  $-700^{\circ}$ C; as further explained in the following sections, induction heating of asphalt requires quick rise in temperature and is capable of reducing the viscosity of the bitumen surrounding the microcracks. The maximum temperature of the system in asphalt materials does commonly not exceed  $80^{\circ}$ C $-90^{\circ}$ C. Hysteresis losses generate additional heat in ferromagnetic materials used for asphalt healing, although it is estimated that hysteresis produces between 6% and 8% of the overall heat [4].

Asphalt is a nonconductive material and induction heating does not work unless conductive powders, fibers, or special aggregates are added to the mix design. Heating a piece of "conductive" asphalt through induction is also very different from heating a continuous, dense, and homogenous piece of metal. The conductive material added to asphalt is far from being dispersed homogeneously due to the heterogeneity of asphalt that includes voids and aggregates of various sizes. Microcracks can form anywhere; if there is no conductive material in the close proximity of the crack, this will not heal due to induction heating.

In addition, induction heating on a solid and continuous piece of steel generates heat on the surface (skin effect), which is then transferred internally due to the thermal conductivity of the material. If there is no continuity between the conductive particles in the asphalt mix, the thermal conductivity is then exerted between very different materials; some of them have a very low capacity of transferring heat (i.e., bitumen and aggregate) while some others have a high thermal conductivity (i.e., the conductive particles). This further increases the variables to be studied and decreases the efficiency of the overall process, hence needing more power at the input.

One of the goals to broaden the use of this technology in roads is to use low power to quickly generate enough heat to reduce the bitumen viscosity and fill the microcrack.

# 12.3 Review of ferrous and ferromagnetic materials in road applications to promote healing

The ability of bitumen to self-heal under specific circumstances is well acknowledged with the first studies dating back to 1968 [5]. Self-healing of asphalt is commonly attributed to a combination of rest from loadings and high temperature. During the pavement's service life, the combination of those two conditions does not take place often, mainly because of the continuous loading from traffic and ever-changing climatic conditions. In addition, many authors have studied the intrinsic property of asphalt self-healing using multiple approaches [6-8], both mechanical and physiochemical or molecular [9]. However, although it has been proved that the self-healing effect of bitumen (or bituminous mastic) is tangible, this is evidently not enough to extend the service life of the entire asphalt road pavement. It is thus important to trigger healing of asphalt mixes using external stimuli.

Various healing techniques have been evaluated in the last decade, with induction healing being one of them. The use of induction heating on asphalt (nonconductive material) requires a conductor to be included in the mix design.

Several materials have been experimented to trigger induction heating in asphalt materials.

Among the most common, carbon black [10,11], graphite [10,12], carbon fibers [13], iron powder [14], steel wool [15,16], various metal fibers, and more recently, mining materials [17].

If nonmagnetic ferrous materials are used for the purpose of generating induction heating, the installation of eddy currents is the only way of triggering heat inside the asphalt sample. However, when ferromagnetic materials are included in the asphalt mix design, two main processes occur: the formation of eddy currents and hysteresis losses. The latter process is believed to provide additional healing potential to the material exposed to induction heating [4].

Most of the studies focused on the addition of metallic fibers because their addition has proved to be functional in increasing fatigue life and mechanical strength of a pavement [18]. Generally, the conductivity is linearly related to the volume of fiber in the mix although exceeding the fiber content can generate mechanical problems and workability issues [19]. García et al.[20] found that the radius of the fiber was preponderant in modifying the heating rate of conductive asphalt compared to the volume of fibers in the mix. Liu et al.[21] argued that long fibers provided higher induction heating rate to asphalt than short fibers; however, these experiments were conducted on open-graded mixes with large void content. Generally, shorter fibers (i.e., 10 mm-long) have been used in full-scale trials to avoid clustering and potential difficulties during the mixing and compaction phase. Ajam et al.[22] also tested recycled steel fibers from shredded tires, proving their beneficial effect in induction heating of asphalt.

In a recent study [23], the authors discussed about the potential oxidation and corrosion of steel fibers in asphalt mixes with time and the need to address this aspect of induction healing in road pavements.

Conductive filler has also been investigated thoroughly. The advantage of conductive fillers compared to fibers is the greater dispersion in the bituminous matrix; this is mainly due to the very small size (less than 75  $\mu$ m) and greater compatibility with bitumen [14]. Similarly to fibers, the electrical conductivity of asphalt increases with the greater content of conductive filler. The authors found that adding approximately 10% of graphite filler decreased the resistivity of the bituminous mastic from 10<sup>12</sup> to 10<sup>3</sup>  $\Omega$ m. However, increasing the filler content commonly also stiffens the bituminous mastic and can generate mechanical and durability issues in pavements. In addition, the induction heating rates with conductive fillers are generally lower than conductive fibers.

Some of the fillers are expensive as well (i.e., carbon nanofibers, graphite, carbon black), especially if compared to the standard limestone filler. Many researchers in fact used generic iron powder for the same purpose [24]. More recently, ferromagnetic powders have been used to trigger induction heating exploiting both eddy currents and hysteresis losses [17,25]. However, some studies [26] used magnetic nanoparticles, which required time-consuming (more than 24 h) processing before mixing them with bitumen.

Wang et al.[27] finally used magnetite aggregates to generate heat and trigger deicing features. The exploration of magnetic particles in asphalt mixes and the induction heating component due to hysteresis losses still deserves more investigation.

#### 12.4 Induction healing of bituminous mastics using ferromagnetic filler

Australia is one of the largest iron-producing countries together with Brazil, China, India, Russia, and South Africa. Banded iron formations are widely present in the western part of the country.

Most of the iron ore mines are open cut. The iron-ore bearing rock is blasted and consequently excavated from the open-pit mines. The ores are then hauled to the processing plant for crushing and screening using large dumpers. The most frequent iron ores mined today comprise hematite (Fe<sub>2</sub>O<sub>3</sub>), goethite (Fe<sub>2</sub>O<sub>3</sub>s H<sub>2</sub>O), limonite, and magnetite (Fe<sub>3</sub>O<sub>4</sub>).

Commonly, the latter is the one with the greatest iron content (72% Fe), whereas limonite is the smallest (about 60% Fe).

Magnetite is then refined to remove impurities through a process called "concentration," which is basically a magnetic separation that removes nonmagnetic material in the ore. This process commonly increases the iron content of magnetite up to 80% and more. The overall processing waste is finally dumped into tailing basins.

Using small amounts of magnetite powder, a ferromagnetic material directly coming from mining operations with no additional preliminary treatment can be a viable alternative to trigger engineered induction heating of magnetite-asphalt composites. Magnetite powder (i.e., less than 75  $\mu$ m) can be used as natural rock filler replacement in asphalt mixes.

Some previous studies [17] used pure magnetite nanoparticles to trigger induction healing of small magnetite-bitumen samples; however, the use of expensive pure nanoparticles entails several processing steps that are not suitable for a standard asphalt plant. Among the processes: pretreatment of nanoparticles, with oleic acid, sealing, and sonication in an ultrasonic bath to avoid clustering, hence resulting in more than 24 h of handling before any possibility of using them with bitumen.

Due to the use of magnetite powder in its current form, this study overcomes common disadvantages of nanoparticle processing and handling operations at the asphalt plant. In addition, a common drawback of induction heating in pavement mixes is related to the slow heating rate; increasing the time for heating up the asphalt will result in additional time to trigger induction healing on the pavement surface with a coil. This will in turn slow down the machine carrying out the generator and the coil in the field, hence making the entire process hard to be applied on a lengthy road section, let alone a network of roads. Filler-size particles of magnetite have a very large specific surface area and are able to raise the asphalt temperature quickly if well dispersed.

Fig. 12.1 shows the morphology of the magnetite powder used in the experiments. Magnetite has fragmented shape with sharp edges and a lack of rounded particles.

The particle size analysis distribution chart (Fig. 12.2) shows a uniformly distributed grain size (below 75  $\mu$ m); this was evaluated according to the wet dispersion method where water is used as dispersing solvent [28] in a Mastersizer 3000 HydroLV. The specific surface area of the magnetite filler is 664.9 m<sup>2</sup>/kg.

Electron dispersive spectroscopy (EDS) was used to identify the iron content in the sample and possible external contaminations. Results from EDS are summarized in Fig. 12.2.

Magnetite particles (Fe<sub>3</sub>O<sub>4</sub>) present a high content of iron (71.4%) and oxygen (23.1%). Traces of silicon (Si) were detected possibly due to the siliceous holder. A small percentage of carbon (C)—4.5% by mass—was also identified. The use of magnetite in coal washing is a common process in several mining sites and coal is mainly constituted by carbon (almost 50%). Being carbon a conductive material, it



Figure 12.1 SEM images of magnetite powder at different magnifications.



Figure 12.2 Particle size analysis (PSA) and electron dispersive spectroscopy images of magnetite powder.

was not considered as a contaminant but rather a participant in the overall induction healing process. Though it should be noticed that the electrical conductivity of amorphous carbon is generally smaller than iron.

Disk-shaped samples of bituminous mastic (i.e., bitumen and filler) were prepared by mixing standard pen grade 50–70 bitumen with magnetite for 6 min at 2000 rpm using an overhead stirrer. The magnetite-bitumen ratio was 0.5, 1.0, and 1.5, according to Superpave specifications [29]. Disk samples were 25 mm in diameter and of variable thicknesses (1, 2, and 3 mm).

Differential scanning calorimetry was used to characterize the specific heat of magnetite and bitumen; this information is essential to understand the amount of heat required to increase the temperature of different materials. It was found that the specific heat of magnetite powder is  $0.6-0.7 \text{ J} \cdot \text{g/C}$  for a temperature range from 0°C to 80°C. Bitumen requires instead much more energy to heat the same mass of material and its specific heat is  $1.4-1.6 \text{ J} \cdot \text{g/C}$  in the same temperature interval.

An Ambrell EASYHEAT 2.4 kW induction machine was used to test the induction heating behavior of samples. A flat coil with a flux concentrator was also used. An electric current of 480 A was applied through the coil at a constant frequency of 380 kHz. As previously explained, the higher the frequency, the lower the penetration depth of eddy currents in the material subject to induction heating. An infrared camera with a 100  $\mu$ m magnification lens (FLIR T430Sc) was used to record videos of the experiments and finally evaluate the temperature increase of the bitumen-magnetite samples using the integrated software.

The data in Fig. 12.3 shows the temperature increasing rates of the composite samples at different filler/bitumen ratios and for a current of 480 A (380 kHz).

The heating rate at very low filler/bitumen ratios (i.e., f/b = 0.5) was proved to be inappropriate regardless of the sample thickness. Several seconds are in fact required to reach a suitable healing temperature; this will slow down the potential induction heating machine traveling on the road pavement during preventive maintenance healing treatments. Filler/bitumen ratio of 1 significantly enhanced the heating rate characteristics of the samples and reduced the heating time to a



Figure 12.3 Induction heating of bitumen-magnetite samples.

few seconds only. Obviously, the use of f/b = 1.5 resulted in the fastest rates with the functional healing temperature range (i.e.,  $60^{\circ}C-90^{\circ}C$ ) reached in approximately 1 s.

In addition, it was observed that reducing the mass of the sample by limiting the thickness to 1 mm significantly improved the induction heating potential of the bitumen-magnetite composite material. The heating rate at a filler/bitumen ratio of 1.5 jumped from approximately 30 to almost 53 °C/s when the thickness of the sample was reduced from 3 to 1 mm, respectively. If the actual film thickness of bitumen surrounding the aggregate is considered (approximately  $6-8 \mu m$ ) according to Refs. [30,31], then much higher induction heating rates can be achieved. However, it should be noticed that, on a real road, the thickness of the overall asphalt layer also plays a major role because the penetration depth of the electromagnetic field depends on the power and frequency supplied by the inductor. In addition, aggregates are nonconductive aggregates and internal voids reduce the overall efficiency of the phenomenon.

Compared to other healing techniques in asphalt (e.g., microcapsules containing rejuvenators), ferromagnetic particles will not modify their characteristics after being heated and can possibly be reused for several consecutive healing applications. Though repeated heating of bitumen can cause oxidative aging; however, as the maximum temperature of interest for healing purposes is around  $60^{\circ}C-90^{\circ}C-$  depending on the viscosity of the bitumen—and it only lasts for a few seconds, aging is not considered to be a preponderant factor.

Testing bituminous mastics is not an accurate representation of field conditions; hence, the following section presents the induction healing potential of asphalt mixes with ferrous and ferromagnetic materials.

#### 12.5 Induction healing of asphalt mixes using metals and mixed-metal alloy fibers and ferromagnetic filler

Several asphalt mixes were prepared, including ferromagnetic filler, metal fibers, and a combination of both. The scope of the study was to maximize the induction heating performance of engineered asphalt mixes using small input power. Although greater penetration depth can be achieved by increasing the power supply and reducing the frequency of the electromagnetic field generated by the coil, two main issues arise. First, increasing the power supply significantly increases the cost and the size of the induction machine, hence reducing the benefits associated with the preventive maintenance technology and requiring a larger vehicle to conduct field maintenance. Second, using a too powerful induction heating equipment significantly raises the asphalt surface temperature (i.e., 5-10 mm of depth) in a very short time, hence deterring the heat to further penetrate in the lower layers. In other words, hovering for too long with the induction coil on one point can result in burning the surface layer due to overheating; in addition, more cooling time would be needed before reopening to traffic.

Initially, asphalt mixes that include magnetite powder as the conductive filler material replacement were used to determine how well ferromagnetic materials assist with the heating of asphalt samples. Afterward, other conductive materials such as steel, steel-copper alloy, and recycled steel fibers from shredded tires were added to the mix design to determine their potential in generating induction heating in asphalt samples.

For the purpose of simulating a generic dense-graded asphalt surface mix—nominal maximum size of the aggregate of 12.5 mm—aggregates and bitumen were mixed at 170°C and compacted at 135°C using a standard gyratory compactor. Cylindrical samples with a diameter of 150 mm and a height of 85 mm were prepared for each of the mixes. The bitumen content was 5% by weight of the mix, filler was 4% and the void content of all the mixes was kept constant at 4%  $\pm$  0.5%.

Cylindrical samples were then cut into twelve beams of  $100 \times 15 \times 42$  mm (length  $\times$  width  $\times$  thickness). The full sequence of cuts, as well as sample sizes, is shown in Fig. 12.4. The final beams were tested under an induction coil to study the induction heating potential of each asphalt mixture at two locations: the top and the bottom sections of the sample.

Overall, eight mixes were prepared for this part of the study, each with different quantities of conductive materials—either ferromagnetic fillers or conductive fibers. The proportion of conductive materials, as well as the amount of each material in the mix, was

- Mix 1: 4% magnetite powder as filler replacement (i.e., the entire filler content in the asphalt mix was replaced by magnetite);
- Mix 2: 2.7% magnetite as 2/3 of filler replacement; the remaining 1/3 was natural limestone filler;
- Mix 3: 4% magnetite + 3% (by volume of bitumen) of steel fibers;



Figure 12.4 Asphalt samples preparation and cutting.

- Mix 4: 4% magnetite + 6% (by volume of bitumen) of steel fibers;
- Mix 5: 4% magnetite + 3% (by volume of bitumen) of steel/copper composite fibers;
- Mix 6: 4% magnetite + 6% (by volume of bitumen) of steel/copper composite fibers;
- Mix 7: 4% magnetite + 3% (by volume of bitumen) of recycled fibers from shredded tires;
- Mix 8: 4% magnetite + 6% (by volume of bitumen) of recycled fibers from shredded tires.

Although it was previously demonstrated that the greatest induction rate was achieved by filler/bitumen ratio of 1.5, it was decided here to limit this value to 0.8. This was done to reduce the overall cost of the asphalt mix [32] and after the outcomes of rheological tests conducted on the bitumen-magnetite samples [25]. Higher magnetite content proved in fact to generate significantly stiffer bituminous mastic than standard filler. This was partially due to the shape of the magnetite particles.

In addition, the base mix without ferromagnetic filler and conductive fibers was not tested due to obvious reasons; induction heating is only possible if conductive materials are exposed to an alternating magnetic field and standard bitumen and aggregates are clearly not conductive.

The dispersion of the fibers and magnetite within the asphalt samples was evaluated with a Bruker SkyScan 1275 X-ray microtomography as the distribution of the conductive material within the asphalt determines how well the sample heats up under the induction coil. Clustering of conductive material should thus be avoided to ensure an evenly distribution of the heating gradient throughout the sample during the exposure to the magnetic field. The X-ray microtomography was set to its maximum capacity with x-ray source of 100 kV tube voltage and 100  $\mu$ A tube current; for superior transmission of the X-ray through the asphalt samples and to obtain minimum noise level, a 1 mm copper film was also placed between the X-ray source and the asphalt sample. The sample was subjected to a 0.2-degree rotation around its axis and images with a resolution of  $1540 \times 1540$  were collected to reconstruct the 3D model.

Results from the X-ray scan analysis were useful to analytically calculate the percentage of fibers at various locations inside the sample (i.e., the density of the fibers at different depth). The magnetite filler, being less than 75  $\mu$ m in size, was uniformly spread throughout the entire sample (see Fig. 12.5, for instance); hence, the distribution analysis was only conducted on the metal fibers.

All samples undertook the X-ray CT scan before being tested for induction heating because the scan itself is a nondestructive method of analysis. By doing this,



**Figure 12.5** X-ray scanning of engineered asphalt healing mixes, namely 6% steel fibers (top left), 6% steel-copper alloy fibers (top right), and 3% steel fibers, also highlighting 4% magnetite filler (bottom center).

	Mix 3	Mix 4	Mix 5	Mix 6	Mix 7	Mix 8
0–10 mm (% of the surface volume)	0.52%	0.65%	0.77%	0.92%	0.49%	0.66%

Table 12.1 Internal distribution of conductive filler and fibers.

Notes: magnetite filler-only mixes were found to be uniformly dispersed throughout the entire sample.

X-ray data can be coupled with the heating rate from the induction heating experiments to evaluate the heating rate (i.e.  $^{\circ}C/s$ ) as a percentage of fiber in that specific portion of the sample. In addition, the 40 mm-thick samples were subdivided into two portions to calculate the density of the fibers on the top portion of the sample, close to the coil. Outcomes of the microstructural analysis are shown in Table 12.1.

The same induction heating test configuration was used as per previous indications. The 2.4 kW induction machine was coupled with the flat coil and flux concentrator to provide an electric current of 480 A, providing an electromagnetic field at a constant frequency of 380 kHz. A FLIR T430Sc infrared camera was used to record videos of the experiments and calculate the temperature increase of the composite asphalt samples. This time, the magnification lens was not deployed due to the increased size of the sample. The distance between the coil and the sample was kept constant at 5 mm through a sample holder (Fig. 12.6). All the induction heating tests were stopped as soon as the temperature reached 90°C, this value was considered the maximum functional temperature to generate healing while preserving the road asset from aging and long reopening time.

Three replicates were analyzed for each asphalt mix.

The analysis of the temperature gradient within the samples was conducted subdividing the 40 mm-thick sample into two sections: the surface portion between 0 and 10 mm of depth and the bottom portion from 10 to 40 mm. This approach was followed to collect more information about the penetration depth of eddy currents in the material as a function of power and frequency of the electromagnetic field.

Replacing natural filler with ferromagnetic material significantly increased the induction heating rate of the asphalt mix (Fig. 12.7). Mix 1 (full replacement of natural filler with magnetite) produced an average heating rate of  $1.37^{\circ}$ C/s on the surface (i.e., first 10 mm below the surface) while Mix 2 (2/3 replacement of natural filler with magnetite) showed a minor thermal inclination with a modest 0.82 °C/s. Being limestone filler, a nonconductive material hindered the production of eddy currents within the sample and hysteresis losses.

It was also evident that the bottom part of the sample was only marginally heated by the induction process. After approximately 90 s, the temperature in the bottom section was only raised to  $10^{\circ}C-12^{\circ}C$  with a heating rate of 0.14 and 0.12 °C/s for Mix 1 and Mix 2, respectively.

The electromagnetic field generated by the inductor was not enough powerful to penetrate the entire sample, though it was observed that the first 8-10 mm were effectively heated and up to 14-16 mm the penetration depth was still strong. However, from 17-18 mm onward there was a sharp decrease in the heating rate.



Figure 12.6 Induction heating of asphalt samples.



Figure 12.7 Induction heating rates of composite asphalt mixes.

Other than eddy currents and hysteresis losses, the thermal conductivity of the material is increasingly being accounted for when asphalt starts to develop internal heat. Thermal conductivity of asphalt at  $25^{\circ}$ C is approximately 0.70-0.75 W/mK,

although this value commonly changes with temperature and asphalt mix properties (i.e., void content, petrographic nature of the aggregate, and bitumen content). Evaluating the thermal diffusion characteristics of such heterogeneous material can be challenging.

Focusing on the penetration depth of the magnetic field for Mix 1 and 2-i.e., the two most homogenous mixes due to the use of very small particles of ferromagnetic filler-a clear drop in temperature was observed at a depth of 15-20 mm from the surface. However, Mix 1 and 2 showed a very dissimilar trend; this is due to the induction heating time. Mix 1, in fact, is very quick in developing eddy currents and hysteresis losses and can achieve the maximum functional temperature (i.e. 90°C) after 50 s. Hence, heat conductivity in Mix 1 does not have much time to develop during the testing time. However, if monitoring of temperature was conducted for an additional amount of time, then the thermal conductivity could have been analyzed more in detail. It should be noted though that the penetration of heat in Fig. 12.7 is calculated on the vertical direction from the surface to the bottom, the result would have been different depending on the specific vertical axis chosen on the 100 mm length of the sample. Also, identifying the thermal conductivity behavior of modified asphalt was not the main goal of this research, which instead was about defining the optimal mix design to trigger rapid induction heating with minimum power.

The use of metal fibers significantly increased the heating rate of the asphalt mixes. Steel fiber mixes reached the maximum functional temperature in half of the time compared to ferromagnetic filler mixes (Fig. 12.8). In particular, increasing



Figure 12.8 Induction heating of steel fiber-asphalt mixes.

the fiber content from 3% (red lines in Fig. 12.8) to 12.6% (blue lines) accelerates the average induction heating rate from 2.10 to 2.68 °C/s on the top portion of the sample. Although not significantly different on the surface, the two mixes show a substantial variation at the bottom section where the average heating rate increases from 0.20 to 0.81 °C/s.

The penetration depth does not follow any linear trend with the addition of fibers; although well dispersed in the asphalt matrix, fibers still represent a local point of heating during the induction process. It was noticed that the electromagnetic field was able to generate eddy currents in the majority of fibers through the entire depth of 40 mm (see Fig. 12.8). Fibers were 15 mm long and with a flat surface, the width was 1.5 mm.

Copper-steel mixed-metal alloy fibers were also tested in Mix 4 and 5. Fibers had an internal core of steel with a surface cover of copper; their length was again 20 mm although their shape was spherical and the diameter was 0.8 mm. The electrical resistivity of copper is much lower than steel and at the same time, the thermal conductivity is 15-20 times higher. On the one hand, the presence of copper can reduce the conversion efficiency when the electromagnetic field is transformed to eddy currents but, on the other hand, once a small temperature gradient is developed this is quickly spread along the entire fiber due to the high thermal conductivity if y of copper.

In addition, the smaller size of the fiber increases the surface area compared to steel fibers; hence, improved heating rates were achieved using copper-mixed alloys.

Fig. 12.9 shows the heating rates of Mix 5 and 6. Increasing the volume of copper mixed-alloy fibers from 3% to 6% raised the average heating rate from 5.04 to 5.91 °C/s on the top portion of the sample. The maximum functional healing temperature was achieved in approximately 11 s although less than 7 s were needed to reach 60°C, which according to previous studies is adequate to trigger the flowing of bitumen and healing microcracks. The bottom section of the sample also exhibited a rapid heating rate due to the induction:  $0.85 \,^{\circ}$ C/s for Mix 5 and  $1.97 \,^{\circ}$ C/s for Mix 6.



Figure 12.9 Induction heating of copper mixed-alloy fiber asphalt mixes.

As it can be observed from Table 12.1, copper fibers occupied a larger portion of the volume of Mix 5 and 6 top sections.

The last two mixes included recycled steel fibers from shredded vehicle tires. Steel and copper fibers can increase the price of the asphalt mix, although their use can bring significant advantages in terms of induction healing capabilities. Shredded tires represent a significant resource of crumb rubber, largely adopted in asphalt mixes. However, metal fibers coming from the same recycling process are often difficult to find a suitable market for. The trial of recycled steel fiber asphalt mix was tested to evaluate the efficiency of induction heating using low-cost material.

Although all of them were made of steel, fibers were very different in length from one another ranging from 10 to 40 mm, approximately. Mostly of rounded shape, with a variable diameter of  $0.15-0.6 \,\mu\text{m}$ . The recycling process involves an initial crushing that can potentially modify the size and shape of the fibers before the magnetic separation from the rubber takes place. Some small pieces of rubber can also get stuck to the fiber at the end of the recycling process.

The average heating rate of the top section is not dissimilar from Mix 3 and 4 (steel fibers) with a value of 2.01 and 2.52 °C/s for Mix 7 and 8, respectively. The bottom sections showed 0.17 °C/s for Mix 7 and 0.86 °C/s for Mix 8 (Fig. 12.10).

It is evident that fibers provide a significant additional help in the induction heating of asphalt samples. Although magnetite powder is easier to disperse as a filler due to the very fine size, it does not generate enough heat to trigger a quick healing reaction by the asphalt material. Hence, if the ferromagnetic filler is to be used, a more powerful induction machine is suggested. The penetration depth of ferromagnetic fillers is also limited and works well in the first 2 cm from the surface. Even if reducing the frequency of the electromagnetic field can slightly improve the penetration potential, the average heating rate still requires a greater power (kW) source to generate fast healing.

Fibers significantly improve the induction heating rate and penetration depth. Due to their larger size, they can pick up the dampened electromagnetic field at higher depth from the surface. Their high thermal conductivity (i.e., copper fiber) additionally helps in conveying the heat to the entire fiber and surrounding places.



Figure 12.10 Induction heating of recycled fiber from shredded tires in asphalt mixes.

However, fibers on the surface layer can generate punctures in the vehicle tires and possibly exacerbate the damages from a road accident.

It was then decided to engineer a dual-layer system to overcome these issues and further promote induction heating in asphalt using a low-power source.

#### 12.6 Engineered dual-layer asphalt healing systems

Other than possible punctures to vehicles, one main concern was about the amount of heat being transmitted to the lower layers (or bottom portion of the surface layers). This methodology was thought of as a preventive maintenance treatment on asphalt road pavements, hence postponing expensive resurfacing (i.e., milling and filling) interventions and extending the service life of the pavement asset.

Although a more powerful inductor can easily generate enough magnetic field to reach the bottom portion of any surface layer, the induction heating rates of the top surface portion and the lower bottom will still be very different. In some cases, while conducting the experiments in the previous section, the inductor was left active after the surface portion reached the maximum functional healing temperature. This was done on purpose to evaluate the time needed for the heat to reach the bottom of the sample. Unfortunately, during the experiments, the surface temperature was well above  $150^{\circ}$ C by the time the bottom portion reached  $60^{\circ}$ C. In Mix 6 (copper fiber), the temperature at the surface was  $187^{\circ}$ C when the bottom portion reached  $60^{\circ}$ C and part of the asphalt sample started emitting fumes. After the cooling down, the sample was visibly damaged.

On a real-field installation, the electromagnetic field has to be generated by a coil that can only be positioned on top of the pavement. There will then be points (at the surface) reached by a stronger field and other points (at a certain depth from the surface) where this field has been weakened. The idea of a dual-layer asphalt heating system was thus tested to ensure a minimal induction heating difference between the top and the bottom sections of the surface layer.

Two layers of conductive asphalt material were prepared according to the same procedure highlighted in the previous section. The top layer consisted of a material having the lowest induction heating rate (i.e., ferromagnetic filler) while the bottom layer was made out of mixes with the fastest potential for heating (i.e., ferromagnetic filler plus conductive fibers). In addition, the relative thickness of the two layers was also adapted to test the heating potential.

The top layer was either 10 or 20 mm while the bottom layer was 30 or 20 mm. The bonding of the two layers was ensured by a magnetite-modified bituminous tack coat, applied at a rate of  $0.5 \text{ kg/m}^2$ .

Mix 1 was identified as the top layer in all the experiments while Mixes 3-8 were considered for the bottom layers. An example of the dual-layer system is shown in Fig. 12.11.

The same inductor (2.4 kW), frequency (380 kHz), type, and distance from the coil (5 mm), and infrared thermal camera (FLIR T430Sc) were used in the experiments.



Figure 12.11 Dual-layer induction heating system.

One of the main objectives was to find out the proper combination of materials and thicknesses to deliver a uniform heating rate throughout the entire sample.

Table 12.2 shows the average heating rate due to induction calculated as an average of three replicates.

Results in the previous table show that the average heating rate of the surface (ferromagnetic) mix is still quicker than the bottom (ferromagnetic plus fibers) layer. It is expected that some improvement can be done by calibrating the induction frequency to lower values in order to ensure greater penetration depth.

It can be noted that the first two sublayers (i.e., 0-10 and 10-20 mm) exhibited a very close heating rate for some of the mixes. For instance, copper fibers at 6% volume (Mix 6) had a similar heating rate between the two top sublayers. However, the heating potential of the third sublayer (i.e. 20-30 mm) was about 60% of the sublayer above (i.e., 10-20 mm). The fourth sublayer at the very bottom of the sample (i.e., 30-40 mm) showed a significant drop in temperature compared to the other three sublayers.

Recycled fibers from shredded vehicles (Mix 1 + 7 and Mix 1 + 8) had better heating behavior compared to the standard steel fibers (Mix 1 + 3 and Mix 1 + 4). This was probably due to the different size and shape of the recycled fibers ranging from very small pieces to big chunks. The size of the fiber is capable of modifying the induction heating rate due to the variation in the surface area exposed to the magnetic field.

#### 12.7 Conclusions and future developments

The present research study highlighted the possibility of converting simple asphalt material into a conductive material capable of generating significant induction heating with very small power.

	Top/bottom layer								
Material	4% magnetite (Mix 1)/3% steel fiber (Mix 3)	4% magnetite (Mix 1)/6% steel fiber (Mix 4)	4% magnetite (Mix 1)/3% copper fibers (Mix 5)	4% magnetite (Mix 1)/6% copper fibers (Mix 6)	4% magnetite (Mix 1)/3% shredded tire steel fibers (Mix 7)	4% magnetite (Mix 1)/6% shredded tire steel fibers (Mix 8)			
Thickness top/	10/30	10/30	10/30	10/30	10/30	10/30			
Heating rate top 10 mm [°C/s]	1.34	1.38	1.29	1.39	1.41	1.39			
Heating rate bottom 10-20 mm [°C/s]	0.32	0.70	0.78	1.11	0.68	0.86			
Heating rate bottom $20-30 \text{ mm} [^{\circ}\text{C/s}]$	0.07	0.26	0.27	0.67	0.15	0.34			
Heating rate bottom 30–40 mm [°C/s]	0.02	0.08	0.07	0.11	0.04	0.09			

 Table 12.2 Dual-layer induction heating (top layer, 10 mm).

Although remarkable values of heating rate were achieved on small ferromagneticmodified bitumen samples (i.e., up to 53 °C/s), the numbers drastically change when preparing bigger asphalt samples due to the large volume of nonconductive aggregate compared to electrically conductive bitumen. Ferromagnetic materials, such as magnetite powder from mining operations, can generate induction heating due to the formation of eddy currents and hysteresis losses. However, conductive metal fiber showed improved heating capabilities and added significant value to the ferromagnetic filleronly mixes.

One of the main issues identified during the study was the capability of penetrating inside the asphalt sample at greater depths from the surface. Although the surface temperature was growing quickly, also reaching considerable values in a short time, the bottom portions of the samples showed a delayed response to induction. This was due to the low penetration depth; however, the latter can be adjusted by changing the inductor power and frequency of the electromagnetic field.

Waiting for the diffusion of heat in the bottom sections due to thermal conductivity caused the overheat of the surface portion of the samples. This aspect will be exacerbated even more if greater power is used as input. In addition, fiber-modified mixes can generate punctures of incoming traffic driving on the pavement.

A dual-layer asphalt healing system was then engineered and tested. The top layer (10 mm) was prepared using ferromagnetic filler—slower to heat—while the bottom (30 mm) layer included several types of fibers at different concentrations—faster to heat. The main goal was to achieve a constant rate of heating in all the sections of the sample.

Some of the combined mixes performed well ensuring that at least 2/3 of the sample thickness was heated almost simultaneously; however, the 20-30 mm section's heating rate was reduced to 60% compared to the top 0-20 mm and the bottom (30-40 mm) layer was only marginally heated.

More research is needed to fine-tune the induction heating of asphalt materials. The input power of the inductor should be gradually increased and the frequency reduced to find out the optimal heating rate. This should guarantee that at least 40-50 mm of asphalt are heated thoroughly in order to use the induction healing methodology to preserve the pavement asset and increase its service life. In addition, the heating rate should be quick enough to allow for a standard maintenance vehicle carrying the induction heating machine to only linger on one spot for a minimum amount of time (i.e., a few seconds).

The dual-layer system has shown some interesting potential and is also capable of overcoming common issues often ascribed to fiber-modified mixes such as punctures to vehicles and surface overheating.

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Part 4

## Pavements with energy harvesting potential and vehicle power charging ability

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### Thermoelectric technologies for harvesting energy from pavements

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#### 13.1 Introduction

There are abundant light and heat resources stored in the road pavement. How to make full and efficient use of these resources becomes a research hotspot in the field of road engineering in recent years [1,2]. Converting the light and heat acquired by the road surface into usable energy sources (e.g., electricity) can provide power for local transportation infrastructure (e.g., lighting, signal control, sensors) [3,4]. When the efficiency of energy harvesting from the road surface is high enough, electric power may even be used to power electric vehicles in the future.

Heat is one of the main forms of energy available in the road. The black or dark gray surface of the asphalt pavement makes it absorb solar heat radiation quickly, which causes a large amount of heat to accumulate inside the pavement, increasing the temperature. In summer, the temperature of asphalt pavement can reach  $60^{\circ}$ C or higher during the day, and the accumulation of heat in the pavement will have the following adverse effects [5].

(1) It will increase plastic deformation of the pavement. As a viscoelastic material, most grades of bitumen begin to soften when the temperature reaches 40°C. Combined with the repeated load of vehicles, it will cause irreversible damages such as rutting and ripples [6]. (2) It will accelerate the aging of bitumen. Being an organic material, the oxidation of bitumen and the volatilization of its light components become faster when the temperature rises [7]. As a result, the ductility and cohesive strength of the asphalt mixture decrease, and so do the water stability and low-temperature crack resistance, shortening the service life of the pavement. (3) It will aggravate the urban heat island effect. Road surface accounts for 30% or more of the city's area, and asphalt pavement is the primary type of road surface. During the day, a large amount of heat can be absorbed by the surface layers, which is then radiated to the environment, increasing the ambient temperature of the road [8,9].

Thermoelectric technology is one of the main ways of using energy from road, which converts the heat absorbed by the road into electrical energy by installing thermoelectric generators (TEGs) in or outside the road surface [4]. Compared with other methods of road energy acquisition such as piezoelectric and photovoltaic,

thermoelectric technology has significant economic and environmental benefits [10,11]. In addition to energy output, this method can extend the service life of the road and alleviate the urban heat island effect. Besides, by analyzing the voltage output of thermoelectric pavement, the temperature characteristics of the road surface can be back-calculated, which is another application of the pavement thermoelectric technology to understanding the road conditions.

This chapter will explain the principles of pavement thermoelectric technology and analyze the characteristics of temperature gradient within the pavement. Two road thermoelectric systems will be introduced, which are based on: (1) the road surface-environment temperature difference and (2) the road surface-subgrade temperature difference. The thermoelectric output of the system and its influencing factors, the effect on pavement temperature, and the areas for further research will be discussed, which hope to provoke new ideas and provide references for the development and application of energy harvesting technology from roads.

#### 13.2 Principle of pavement thermoelectric technology

The principle of thermoelectric pavement is based on the concept of "thermoelectric effect," which is first discovered by German scientist Seebeck in 1821, and thus is also known as the "Seebeck effect." This effect explained a phenomenon that the temperature differences between two joints in a closed loop formed by two conductors have potential to generate electricity [12], which lays the foundation for the theory of thermoelectric pavement.

Two different types of thermoelectric materials (P-type and N-type) are connected to form a PN junction, and pairs of PN junctions are connected in series to form a thermoelectric module, as shown in Fig. 13.1. When the two ends of the



Figure 13.1 Schematic of thermoelectric generator [13].

PN junction are placed in different temperatures, the higher hole (electron) concentration at the high-temperature end will drive the holes (electrons) toward the low-temperature end, thereby converting the temperature difference into electrical energy [14].

The electrical energy generated by the thermoelectric module is one of the desirable sources of energy. On the one hand, it can convert temperature differences into electrical energy with no requirement for additional mechanical parts; on the other hand, it does not involve gas or liquid, and thus it is safe to use. Besides, it generates no waste or pollution to the environment.

The energy output of the thermoelectric module is related to the temperature difference ( $\Delta T$ ) between the high-temperature end and the low-temperature end, and the conversion efficiency of the thermoelectric module is largely determined by the properties of the constituent materials (such as "Seebeck coefficient,"  $\alpha_s$ ) and the thermal conductivity and the electrical conductivity of the material. Therefore the thermoelectric properties of the material are generally evaluated by the "thermoelectric figure of merit" (Z), as shown in Eq. (13.1). Generally, the greater the temperature difference between the two ends, the higher the "thermoelectric figure of merit" of the module, and the higher the temperature of the heat source, the higher the power generation efficiency of the thermoelectric module [14]. At present, high-merit-value materials commonly used are solid solutions such as Bi<sub>2</sub>Te<sub>3</sub>, PbTe, and SiGe.

$$z = \frac{(a_s)^2 \sigma}{\lambda} \tag{13.1}$$

where Z is the merit value,  $\alpha_s$  is the "Seebeck coefficient,"  $\sigma$  is the conductivity, and  $\lambda$  is the thermal conductivity.

Thermoelectric technology is widely used in aerospace technology because of its compact structure, reliable performance, long service life, and the ability to operate in extremely harsh environments [15]. For example, it was used on the Apollo Lunar Module, Pioneer, Pirate, Traveler, Galileo, and Ulysses. It was also applied in the military field [16]. As early as the 1980s, the United States completed the development of a 500–1000 W military TEG [17].

In recent years, in the field of waste heat/redundant heat recovery, thermoelectric technology has gained widespread attention and applications in various industries, including chemical industry, steel industry, cement industry, paper industry, and petroleum smelting industry. For example, it could be used to utilize waste automobile heat and waste incineration heat to generate electricity [18]. Moreover, in the fields of biomedicine and the like, thermoelectric technology using human body heat was also used to power cardiac pacemakers and wearable devices [19,20].

Black asphalt pavements tend to absorb a large amount of heat. How to make full use of this heat and convert it efficiently into electrical energy are problems that need to be solved for thermoelectric pavement. Different from using waste heat, the challenges of using the heat in asphalt pavement lie in the wide heat dispersion range and the small temperature gradient. It is difficult to have a large temperature difference within the pavement. In response, researchers have proposed various solutions to this problem. For example, Wu et al. [21,22] connected the subgrade with highly thermally conductive materials, which increased the temperature difference between the subgrade and the road surface, and enhanced the power generation efficiency. Hasebe et al. [23] increased the temperature difference between the two ends of the thermoelectric module, by embedding water pipes in the pavement and introducing cold water from the outside (nearby river water). Guo and Lu [24] performed a simulation analysis using a pipe-pavement TEG system and estimated the electrical energy production by studying the experimental data from Florida.

In summary, the main problems in pavement thermoelectric technology include: (1) rational use of pavement heat, (2) techniques for increasing and maintaining the temperature difference, and (3) ensure thermoelectric conversion efficiency under different environmental conditions [13,25].

#### 13.3 Pavement temperature characteristics

This section defines the temperature characteristics of the road and ambient environment, which are the prerequisite for the design of a thermoelectric pavement system. A set of temperature measurements on road pavement in a city of China is used as an example to demonstrate the temperature characteristics. Making efficient use of the temperature difference between the road surface and the ambient is of great significance to enhance the output of thermoelectric system.

#### 13.3.1 Pavement and ambient air temperature

The temperature difference between the road surface and the ambient air is affected by the climate, such as radiation intensity, duration of sunshine, temperature, humidity, and wind speed. Fig. 13.2 shows the daily temperature profiles of the city of Xi'an, P.R. China, measured on a typical day in summer (August 16, 2016) and winter (December 8, 2016). The temperature of the road surface was collected by an infrared thermal detector, and the ambient temperature and radiation intensity were collected by an outdoor weather station (Fig. 13.3). During data acquisition, movements of the clouds or shadows of surrounding buildings can cause fluctuations in the radiation intensity, such as seen from 12 o'clock to 14 o'clock in Fig. 13.2B.

It can be seen from Fig. 13.2 that external environmental conditions have a significant influence on the temperature difference between the road surface and ambient. On August 16 in summer, the ambient temperature was  $25^{\circ}C-39^{\circ}C$ , the peak solar radiation intensity was 833 W/m<sup>2</sup>, the maximum temperature of the road surface was  $65^{\circ}C$ , the greatest temperature difference between the road surface and the ambient was  $25^{\circ}C$ . The temperature difference was greater than  $10^{\circ}C$  for a duration of 7-8 h, greater than  $20^{\circ}C$  for about 5 h. On December 8 in winter, the ambient temperature was  $1^{\circ}C-16^{\circ}C$ , and the peak solar radiation intensity was  $506 \text{ W/m}^2$ . Although the solar radiation intensity was lower than that in summer, the highest



Figure 13.2 Road surface and the ambient air temperature [13]: (A) August 16, 2016 and (B) December 8, 2016.

temperature of the road surface was  $30^{\circ}$ C due to the heat-absorbing effect of the asphalt pavement. The maximum temperature difference exceeded  $15^{\circ}$ C, and the temperature difference was greater than  $10^{\circ}$ C for about 5 h. Therefore the temperature difference between the asphalt pavement and the ambient is noticeable in both a high-temperature summer and a low-temperature winter.

#### 13.3.2 Pavement and subgrade temperature

Solar radiation is not only the primary source of heat on the road surface but also the primary source of heat in the subgrade soil. The temperature of the subgrade



Figure 13.3 Mobile weather station device [13].

soil will rise with an increase in the ambient temperature, and the shallower the depth, the more significantly it is affected by solar radiation and the ambient temperature. However, due to the thermal properties of the soil, water content, and surface vegetation of the subgrade soil, its temperature change is less than, and always lags behind, the change in ambient temperature. Therefore the temperature of the subgrade soil at a certain depth is relatively stable, which is lower than the ambient temperature in summer and consistently higher in winter [13].

Fig. 13.4 shows the temperatures of the road surface, ambient air, and subgrade soil measured on January 26, February 25, April 14, and May 2, 2019 in Xi'an. The road surface temperature was collected by an infrared thermal detector, and the ambient temperature was collected by an outdoor weather station. The temperatures of the subgrade soil were measured at 10, 50, and 100 cm depth below the road surface, as shown in Fig. 13.5. From January to May, the subgrade soil temperature at a depth of 10 cm was lower than that at a depth of 50 and 100 cm, and the temperature difference ranged from  $2^{\circ}$ C to  $6^{\circ}$ C. At the same time, the temperature change of the subgrade soil at a depth of 10 cm was more remarkable than that at a depth of 50 and 100 cm.

Fig. 13.6 shows the temperature difference between the subgrade soil and the road pavement at a depth of 10 cm. On the four test days from January to May in 2019, as a result of an increase in the ambient temperature, the temperature difference between the subgrade soil and the road surface steadily increased. Table 13.1 outlines the temperature characteristics of the pavement and ambient environment on the test days.



**Figure 13.4** Road surface, ambient air, and subgrade temperature at different depths: (A) January 26, (B) February 25, (C) April 14, and (D) May 2, 2019.

It is anticipated that the temperature difference between the road surface and the subgrade soil will further increase in June–August, when solar radiation and ambient temperature are higher.

The above results show that there is a significant difference between  $TD_{ra}$  (the temperature difference between the road surface and the ambient) and  $TD_{rs}$


Figure 13.4 (Continued).

(the temperature difference between the road surface and the subgrade soil). When the ambient temperature is high, the  $TD_{rs}$  is more significant than the  $TD_{ra}$ .

While direct contact with the wheels by pavement surface makes it challenging to utilize the solar heat directly, the temperature gradient along the depth of the pavement gives valuable information for designing the layout of the thermoelectric devices.



Figure 13.5 Temperature of subgrade soil at different depths.



Figure 13.6 Temperature difference between road surface and subgrade at 50 cm depth.

Generally, the temperature of the road surface is the highest under solar radiation during the day, and the temperature gradually decreases along the depth of the pavement. Though the temperature at a depth of 20-30 mm is about 4°C lower than the road surface, it can still maintain a significant temperature difference with the ambient. At the same time, temperature at this depth is influenced very little by the solar radiation and vehicle load. Therefore it is ideal to have the thermoelectric devices embedded at this depth when designing the road thermoelectric system for energy harvesting.

Test index	On January 26, 2019	On May 2, 2019
Range of ambient temperature	3.8°C−9.1°C	14.6°C-26.1°C
Maximum temperature of road surface	15.7°C	45.6°C
Maximum temperature difference between the road surface and the ambient	8.0°C	20.9°C
Maximum temperature difference between the road surface and the subgrade soil at 10 cm depth	8.6°C	26.4°C
The duration of the temperature difference between the road surface and the subgrade soil (greater than 5°C and 10°C)	3 h	6 h

Table 13.1 Temperature characteristics of the pavement and ambient.

It is worth noting that the test area is located within a temperate climate zone  $(107.40^{\circ}\text{E} - 109.49^{\circ}\text{E}, 33.42^{\circ}\text{N} - 34.45^{\circ}\text{N})$ . It can be expected that the temperature difference between the road surface and the ambient will be more significant, and the duration of the temperature difference will be longer in tropical and subtropical regions, making the road thermoelectric system more applicable.

## 13.4 Design of road pavement thermoelectric generator system

The design of road pavement thermoelectric system has the following key aspects: first, conduct and collect thermal energy efficiently from the pavement; second, utilize the temperature difference between the pavement and the ambient (subgrade soil); third, minimize the influence of this system on pavement structure and performance.

The pavement thermoelectric system generally includes a thermal-conductive module, a thermoelectric conversion module, and a cooling module. (1) The primary function of the thermal-conductive module is to collect heat from the road and transmit it to the high-temperature end of the TEG. (2) The primary function of the thermoelectric conversion module (usually TEG) is to convert the heat conducted by the vapor chamber into electrical energy. (3) The primary function of the cooling module is to reduce the temperature of the low-temperature end of the TEG, thereby enhancing the power output and improving the efficiency of the system. Different pavement thermoelectric system can be designed depending on how the temperature difference is utilized.

#### 13.4.1 Pavement-ambient thermoelectric system

Using the temperature difference between the road surface and the ambient, the following thermoelectric system can be designed.

- 1. Thermal-conductive module. Materials with high thermal conductivity (e.g., soaking plate, metal plate, a combination of metal plate and wire) are embedded in the pavement perpendicular to the direction of traffic, transmitting the heat of the road to a concentrated point by the roadside. These thermal-conductive materials are typically embedded 20–30 mm below the road surface considering the ease of construction, vehicle loads influence, and temperature gradients of the pavement, allowing them to collect heat from a larger area within the road and meet the needs of thermoelectric conversion module by the roadside. At the same time, these materials should have excellent resistance to the load from construction machinery and vehicles during the construction and service life.
- 2. Thermoelectric conversion module. The TEG is placed at one end of the thermoelectric conversion module and is located at the roadside. TEG can convert thermal energy into electrical energy in the presence of temperature differences between the two ends (high-temperature end and low-temperature end) of the module. In order to create and maintain a temperature difference, the high-temperature end of the TEG is connected to the thermal-conductive module, and the low-temperature end of the TEG is connected to the cooling module.
- **3.** Cooling module. The cooling module can be air-cooled or water-cooled; the water cooling system usually has a better cooling effect. Thus a cooling water tank can be provided at the low-temperature end of the TEG. The bottom of the water tank is made of a material with good thermal conductivity (e.g., a soaking plate or a metal aluminum), and the side wall of the water tank is made of a material with excellent heat dissipation performance. To avoid direct sunlight, which could increase the temperature of the water, a light shield is provided outside the water tank.

When the asphalt pavement is heated by solar radiation, heat is transferred to the high-temperature end of the TEG module through the vapor chambers. Combined with the low-temperature end of the TEG, electrical energy output can be obtained, which not only generates additional energy but also cools the road pavement in the hot climate. The design concept of this thermoelectric system is shown in Fig. 13.7, which is based on the temperature difference between the asphalt pavement and the ambient, using thermoelectric conversion module TEG.

#### 13.4.2 Pavement-subgrade thermoelectric system

The pavement-subgrade thermoelectric system can be designed using the temperature difference between the road surface and the subgrade soil.

- 1. Thermal-conductive module. The layout and materials of the thermal-conductive module are the same as in the pavement thermoelectric system based on the pavement-ambient temperature difference, which is described in Section 13.4.1.
- **2.** Thermoelectric conversion module. The TEG is also used in this system and is connected with the high-temperature end of the vapor chamber at the roadside.
- **3.** Cooling module. The low-temperature end of the TEG is connected with the vapor chamber, which is embedded vertically in the soil at roadside. As the depth increases, the soil temperature will be less affected by the external environment. Considering the potential adverse effects of the construction process, the depth of the vapor chamber can be about 1 m. At the same time, the middle part of the vapor chamber, which has excellent thermal conductivity and is embedded in the subgrade soil, should be wrapped with an insulating material to ensure more efficient heat transfer at the low-temperature end of the TEG.



Figure 13.7 Schematic of pavement-ambient thermoelectric system [25].

In summary, solar radiation causes an increase in temperature within the asphalt pavement, and the radiant energy is conducted to the high-temperature end of the TEG through a vapor chamber. With the vapor chamber at the low-temperature end of the TEG maintaining a relatively low temperature by exchanging heat with the soil, the TEG is able to convert the temperature difference between the two ends into a voltage. In general, the temperature difference created in the pavement-subgrade thermoelectric system (as shown in Fig. 13.8) is larger in summer and smaller in winter, indicating higher voltage outputs in summer.

#### 13.4.3 Design of thermoelectric generator

According to the design concept illustrated in Fig. 13.7, a thermoelectric system model is built to test the power generation capability. Asphalt mixture samples of 300 mm (length)  $\times$  300 mm (width)  $\times$  100 mm (high) are used to represent the pavement structure, which contained a 40-mm-thick upper layer of asphalt concrete (AC)-13 [styrene-butadiene-styrene (SBS)-modified] and a 60-mm-thick lower layer of AC-20 (SBS-modified).

Aluminum vapor chamber (Fig. 13.9A) is used as the thermal-conductive module with a dimension of 3 mm (thickness)  $\times$  300 mm (length)  $\times$  60 mm (width), a compressive strength of 4 MPa, and a thermal conductivity of 106 W/m · K. Three aluminum vapor chambers are placed at a depth of 20 mm in the sample of the asphalt mixture, with a length of 200 mm embedded inside, and a length of 100 mm exposed outside, as shown in Fig. 13.9B.

The thermoelectric-conductive module TEG-199 (shown in Fig. 13.10) has a size of 62 mm (length)  $\times$  62 mm (width)  $\times$  4 mm (height) and includes 199 pairs of thermoelectric elements. The high-temperature ends of the three TEG-199s,



Figure 13.8 Schematic of pavement-subgrade thermoelectric system.



Figure 13.9 (A) Aluminum vapor chamber and (B) their arrangement in the slab specimen [25].



Figure 13.10 Thermoelectric-conductive module TEG-199.

connected in series, are bonded to the three aluminum vapor chambers by thermal silica gel and exposed at the side of the sample.

An organic glass water tank (Fig. 13.11) is combined with a sun shield as a cooling module in the thermoelectric system. The size of the water tank is 350 mm (length)  $\times$  150 mm (width)  $\times$  160 mm (height), the tank bottom is made of aluminum vapor chambers, and the side wall is equipped with cooling units in order to conduct the heat more efficiently. The low-temperature end of the three TEG-199s is bonded to the bottom of the water tank by thermal silica gel. When testing outdoors, a sun shield is placed over the tank to avoid direct sunlight, which otherwise could increase the temperature of water in it.

When tested indoors, a 500 W iodine-tungsten light is used to simulate solar radiation, which is placed 500 mm above the surface of the sample. During the test, the maximum temperature on the surface of the sample is 65°C. The temperature of the sample is recorded every 10 min by an infrared thermoforming instrument. The temperature in the water tank is measured by a thermal sensor, and the generated power voltage is recorded by an electronic instrument.

## 13.5 Energy output and influencing factors

#### 13.5.1 Energy output

#### 13.5.1.1 Indoor test

The relationship between output voltage and temperature difference, obtained by the indoor test described in Section 13.4.3, is shown in Fig. 13.12. The regression



Figure 13.11 Organic glass water tank: (A) top view and (B) side view.



Figure 13.12 Relationship between temperature difference and output voltage of TEG by indoor test [13].

relationships between output voltage and temperature difference are different during the heating process, stabilizing process, and cooling process. The temperature in the water tank is close to the temperature at the low-temperature end of the TEG. The surface temperature of the pavement sample, however, is different from the temperature at the high-temperature end of the TEG. There is a temperature gradient along the depth of the sample. In the heating process, the surface temperature of the pavement sample is higher than the temperature of the TEG; the opposite is the case in the cooling process. Therefore the voltage has a linear relationship with the temperature difference in both heating and cooling processes, but the intercept of the regression line is different, see Eqs. (13.2) and (13.3). A peak voltage of 0.737 V obtained in the laboratory occurred at the beginning of the cooling process with a temperature difference of  $34.7^{\circ}$ C.

The functional relationship of the heating process:

$$U_i = 0.018T_d - 0.1733(R^2 = 0.9899)$$
(13.2)

The functional relationship of the cooling process:

$$U_d = 0.0186T_d + 0.1532(R^2 = 0.9671)$$
(13.3)

where  $T_d$  is the temperature difference between the surface of the pavement sample and the water in the tank.

The computer-aided simulation can also be used to design and optimize the pavement thermoelectric system [26]. Wu and Yu designed an energy harvesting system using the soil and pavement and estimated the energy output [27]. The system was mainly composed of TEGs, aluminum plates, and aluminum rods. TEGs were set on the road surface. In order to increase the temperature difference between the two ends of the TEGs, the low-temperature end of the TEGs was in contact with an aluminum plate (40 mm  $\times$  40 mm  $\times$  5 mm), which was connected to a 1000 mm long aluminum rod. The aluminum rod was vertically inserted into the road, and the low-temperature feature of the roadbed soil can be utilized more efficiently by wrapping the 590 mm part of rod near the surface end with a thermal insulating layer. According to the survey data from Ohio, the soil temperature at a depth of 150 cm is about 15°C, while in summer, the temperature of the road can be as high as  $40^{\circ}$ C, and as low as  $-20^{\circ}$ C in winter. The recorded maximum temperature difference was about 16°C between the road surface and the soil. Based on this, it is estimated that the system can provide 0.02 W of power and more than 1000 J of energy in one day, which is sufficient to provide energy for the sensors used to monitor the road.

#### 13.5.1.2 Outdoor test

Outdoor tests benefit from having the real radiation intensity, radiation time, and ambient temperature conditions, and thus the thermoelectric system is able to demonstrate its real voltage output performance. The pavement sample with the



Figure 13.13 Daily voltage output in different seasons [25].

TEGs is placed outdoors and is heated by absorbing solar radiation. The data can be collected in the same way as in the laboratory.

Fig. 13.13 shows the voltage output of the thermoelectric system on six test days from February to July in 2016. The voltage output of the pavement sample changed with time, that is, it increased first with the solar radiation intensity and then decreased. The environmental conditions in different seasons had a significant effect on the voltage output. In May, June, and July, when the ambient temperature and radiation intensity were high, the voltage output of the thermoelectric system was higher and the production period was longer. The peak voltage measured on July 24 was 0.564 V, and the voltage was above 0.3 V for more than 8 h. For February and March, when the ambient temperature and radiation intensity were low, so was the voltage output. For example, the peak voltage measured on February 24 was 0.275 V.

Fig. 13.14 shows the relationship between temperature difference and voltage output obtained from indoor and outdoor tests for over 6 months. The overall finding is that as the temperature difference increases, the voltage output obtained by the pavement thermoelectric system increases almost linearly. The voltage output obtained by the outdoor test is largely consistent with the indoor test results despite the indoor results showed a wider range of data. This indicates that the combined environmental conditions outdoor have "moderated" the change in voltage output, which is more obvious under indoor, controlled laboratory conditions.

According to the temperature difference between the road surface and the subgrade soil demonstrated in Section 13.3.2, the result from the thermoelectric system based on the surface-ambient temperature difference can be used to estimate the voltage output of the thermoelectric system based on the road-subgrade temperature difference. It is worth noting that due to the difference in the cooling method of the two pavement thermoelectric systems, the voltage output performance of the two systems may be different and experimental tests are recommended to quantify the difference in outputs between the two systems.



Figure 13.14 Correlation between voltage output and temperature difference.

Tahami et al. developed a soil-pavement generation system using the temperature difference between the road surface and the soil underneath it [28]. The system consisted of the following components: (1) A heat transfer module using an L-shaped copper plate with a thickness of 1.5 mm: one end of the plate was embedded at a depth of 20-30 mm in the pavement, and the length was 500 mm; the other end was embedded in the soil, and the length was 180 mm; (2) the hightemperature end of the TEGs was connected to the copper plate in the soil by a thermal paste, and the low-temperature end was connected to an aluminum heat sink(180 mm  $\times$  100 mm  $\times$  50 mm); (3) The aluminum heat sink was filled with microencapsulated phase change material (MPCM) to help stabilize its temperature. In addition, there were three layers of insulation materials at the outside of the aluminum heat sink, from inside to outside, 10 mm thick aerogel, 20 mm thick Styrofoam, and 10 mm polyvinyl chloride, which also help to stabilize its temperature.

The system was tested outdoor in San Antonio, United States, on July 21, between 2:00 p.m. and 7:00 p.m. During the test, the surface temperature of the asphalt mixture specimen was  $55^{\circ}C-62^{\circ}C$ , the temperature of the copper plate was  $48^{\circ}C-50^{\circ}C$ , the soil temperature at a depth of 10 cm was  $34^{\circ}C-38^{\circ}C$ , and the temperature of the aluminum heat sink was  $18.3^{\circ}C-18.8^{\circ}C$ . Thus the maximum temperature difference is approximately  $32^{\circ}C$ . According to the test results, the

Date	Ambient temperature, °C	Peak radiation intensity, W/m <sup>2</sup>	Weather	Wind, level	Temperature difference, °C	Voltage peak, V
Feb. 24, 2016	0-11	779	Sunny	1	22.5	0.275
Mar. 22, 2016	5-16	902	Cloudy	3	19.5	0.310
Apr. 22, 2016	8-25	992	Sunny	2	29.7	0.391
May 26, 2016	18-32	1038	Sunny	3	30.3	0.534
Jun. 26, 2016	23-35	1022	Sunny	2	29.7	0.485
Jul. 24, 2016	27-40	999	Sunny	1	29.6	0.564

Table 13.2 Environmental conditions in outdoor tests.

Note: Temperature difference refers to the difference between the temperature of the sample surface and that of the water tank.

road with a width of 10 m and a length of 1 km will have an output power of  $83.5 \times 10^6$  J, which is equivalent to  $23.2 \text{ kW} \cdot \text{h}$  electricity per day. The study also found that the introduction of MPCM helped to increase the temperature difference between the two ends of the TEGs, thereby increasing the energy output [28].

#### 13.5.2 Influencing factors

#### 13.5.2.1 External environment

From the previous analysis, the voltage output of the thermoelectric system is related to the temperature difference between the two ends of the TEG, which is affected by the external environment such as radiation intensity, ambient temperature, wind speed, and humidity. Table 13.2 lists the environmental conditions for the six tests in Figs. 13.13 and 13.14.

The voltage or electrical energy of the thermoelectric system is obtained by converting the heat absorbed by the road surface, so in theory, the magnitude of the voltage should be positively correlated with the radiation intensity. From February to July, as the radiation intensity increases, the ambient temperature and the temperature difference between the road surface and the ambient also increases, which led to an increase in the voltage output of the thermoelectric system. As the external environment includes other factors such as humidity and wind speed, the correlation between the peak radiation intensity and the peak output voltage is not quite concurrent, as shown in Fig. 13.15. For example, for a thermoelectric system with a water tank and cooling units at the low-temperature end, higher wind speed is beneficial to the cooling of the TEG's low-temperature end, resulting in a larger temperature difference and thereby a higher voltage output.

The test results show that regardless of hot summer or cold winter, under sufficient solar radiation, a temperature difference can be obtained between the road surface and the ambient, which provides a prerequisite for voltage output of the thermoelectric system. The thermoelectric module used in this test has a resistance of  $1.25 \Omega$  at  $20^{\circ}\text{C}-60^{\circ}\text{C}$ , and the current and power generated by the system can be calculated according to Ohm's law. Based on the results obtained on May 26,



Figure 13.15 Voltage output and radiation intensity.

the 300 mm  $\times$  300 mm pavement sample produced about 1080 J of energy, which means with the same output efficiency, a road of 1 km long and 10 m wide could produce 1.2  $\times$  10<sup>8</sup> J of energy, equivalent to 33 kW  $\cdot$  h of electrical energy.

#### 13.5.2.2 Traffic loading

The impacts of traffic loading on the thermoelectric system mainly include two aspects.

On the one hand, the impact of traffic loads on the thermoelectric devices embedded in the road surface should be considered. As mentioned above, the aluminum vapor chamber used in the experiment had a bearing strength of 4 MPa when subject to a uniform pressure, which enables it to take the traffic load. In subsequent research and practice, other materials with good thermal conductivity and high compressive strength, such as copper and aluminum, may also be considered. In addition, some thermally conductive materials are flexible, such as graphitesoaked films, and may also be fit for this purpose.

On the other hand, the impact of traffic load on power generation should also be considered. The power generation efficiency of the system is mainly related to the temperature difference. However, due to the abrasion of the asphalt and the covering by dust and debris, the color of the asphalt road surface may become lighter and the road surface temperature during the day may be consequently reduced. Fig. 13.16 shows the comparison of temperature, collected by infrared thermal imager, between the new asphalt mixture and the old road surface (in place for about 10 years). The test was conducted on August 24. Overall, the surface temperature of the new mixture was slightly higher than that of the old one, but the difference was mostly within  $2^{\circ}$ C. Interestingly, there were also periods when the temperature of the old mixture was slightly higher. As this heat is less than the solar radiant heat, the traffic load is not considered an important factor that affects the efficiency of thermoelectric system.



Figure 13.16 Temperature of the new asphalt mixture and the old pavement surface [25].

#### 13.5.2.3 Scale effect

The scale effect is another factor that affects the efficiency of the thermoelectric system. Fig. 13.17 shows the indoor test results of a thermoelectric system with one, two, and three TEG modules connected in series. The test conditions are as follows: by adjusting the height of the light source, the surface temperature of the sample is maintained at 40°C; the water temperature is maintained at 20°C, 25°C, and 30°C, which results in a temperature difference of 20°C, 15°C, and 10°C, respectively, as shown in Fig. 13.17. Results show that the voltage output of the thermoelectric system is positively correlated with the number of TEG modules. There is no significant attenuation of the voltage output when two or three TEGs are connected in series. However, when the number of TEG modules is further increased, the system's voltage output efficiency may be reduced. In future studies, the power generation efficiency of different scales can be evaluated.

#### 13.5.2.4 Thermoelectric generator

As the most important component in the thermal-electrical conversion process, the performance of the TEG module directly affects the conversion efficiency and voltage output. The thermal-electrical conversion efficiency is mainly related to the "thermoelectric figure of merit" of the semiconductor material in TEG (generally, the higher the "merit" of the material, the higher the thermoelectric conversion efficiency). In addition, the size, quality of manufacture, and especially the number of thermoelectric elements inside the TEG have significant effects on the voltage output. Table 13.3 lists the TEGs of six different models and the voltage output measured under the same temperature difference (i.e., 13.6°C), is shown in Fig. 13.18. It can be seen that the difference in voltage output of the six TEG modules with similar dimensions under the same test conditions is significant. In general, the more semiconductor pairs in the TEG, the greater the voltage output will be.



**Figure 13.17** Effects of TEG number on the voltage output of the thermoelectric specimen [25].  $U_i$  is the voltage output for *i* piece(s) of TEG(s);  $T_d$  is the temperature difference between the sample surface and water in the tank.

	TEG- 199(a)	TEG- 335(a)	TEG- 127	TEG- 199(b)	TEG- 241	TEG- 335(b)
Length and width, mm	62 × 62	62 × 55	62 × 62	$50 \times 50$	55 × 55	62 × 55
Number of semiconductor components, pairs	199	335	127	250	241	335

<b>Table 13.3</b>	Main	parameters	of six	types	of	TEG.
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Figure 13.18 Voltage output of different types of TEG.

## 13.6 Effect of thermoelectric system on pavement temperature

The thermoelectric system can effectively reduce the temperature of the road pavement by converting the heat into electrical energy, which has beneficial effects on the performance and durability of the asphalt mixture.

Fig. 13.19 shows the temperature for 6 days from February to July in 2016 of the ordinary sample (control sample) and the sample equipped with thermoelectric system (test sample). Given a certain solar radiation intensity, the surface temperature of the test sample is significantly lower than that of the control sample. When the ambient temperature and solar radiation are higher, the thermoelectric system has a more significant cooling effect on the road surface, and the cooling effect lasts for a longer period. For example, on May 26, June 26, and July 24 when the ambient temperature was high, the temperature of the test sample decreased by more than 5°C for 7 h, and the temperature decreased by more than 3°C for 11 h. In comparison, on February 24 and March 22 when the ambient temperature was lower, the temperature of the test sample decreased by 2°C for about 3 h. This difference in temperature reduction meets the requirements of road pavement in different seasons. That is, in summer when the ambient temperature is high, it is desirable to reduce the road temperature more significantly, in order to reduce plastic deformation of the asphalt pavement and the urban heat island effect; there is less need to cool the pavement when the ambient temperature is low in winter.

In addition, it can be seen in Fig. 13.19 that the peak temperature of the road surface generally appears between 14:00 and 15:00, while the maximum temperature difference between the two samples occurs relatively late, usually between 16:00 and 18:00. This indicates that the thermoelectric system's cooling effect lags behind the temperature rise in the road surface.

Fig. 13.20 shows the infrared thermal pictures of the test sample and the control sample. The pictures were taken at 14:50 on May 26, 2016, which represent the peak surface temperature of the sample on the day. Fig. 13.20A shows the temperature image of the test sample showing an average temperature of  $60.1^{\circ}$ C; Fig. 13.20B shows the temperature image of the control sample, showing an average temperature of  $67.6^{\circ}$ C. There are two distinct temperature zones on the surface of the test sample. One is where one side of the aluminum vapor chamber was laid, and it accounts for nearly two-thirds of the total area; the temperature is relatively low with an average of  $57.4^{\circ}$ C. The average temperature of the remaining part was  $63.9^{\circ}$ C. It indicates that the aluminum vapor chamber can transfer the heat inside the asphalt mixture more efficiently. As a result, the temperature of the zone around the aluminum vapor chamber is significantly lower than that of the control sample.

It can be seen from the above results that the thermoelectric system can significantly reduce surface temperature of the samples by converting heat into electrical energy, and the temperature reduction is affected by the ambient temperature and the radiation intensity. Fig. 13.21 shows the relationship between the temperature of the test sample and the temperature reduction. When the temperature of the



**Figure 13.19** Temperature of the thermoelectric specimen and the conventional specimen in different seasons [25]: (A) February 24, (B) March 22, (C) April 22, (D) May 26, (E) June 26, and (F) July 24, 2016.

specimen increases from 30°C to 60°C, the temperature reduction also increases. This is particularly the case when temperature of the sample increases from 50°C to 60°C, the temperature reduction of the sample increased from 4°C to 8°C. Then, when the radiation intensity and the ambient temperature increase so that the temperature of the sample reaches 70°C-75°C, the temperature reduction does not increase further, indicating that the thermoelectric system's cooling capacity has an



**Figure 13.20** Infrared thermal image of surface temperature of the thermoelectric specimen and the conventional specimen [25]: (A) thermoelectric specimen and (B) conventional specimen.



Figure 13.21 Relationship between surface temperature and temperature reduction [25].

upper limit. The reason is that the thermoelectric conversion efficiency of the system is limited. When the conversion capacity of the system is exceeded, the excess radiant energy will still cause the road to heat up.

## 13.7 Conclusion and future developments

Although thermoelectric technology has been widely used in military, aerospace, medical and waste heat utilization, research in the field of road engineering is still limited to indoor test and small-scale outdoor test. In the future, research and practices of pavement thermoelectric technology can focus on the following aspects.

- 1. Design for superior performance. Analyze the temperature characteristics and gradient within the road pavement, improve the thermoelectric conversion efficiency, and maximize the output power by further exploiting the temperature difference. In the meantime, the interference of the system to road construction, operation, and maintenance should be minimized; road pavement embedded with the thermoelectric system should have good durability and recyclability.
- **2.** An energy storage system that meets the needs of the output of the thermoelectric system. The energy generated by the thermoelectric system may not be used immediately in the close vicinity, and thus the design of an energy storage system is required. At the same time, thermoelectric technology usually generates direct current power, which makes it necessary to have a comprehensive system including inverters and transformers.
- **3.** A coordinated design of thermoelectric systems and other road facilities. For example, it is considered by some researchers to connect the water supply pipe in the city with the low-temperature end of the thermoelectric system and make full use of the cooling effect of flowing water in the pipeline.
- **4.** Improve economic benefits of the thermoelectric systems. It is crucial to improve the energy output efficiency of the system and reduce installation costs and maintenance needs of the system. Whole life cost analysis is helpful to quantify the costs and benefits throughout the entire service life of the system, and using it in comparative studies of alternative energy harvesting systems and the do-nothing option will help road authorities to make informed decision on strategic investment and procurement.
- **5.** Engineering applications of the thermoelectric systems. Use the collected energy to provide power for roadside infrastructure such as lighting, signal control, communication, etc. In the light of large-scale application, research on a comprehensive design of the system, enhancement of the power generation efficiency, and cost effectiveness of the system during the service period shall prove to be valuable.

## 13.8 Sources of further information and advice

As an important means to harvest energy from the road, thermoelectric technology has many advantages such as simple structure, strong and durable, no noise and long service life, etc. It has received more and more attention in the field of road engineering. At present, the main obstacle to the application of pavement thermoelectric technology is that the temperature difference within the pavement is subject to climatic and seasonable variation and that the thermoelectric conversion efficiency of TEG is generally low. The advancement of road thermoelectric technology requires optimization in the design of pavement, structures, and thermoelectric system, as well as the development in related fields such as thermoelectric materials, heat transfer, and storage. Therefore the application of pavement thermoelectric technology requires collaboration by researchers from a number of disciplines such as engineering, materials science, physics, and thermodynamics. Finally, a highperformance pavement thermoelectric system, which is compatible with the ongoing development in road design and asset management, resilience, and automation, should be developed to promote the energy harvesting technology.

## 13.9 Acknowledgments

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# Piezoelectric energy harvesting from pavement



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## 14.1 Introduction

Energy harvesting is a promising technique that can help to solve the global energy challenge without depleting natural resources. As one of the green energy solutions, energy harvesting can reduce greenhouse gas emission generated with traditional energy sources. Piezoelectric, thermoelectric, electromagnetic, and photovoltaic techniques are some examples of energy harvesting technologies that can be used to collect energy from the ambient environment to generate electricity nowadays. These technologies work in different principles and thus have different application requirements and energy conversion efficiencies [1].

The roadway is designed to provide serviceability and durability under repeated traffic loading and climate conditions. The pavement deforms under truck loads and experiences high temperature in summer due to solar radiation. There are many opportunities to collect waste kinetic energy (vibrations, deflections, etc.) and environmental energy (solar, heat, etc.) from the roadway environment. In particular, mechanical energy can be converted into electricity via stress or vibration using piezoelectric transducers placed on pavement surface or embedded in the pavement. It is projected that truck traffic will continuously grow in the highway network of the United States due to the desire of economic development for freight transportation. Therefore, there is a high potential to harvest kinetic energy from pavement using piezoelectric materials.

This chapter provides a comprehensive review on the topic of piezoelectric energy harvesting from pavement. Various transducer designs of piezoelectric energy harvesting systems are introduced and their advantages and disadvantages are compared. The previous work that studied piezoelectric energy harvesting in the pavement was synthesized, including theoretical analysis, laboratory tests, and field trials. Different ways of using the harvested energy are discussed. Finally, the challenges and recommendations for future studies are provided.

## 14.2 Piezoelectric materials and principle of energy harvesting

Piezoelectric materials generate an electric charge when subjected to mechanical stress and change dimensions when an electric field is applied across the material. These are known respectively as the direct and the inverse piezoelectric effect [2]. Piezoelectric materials can be classified into the following categories: (1) single crystalline material (such as quartz), (2) piezoeramics (such as lead zirconate titanate, PZTs), (3) piezoelectric semiconductor (such as ZnO<sub>2</sub>), (4) polymer (such as polyvinylidene fluoride, PVDF), (5) piezoelectric composites, and (6) glass ceramics (such as Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> and Ba<sub>2</sub>TiSiO<sub>6</sub>). In addition, PZT fibers can be mixed with resin to form macro fiber composite or PZT composite. The mechanical and piezoelectric properties vary among these different piezoelectric materials. PZT material has been used in many applications among commercial products due to the high efficiency of electricity generation.

Increasing the applied stress or strain on piezoelectric material that provides more mechanical energy is one of the main methods to increase the power generation. The other method is to use the coupling mode more efficiently. Fig. 14.1 shows two possible coupling methods of piezoelectric material: the  $d_{31}$  and  $d_{33}$ mode, depending on material poling and applied force directions. The coupling mode is dependent on the relationship between the direction of the applied load and the poling direction. The coupling mode is  $d_{33}$  mode when the applied load is parallel to the poling direction, while the  $d_{31}$  mode is defined when the applied load is perpendicular to the poling direction. The  $d_{33}$  mode provides higher electromechanical coupling when compared with the d31 mode for most typical piezoelectric materials [4].

The piezoelectric charge constant (effective piezoelectric strain constant) and the piezoelectric voltage constant are governing the magnitude of energy conversion.



Figure 14.1 Piezoelectric material coupling modes.

*Source*: After [3] S. Roundy, P.K. Wright, J. Rabaey, A study of low level vibrations as a power source for wireless sensor nodes. Comput. Commun. 26 (11) (2003) 1131–1144.

Quality factor is another important factor of piezoelectric material because the greater quality factor indicates the less damping and heat generation during energy conversion. In order to increase the efficiency of piezoelectric energy harvesting, the preferred material properties are higher piezoelectric coefficient, higher quality, and lower elastic compliance.

For piezoelectric material, the stored electric energy under a low-frequency load can be calculated using Eq. 14.1 [5].

$$U_E = \frac{1}{2} PEAt = \frac{1}{2} V_0^2 \frac{S_r^T S_o A}{t}$$
(14.1)

where  $U_E$  is the electric energy storage in the piezoelectric device, *P* is the polarization caused by the vehicle load, *E* is the inner electric field,  $V_0$  is the electric potential in an open circuit, *A* is the area of PZT, *t* is the thickness of PZT, and  $s_r^T$  is the relative dielectric constant of PZT.

From Eq. 14.1, the density of stored electric energy can be obtained using Eq. 14.2.

$$U_E = \frac{1}{2}PE = \frac{1}{2}dgT^2$$
(14.2)

where d is the piezoelectric charge constant, g is the piezoelectric voltage constant, and T is the external stress.

From Eq. 14.2, it can be concluded that  $U_{\rm E}$  is related to  $(d \cdot g)$  value if the external stress is constant. Therefore, the PZT materials with a high  $(d \cdot g)$  value are improved for energy harvesting.

### 14.3 Piezoelectric transducer designs and types

For the application of piezoelectric energy harvesting in pavement, four common types of piezoelectric materials have been used, which are cantilever beam transducer, disk-/ rod-shaped transducer, cymbal/bridge transducer, and PZT composite layer.

#### 14.3.1 Cantilever beam transducer

Bimorph piezoelectric cantilever beam is assembled with beam material and two piezoelectric ceramic strips, conductive adhesive, and two piezoelectric films by series or parallel connection, Toward the cantilever end is a block that can enhance the vibration amplitude of cantilever beam as well as alter the vibration frequency. Fig. 14.2 presents the illustration of a bimorph cantilever beam.

Many factors can affect the energy output of cantilever beam, including mainly coupling mode, frequency, thickness and type of material used for each layer of the cantilever beam, total length of cantilever beam, and tip mass. Jiang et al. [6]



Figure 14.2 Illustration of a bimorph cantilever beam with PZT.

studied the effects of material thickness and tip mass of cantilever beam. They concluded that by reducing the cantilever thickness and increasing the tip mass, the maximum harvested power was achieved. The length and width of tip mass were also found affecting the output of the harvested power [7].

Kim et al. [8] assessed piezoelectric cantilever transducer and placed them on speed bumps and underground, respectively. They observed that the electric power generated from energy harvester on speed bump that was much less compared to the underground one. The harvested energy changed with vehicle speed and the cantilever vibration method.

#### 14.3.2 Disk-/rod-shaped transducer

The most frequently used piezoelectric transducer in pavement applications is disk/ rod-shaped PZT transducers due to its durability and easy fabrication, as shown in Fig. 14.3. The transducer has two main geometric variables, which are diameter and thickness. As the diameter-to-thickness ratio is equal to or more than 5, it is usually called disk-shaped transducers [9]. The geometry of PZT affects the stress applied on the top surface of transducer and thus the harvested energy. In order to optimize disk/rod geometry for energy harvesting application, the diameter-to-thickness ratio need be adjusted [10].

The generated energy output, however, tends to be small concerning a single piezoelectric transducer. Hence, multiple transducer arrays are often stacked, as shown in Fig. 14.4. Zhao et al. [11] analyzed the effect of piezoelectric element geometry on the performance of an energy harvesting unit. The study considered multiple PZT rods with circular, square, and hexagonal cross sections. The finite-element (FE) analysis results showed that circular cross-sectional rods were preferable to maximize power output.

Jiang et al. [12] created a two-degree-of-freedom model to analyze the energy harvester performance, which was made up of three piezoelectric stacks per unit. Using the findings from electromechanical model and experimental tests, it was clearly proven that the energy harvester was able to produce 200 mW electric power to cater to electrical devices used in transportation infrastructure.

#### 14.3.3 Cymbal and bridge transducer

The cymbal transducer can be regarded as a developed version upon Moonie transducer that can help diminish the PZT disk's stress concentration and enhance



Figure 14.3 Illustration of disk/rod-shaped PZT transducer.



Figure 14.4 Illustration of stacked PZT for energy harvesting.

displacement. It includes two metal end caps with one piezoelectric ceramic disk between them. The vertical load being applied on the end cap causes PZT disk having radial stress that results in electric field due to piezoelectric effect. The bridge transducer has a similar geometry to the cymbal transducer except that the shape is square instead of circle square. Fig. 14.5A and B illustrates the geometry of cymbal and bridge transducers, respectively.

Zhao et al. [5] used FE analysis for evaluating the cymbal transducer's performance and observed that the potential electric energy can be enhanced by increasing the transducer diameter and cavity base. Yao et al. [13] examined the arch and arc designs of steel caps in a bridge transducer, as shown in Fig. 14.5C and D, respectively. It was found that bridge transducer with arc cap created an electric voltage of 232 V under 0.4 MPa pressure, which was 78 V more compared to the bridge transducer with trapezoidal cap. They found that although the trapezoidal cap transducer could more effectively resist the applied pressure compared to the arc and arch structures, the energy conversion efficiency was higher when the curved cap was used.

In order to increase the power output of bridge transducer, Jasim et al. [14] and Yesner et al. [15] proposed the new layered poling pattern (electrode configuration)



**Figure 14.5** Illustration of PZT transducers for (A) cymbal, (B) bridge, (C) bridge with arc cap, and (D) bridge with arch cap.



Figure 14.6 Energy harvester with 64-transducer assembly [16].

that increased capacitance and piezoelectric coefficient of transducer and thus energy harvesting performance. In their study, the geometry of new bridge transducer was optimized, taking into account the effects of mechanical stress on material failure and power output. The results showed that the individual bridge transducer with the new design generated an electrical potential of 556 V, creating approximately 0.743 mJ of potential energy (open-circuit condition) under 0.7 MPa external stress. Jasim et al. [16] further assembled 64 bridge transducers into an energy harvester with aluminum packaging and evaluated its energy output and fatigue life through laboratory experiment and numerical simulation (Fig. 14.6). It was found that the simulation results were consistent with the measured power. The output power of 2.1 mW was observed at 400 k $\Omega$  resistive load when the load was 70 kPa at 5 Hz.



Figure 14.7 Illustration of PZT composite with conductive layers [18].

#### 14.3.4 PZT composite

Another type of piezoelectric energy harvesting is to transform the entire pavement layer into piezoelectric energy harvester using the composite of PZT fibers with epoxy (serve as energy harvesting layer). The PZT composite improves flexibility as compared to pure PZT and thus increases the fatigue life, although the energy output is reduced. Panda et al. [17] installed flexible ceramic/polymer composite strip on a test road as piezoelectric sensors for collecting traffic data, including weight-in-motion to measure large vehicle loads.

Guo and Lu [18] proposed an innovative energy harvester system by placing PZT composite sandwiched between two conductive asphalt layers (serve as electrodes), as shown in Fig. 14.7. The conductive asphalt layer can be made by mixing steel fiber and wool, graphite, or carbon fiber with asphalt, which may significantly reduce the electrical resistivity of asphalt concrete from 1011 to 10  $\Omega$ m. The electrical resistivity can further drop to 10 to 2  $\Omega$ m if the sand-asphalt mix was added with 8.76% of steel fibers [19]. Alternatively, nano iron oxide can be used for conductive asphalt if the concern is that steel fiber or wool might be electrically eroded under electric current.

#### 14.3.5 Placement of piezoelectric transducer in pavement

The geometric design of piezoelectric transducer decides not only the output power and power conversion efficiency of piezoelectric transducer, but also the way of placement in the pavement. The cantilever beam transducer is usually installed on pavement surface, while the disk/rod-shaped and cymbal/bridge transducer are usually embedded in the pavement layer. The PZT composite can be placed as a continuous layer in the pavement. The total harvested energy is small from single transducer or layer of PZT material. In order to increase energy generation, multiple piezoelectric transducers or layers can be assembled into an energy harvester system with appropriate configuration of electrodes. On the other hand, the placement of energy harvester may change pavement surface condition and the uniformity of pavement layers and affect long-term pavement performance [20].

To protect the embedded energy harvester from the impact brought by vehicle loading and ambient environment, protective package needs to be prepared with appropriate insulation. Different package materials vary in terms of stiffness compatibility with surrounding pavement materials. The protective package needs to have the capability to block fluid or any other chemical and contaminants from the hosting material. The piezoelectric material shall not have contact with any fluid, which may cause a short circuit of positive and negative poles of the material. Another important function of the protective package is the ability of transferring the stress applied by vehicles uniformly onto the piezoelectric material.

## 14.4 Previous studies of piezoelectric energy harvesting in pavement

#### 14.4.1 Theoretical studies

A number of theoretical studies have been conducted using analytical models and numerical simulations to study each influencing factor of energy harvesting and help to optimize the design of piezoelectric transducer. Table 14.1 summarizes theoretical studies in the literature on piezoelectric energy harvesting in roadways. The results show that the transducer configuration affects the amount of energy harvesting form pavement significantly. Papagiannakis et al. [21] and Roshani and Dessouky [25] used disk/rod-shaped transducers while Jasim et al. [14,16], and Moure et al. [22] used cymbal/bridge transducers. On the other hand, some researchers used multilayer stack to increase power generation, such as Hu [23] and Yang et al. [27]. Few studies used cantilever beam for energy harvesting in pavement except the one by Song et al. [24]. The cantilever device showed that 1 km of the road could generate around 2.95 kWh/h under vehicle wheel path, which is much smaller than those obtained from other types of transducers. Vehicle weight, loading frequency, loading magnitude, and embedded depth of the energy harvester module are important factors that determine energy harvesting performance. There is a positive relationship between the amount of generated energy and the loading frequency and magnitude.

#### 14.4.2 Laboratory and field studies

Laboratory and field studies have been performed to evaluate the actual power output of piezoelectric energy harvesting. Table 14.2 summarizes the laboratory field studies on piezoelectric energy harvesting from pavement. Testing scenario and piezoelectric transducer configurations are two important factors that affect the amount of generated energy. In general, all studies prove that using higher piezoelectric stress constant can improve the electrical output linearly. The stacked transducers were used to increase the amount of generated power. The disk-shaped transducers were tested by Roshani et al. [32,33], Xiong and Wang [34], and Wang et al. [36]. Several field studies were conducted for cantilever beam and disk/rod-shaped piezoelectric transducers [34,35]. The field study by Innowattech company is the only study that used large amounts of units to cover 1-km road [30]. The Innowattech claimed that their technology could produce 200 kWh/h from 600 heavy vehicles per hour traveling at a speed of 72 km/h.

Simulation assumption	Transducer configuration	Power output	Reference
	Rod elements	Prototype I and IV generated 10 and 241 Watt-h	[21]
		per year/module under 30,000 average annual	
		daily traffic (AADT)	
	Cymbal	Single transducer generated 16 $\mu$ W under one	[22]
		heavy vehicle wheel	
	PVDF multilayer harvester	N/A	[23]
	Piezoelectric cantilever beams	N/A	[24]
340, 680, 1020, and	Package of 36 layers of stacked	The power increased from 25 to 200 mW as the	[12]
1360 N; 1, 2, 4, and 6 Hz	piezoelectric wafers	frequency increased from 1 to 6 Hz	
2.1–5.6 kN	One energy module contains 4, 8, or 16	The greatest output surpasses the maximum	[25]
	PZT disks, sandwiched by two copper	voltage measuring ability of the oscilloscope	
	plates with 1.5-in. asphalt layer at the	set at 80 V, when the system has 4 stacks of 2	
	top	PZTs or 8 stacks of 2 PZTs	
30, 60, and 120 m/s vehicle	Thin rectangular piezoelectric transducer	At 30 m/s, the system produced around 41.2 and	[26]
speed; single wheel load	size: $100 \times 100 \times 10$ mm; depth of	47.26 mW under single and four-wheel load,	
or four-single-wheel load	PZT in pavement was 5 cm	respectively	
Under vehicle dynamic	Nine piezoelectric disks (PZT-5H)	The average output was 174 V	[27]
loading of 0.7 MPa	stacked in parallel inside		
(laboratory test)	$30 \times 30 \times 6.8$ cm box (multilayer stack)		
0.7 MPa tire load using finite-element simulation	Bridge shape transducer with parallel electrode	The energy module with gap can produce 28.7 mW at 5 Hz	[14]
Mechanical testing and	Ball-type protective pad	Maximum output power of 50.41 mW	[28,29]
simulation with loading			
of 0.7 MPa and 15 Hz			
frequency			

 Table 14.1 Theoretical studies on piezoelectric energy harvesting from pavement.

Test scenarios	Configuration	Electric output	Reference
Under 600 heavy vehicle/hour traveling at a speed of 72 km/h	Piezoelectric generators with a mechanical- electrical association	Piezoelectric generator produced about 200 kWh per kilometer	[30]
Simulated traffic speeds of 14–116 km/h	80 piezoelectric generator cables	48.2 μW due to heavy traffic or 2.6 μW without heavy traffic	[31]
20 km/h vehicle speed for speed bump and 28 km/h for underground	Cantilever energy harvester with 24 or 72 transducers	The output was 7.61 mW for speed bump while the underground generated 63.9 mW	[8]
Laboratory condition to simulate 8 km/h, tire pressure of 0.82 MPa, and load of 40.03 kN	Cylindrical piezoelectric unit	The energy output estimated per prototypes was 360 W-h annually	[32,33]
Trucks at weight stations	PZT disks	The average power output was 3.1 mW per truck. Only 15% of the applied loading was transmitted to piezoelectric materials	[34]
Average load of a single wheel was 4.41 kN at 2–7 km/h	A multilayer stack array piezoelectric transducer	The open-circuit voltage reached 280 V	[27]
Speed range from 5 to 20 km/h at the field	Cantilever beam- based piezoelectric	The total energy generated by one piezoelectric cantilever from one car is 1.26 mJ	[35]
Wheel load of 25 kN and contact stress at 0.7 MPa and 10 Hz	Disk shape	Output power was 22.8 mW	[36]

 Table 14.2
 Laboratory and field studies on piezoelectric energy harvesting from pavement.

## 14.5 Use and storage of harvested energy

The application of piezoelectric materials provides an alternative source for electrical power generation. Piezoelectric materials can be used for effective energy harvesting by embedding or attaching the harvester module with pavement. The harvested energy can be used for various applications such as traffic signs, lighting,

	Supercapacitor	Lead-acid battery
Energy efficiency (%)	85–98	85-90
Self-discharge rate (%)	14	2
Energy density (Wh/kg)	5	30
Lifetime (year)	12	5-12

**Table 14.3** Performance comparison of supercapacitor and rechargeable battery [18,37–39].

and sensor power source. This saves the use of battery or the connection with power grid, which brings significant advantage in remote areas. It is noted that a electrical circuit is needed to collect the generated energy for different applications. The actual power output from energy harvesters can be lower than from theoretical analysis due to the power loss with electrical circuits. Therefore, it is very important to design the electric circuit efficiently to minimize power loss during transmission.

On the other hand, the harvested energy can be stored for future use. Two energy storage devices (rechargeable battery and supercapacitor) are commonly used with energy harvesters for energy storage. The supercapacitor is an electric double-layer capacitor and has superior performance in energy efficiencies, self-discharge rates, energy densities, and lifetime [37-39].

Sodano et al. [40] were able to recharge a 40 mAh battery through the power from PZT device in less than 30 min, showing that the battery and the PZT device were suitably paired. In addition, the rechargeable battery was shown to have greater beneficial properties when it comes to power harvesting, compared with a traditional capacitor, particularly when the constant power supply is necessary. In order to evaluate the storage of energy produced by the PZT energy harvesting system, NiMH rechargeable battery was used in the field test. It was found that the battery pack increased to 2 V in the first day quickly and reached 3.6 V 6 days later [41].

Recently, supercapacitors are proposed for storing energy with superior properties than rechargeable batteries, including energy efficiency, self-discharge rate, energy density, and lifetime [37–39]. The comparison of performance between supercapacitor and rechargeable battery is shown in Table 14.3. In order to produce the greatest amount of power possible in these random traffic conditions, a diode rectifier should be involved, which can be linked with PZT units. The loading resistors can be used to change the voltage on the charging supercapacitor, depending on the application of interest [37,40,41]. For most cases, the majority of circuits created to gather electrical energy from the piezoelectric generator are primarily made up of rectifiers, capacitors, and resistors.

#### 14.5.1 Challenges of piezoelectric energy harvesting in pavement

In piezoelectric energy harvesting, the devices are often designed to allow the harvested power to recharge the batteries that are part of the system [42]. However, the energy conversion efficiency of the batteries is usually variable and often quite low, which means that the efficiency of the entire piezoelectric energy system is highly dependent on battery technology [43]. As the piezoelectric sensors are embedded within the layers of the pavement, using rechargeable batteries may not be a practicable solution as the battery has its lifespan. However, if the electric energy produced by the system is used as it is generated, then the energy harvesting system becomes self-sufficient, reducing or even eliminating the dependence on batteries that have limited storage capacity. Utilizing a self-sustaining model also means that there is less waste, as all the energy can be used, unlike the battery system in which some of the generated energy is wasted. Synchronization of energy supply to demand can be made possible by adjusting the density of the sensors underneath the pavement layers to release the appropriate amounts of energy, but in order for this to be an effective approach, it requires an understanding of the energy demands in the particular location [44–47].

The service life and durability of piezoelectric transducers need be considered in roadway applications. There are three factors affecting the service life of piezoelectric transducer: mechanical loading, piezoelectric material fatigue, and electrode fatigue. Although the disk/rod-shaped PZT has high durability, the total energy that can be harvested is small. Other types such as cymbal and bridge transducers have much higher energy output, but they are subject to fatigue failure due to the tensile stress generated in the PZT material.

With regard to the maintenance of piezoelectric energy harvesting system in pavements, the repair or replacement of transducers needs to be coordinated with pavement rehabilitation treatments such as milling and overlay. Otherwise, it will add cost of pavement construction. Due to the lack of field data, the failure rate of piezoelectric transducers and the repair and replacement cost data are still very limited. The life-cycle cost of piezoelectric energy harvesting system needs to be investigated for any step toward adoption of piezoelectric energy technology in large scales of roadway network.

The environmental impact of piezoelectric material such as the toxic lead content associated with lead zirconate titanate that is the material most widely used by manufacturers needs to be studied [48]. Some studies have suggested a number of alternatives using lead-free materials that may be biocompatible [48,49]. The use of these alternatives requires further research on their environmental credentials [50].

## 14.6 Summary and recommendations

Piezoelectric energy harvesting shows a high potential to generate renewable clean energy in a distributed manner. The main types of piezoelectric transducer include cantilever beam, disk/rod shape, bridge, cymbal, and layered PZT composite. Different types of piezoelectric transducer vary in the mechanical stress generated in the transducer that affects both the fatigue life and power output of transducer.

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The section of piezoelectric transducer design should be based on the requirement of placement method and the demand for the generated energy.

Further research is needed for large-scale applications, in addition to the improvement of material durability and reduction of material costs. It is important to note that the applicability of piezoelectric energy harvesting may vary depending on the functional classification of roadway and climate conditions. The major roadway has a high volume of truck traffic and high design speed, which are beneficial for the power output. On the other hand, local roads especially can use the harvested energy for the passive safety device or lighting as the vehicle approaches. This advantage is more obvious in remote areas where the power grid is not applicable.

It is expected that the piezoelectric energy harvested from pavement may not be applicable for power grid applications. It is more practical to apply harvested energy for roadway-related applications that avoid power loss during transmission at the same time. The major benefit is that the piezoelectric energy harvesting system provides a distributed system to generate electricity.

Furthermore, piezoelectric transducers can be used to monitor traffic information in real time, such as traffic volume, speed, and even axle weight, which are essential data inputs for traffic management and weight-in-motion system. With the advancement of electric cars and autonomous vehicle technology, energy harvesting from roadway turns roadway into distributed energy generator for traffic devices, monitoring sensors, vehicle-to-infrastructure communication. It is possible that the harvested energy can be used for electric vehicle charging in the future due to its distributed nature of electricity generation.

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## Inductive power transfer technology for road transport electrification

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## 15.1 Introduction

#### 15.1.1 Background

In recent decades, many innovative concepts to make the road infrastructure be able to equip with other new functions have become a new subject of research. The development toward multifunctionality would maximize the utilization efficiency of the infrastructure and, importantly, promote sustainable development in the road transport sector as a whole. To date, a multifunctional road may embrace some of the following features: (1) intelligent communication: sensor technologies that enable improved communication between the infrastructure and vehicles or human, which could promote safety and efficiency in traffic control, or provide cost- and energy-efficient maintenance solutions for the infrastructure by timely monitoring its structural health. (2) Energy harvesting : Novel systems, for example, piezoelectric harvesting units [1,2], solar panel [3], or conductive pipe [4], embedded inside or on top of the road to collect solar energy and, with application for snow melting and deicing in winter, lighting for the traffic or charging other battery-powered sensors around. (3) Autonomous driving : Improved positioning control of autonomous driving can be realized by a combination of detecting the existing physical landmarks such as lane markings, road edges, barriers, traffic signs [5], and artificial roadway-embedded landmarks such as permanent magnets (possibly more robust due to the minimized climatic influence) [6]. (4) On-the-Road charging: Technology that is integrated into the existing roadway surfaces to deliver electrical power to an electric vehicle (EV) in-motion, either in a conductive or contactless way [7].

From the road infrastructure's point of view, the integration of these innovative functions into the existing roads can become a completely new multidisciplinary area of research, which is an important opportunity for the future development in infrastructural engineering. On the other hand, however, our current road design methods, construction, and maintenance principles do not necessarily guarantee that such an integration of new function unit would not affect the long-term performance of the road structure and vice versa. In other words, a premature damage or malfunctioning failure within the integrated system would greatly threaten its feasibility of the use in practice. Therefore, this study aims to deal with this major "compatibility" concern by conducting a comprehensive feasibility analysis of one specific multifunctional road system called the electrified road (eRoad) that enables on-the-road charging function based on the inductive power transfer (IPT) technology.

## 15.1.2 IPT technology for on-the-road charging

The decarbonization in road transportation by using different EVs has given high hope to enhance sustainability in the road transport sector, whereas the actual development is still in its early stage due to the restraints imposed by the on-board energy storage technologies. In order to solve this issue, alternative solutions that allow EVs having easy access to battery charging away from home have been under active development in the past years. These technologies show a possibility to fill the technical gap and support the road electrification at large scale. Particularly, the IPT technology, which allows for wireless charging of EVs and with a possible extension to be in a dynamic way, has gained significant interest worldwide. Being a near-field wireless power transfer technology, the IPT technology can inductively deliver electricity to a receiver device with high power but limited air gap distance. Significant progress in terms of the charging power, transfer distance and efficiency, safety of an IPT system has been made in the last decade, greatly pushing forward its implementation into practice. As illustrated in Fig. 15.1, an IPT system consists of an on-board device and an off-board power delivery device. The working principle is that the on-board part can pick up high-frequency alternating current through magnetic induction and change it into a direct current to charge the battery installed on vehicle body. To detail, the off-board system, which will be embedded inside the road pavement, has three major parts: (1) the power supply that provides a suitably regulated direct current output voltage via a rectifier; (2) a converter to provide high-output frequencies, combined with capacitance to achieve resonance with the transmitter and reduce the switching loss; and (3) a transmitter that is mutually coupled with the pick-up device, mainly consisting of conductive coils, ferrite cores, and a backing plate. The functional performance of an IPT system,



**Figure 15.1** Working principle of an inductive power transfer system. *Source*: Reprinted from F. Chen, N. Taylor, N. Kringos, Electrification of roads: opportunities and challenges. Appl. Energy 150 (2015) 109–119.

mainly in terms of the power transfer efficiency and distance, is influenced by many parameters such as frequency and current in primary charging coil, the distance and the lateral misalignment between the off-board and on-board parts, and the geometry of the coils' layout design. This technology has been under active investigation and many prototypes have been demonstrated around the world.

## 15.1.3 Infrastructural challenges for IPT-based eRoad system

In order to achieve a sustainable implementation of an IPT-based eRoad system, many feasibility studies have been actively advancing, in terms of such as the performance of the charging system (e.g., power efficiency, transfer distance, safety), electricity grid, customer acceptance, legislation, social and environmental benefits and impacts. From the road infrastructural perspective, the eRoad should fulfill the long-term performance requirements for serving as an ordinary road for driving on and, at the same time, efficiently delivering electric power to the moving EVs. However, our current technology and knowledge bases in road engineering are not necessarily ready for a successful integration of such a system into a road structure. As illustrated in Fig. 15.2, the emergent technical challenges of integrating the IPT technology into the physical roads have been suggested by the authors of this chapter, which arise from two major aspects: (1) the influences on the structural performance of the eRoad under operational conditions due to the embedment of the charging technology; (2) the effect on the charging efficiency of the IPT system due to the integration, for example, the power loss caused within eRoad materials when alternating magnetic fields pass through them. In addition, even those technical challenges can be resolved and the technology maturity is close to a deployment level, it could still be important to reflect upon the real environmental benefit of using the technology. This should be achieved by performing a thorough assessment of life-cycle sustainability performance of the eRoad infrastructure, as well as the consequential influences to the holistic sustainability of an eRoad transport. All these technical and environmental concerns are playing the role of core importance in a sustainable implementation of the eRoad technology. Research studies focusing on those concerns will be detailed in the next sections of this chapter.



Figure 15.2 Technical challenges for integrating IPT charging system into a asphalt road pavement.

## 15.2 Structural analysis of the eRoad system

The most important components in IPT-based eRoad system are the charging unit (CU) slabs that are made of: (1) cement concrete module and (2) power electronics consisting of the charging system such as conductive coils and magnetic ferrites. In that, the use of cement concrete module is to hold the venerable changing system electronics and protect them from traffic-induced mechanical damages. In order to successfully embed the IPT charging components into the road pavement structures, two different construction technologies have been considered: (1) prefabricationbased construction method : The CUs are built in prefabricated modules in factory and then finishing the on-site embedment in a relatively short period; (2) in situ construction method : This is to install all the facilities on site, that is, the IPT facilities are fixed in excavated pavement, which is then sealed by cement concrete material and further protected by applying an asphalt overlay on the top. For both methods, the practical constructions can be done either in a limited trench lane or even by making a replacement of the full lane. Supposing the construction of eRoad system is realized by embedding the CUs into a traditional road (tRoad), either in a trend or full-lane manner, its structural response could be quite different from that of a tRoad structure. Hence, it could be of great importance to assess the severity of the potential influences on the long-term structural performance of the eRoad system under operational conditions. In this section, experimental tests in the laboratory, together with numerical simulation investigations using the finite element (FE) method, are presented.

## 15.2.1 Mechanical loading on small-scale eRoad structural sample

In order to demonstrate the possible damages within such kind of composite structure, small-scale eRoad specimens were prepared and used for performing mechanical loading tests in the laboratory. To do so, cylindrical specimens with 150 mm height and 100 mm diameter were compacted using asphalt material, in which onequarter of a concrete cylinder was inserted so as to represent the CU module. The small-scale eRoad sample was subjected to repeated compressive harvesine loading of 10,000 cycles (0.1 s loading and 0.9 s resting time per cycle). In order to simulate the effect of traffic loading, the magnitude of compressive load was 800 kPa, while, in the meanwhile, a static confining pressure of 140 kPa was applied. The test was conducted at 40 °C by using the simple performance tester machine. Computed tomography (CT) scanning test was conducted to obtain the microstructure within eRoad sample prior to and after the loading, using an X5000 system from North Star Imaging. According to the CT images shown in Fig. 15.3, a number of physical changes within the composite structure can be observed after cyclic loading: (1) Significant damage is observed in asphalt material section, indicated by the large permanent deformations (plastic flows) and the microcracks (continuum damage). The damages are nonuniformly distributed, with a concentration around



**Figure 15.3** CT scanning test of e-Road small-scale samples: (A) prior to and (B) after 10,000 cycles of dynamic loading.

*Source*: Reprinted from E.C. Ledesma, F. Chen, R. Balieu, N. Kringoss, Towards an understanding of the structural integrity of electrified roads through a combined numerical and experimental approach, in: Compendium of Transportation Research Board 96th Annual Meeting, 17-02932, 2017.

the CU shoulder. (2) A dramatic separation between the CU concrete region and asphalt region is demonstrated, which shows a significant debonding failure at the interface. (3) The concrete component seems to be safe under the assumed loading condition, displaying that concrete material has enough resistance to the mechanical loading under typical traffic loading. Hence, from a technical (probably as well economical) point of view, cement concrete could be a good choice for using as the module material in CU fabrication.

These experimental observations demonstrate the major failure modes within the composite eRoad structure. In the next section, these experimental results would serve as an important basis for the development of the FE tool and by using which the structural behavior of the eRoad system at full scale can be rationally investigated.

### 15.2.2 Finite element modeling of eRoad structural responses

The cross-sectional geometry of a potential eRoad structure following the trend lane construction method is shown in the FE model in Fig. 15.4, where the CU slabs are accommodated within the asphalt layer of a tRoad and successively embedded in the middle of the lane. In order to realistically simulate the mechanical responses of the eRoad system under continuous traffic loading, it is important to have a good simulation tool that could model the important physical changes that are prone to occur under continuous loading, especially the above phenomena



**Figure 15.4** Finite element model for 3D structural analysis. *Source*: Reprinted from F. Chen, R. Balieu, E. Córdoba, N. Kringos, Towards an understanding of the structural performance of future electrified roads: a finite element simulation study, Int. J. Pavement Eng. 20 (2019a) 204–215.

observed experimentally. Actually, Chen et al. [9,10] developed such an FE tool that successfully addressed those important concerns. The important features of the model are briefly introduced as follows:

- 1. An energy-based constitutive model suitable for asphalt material is used to study the deformations and damages within the asphalt layer of eRoad structure. In that, three individual models have been developed, which are (1) a time-dependent viscoelastic model to account for the recoverable part of deformation under traffic loading, (2) a time-dependent viscoplastic model to account for the irrecoverable part of deformation under loading, and (3) a continuum damage model that coupled to the viscoelastic and visco-plastic models, with the aim to simulate the induced material damages within asphalt continuum under different stress conditions (e.g., microcracks or cohesion failure between mastic and aggregate stones).
- **2.** A cohesive zone model is adopted for simulating the interface debonding failure between the CU slab and asphalt and, based on which, the influences of different interface conditions on the overall structural responses can be examined.
- **3.** Realistic loading condition is used by applying 3D nonuniform tire-pavement contact stresses on pavement surface, which is transiently moving along the road lane in a quasi-static way. Moreover, the effect of tire braking and accelerating is further introduced by applying a horizontal frictional force on pavement surface along the moving direction.

## 15.2.2.1 Stress and strain responses in an eRoady at varying traffic conditions

Assuming the eRoad pavement in Fig. 15.4 is subjected to one single wheel pass of heavy vehicle loading, which carries a load of 50 kN. According to the FE simulation, the maximum longitudinal strains induced by free rolling at different speeds are found to be at the center of the loaded area, which are further plotted versus pavement depth in Fig. 15.5A. The result shows that (1) for both eRoad and tRoad, highly compressive strains  $\epsilon_{yy}$  are generated on road surface, which gradually shift to be tensile and approach the cutoff on top of the base layer; (2) due to the visco-elastic nature of asphalt material, for both eRoad and tRoad, a decrease in traveling speed would increase the longitudinal strains  $\epsilon_{yy}$  at varying depths; (3) importantly as well, for all case scenarios, significantly lower strain amplitudes are observed in eRoad pavement than that of the tRoad pavement. In a normal pavement design manual, one important criterion for controlling the fatigue life is to limit the maximum tensile strain at the bottom of the asphalt layer. Hence, the simulation results show that the stiff CU slab and high-speed rolling could prevent the structure from bending much, delaying thus its fatigue failure potential.

Fig. 15.5B presents the vertical deflections induced at varying depths of eRoad and tRoad, indicating as well that the decrease in vehicle speed incurs higher deflections in both pavements. In comparison with the tRoad, fewer deflections are observed in different layers of the eRoad, owing to the restraint imposed by the stiff CU slab. Vertical deflection at the top of the base layer is normally given focus in restricting the permanent deformation (i.e., rutting) in pavement. Thus, the FE prediction result underscores that the resistance to rutting is fairly well for the eRoad structure, or at least no worse than that of the tRoad. However, one should also



**Figure 15.5** (A) Maximum longitudinal strains ( $\epsilon_{yy}$ ) and (B) maximum deflections at varying depths of the eRoad and tRoad pavements, induced by free tire rolling at different speeds [10]

note these strain and deflection analyses are tenable only when the tire is rolling above the CU, which do not guarantee that less rutting occurs as well in other transverse locations that away from CU.

# 15.2.2.2 Influence of interfacial bonding on eRoad structural performance

The above analyses are based on the fact that the interfaces, especially the ones between CU and asphalt, are fully bonded. However, in practice, it is not easy to guarantee that the eRoad structure is constructed and maintained in a way that having good mechnical integrity in its long service lifetime. In fact, even for a tRoad, the interfaces between different layers could debond if construction defects are present or after long-time exposure to the traffic and environmental loading (e.g., moisture). This raises up the concern over the possible consequential effects of interface debonding to the mechanical performance of the whole structure. Hence, FE simulations are further carried out at varying interface and rolling conditions. In the scenario case study, the rolling speed is assumed to be 8 km/h while the effect of braking force is imposed by applying a horizontal force on pavement surface along the moving direction, which is 0.5 times the vertical tire-pavement force (0.5 is the frictional coefficient). For the interfacial condition between the CU and asphalt, the following scenario cases are considered: (1) undamaged interface with high bonding (HB), (2) undamaged interface with weak bonding (WB), and (3) damageable interface with debonding potential (DB). Moreover, other interfaces between different structural layers are assumed to be fully bonded, so as to reduce the complexity of analysis. Fig. 15.6 presents the simulation results of maximum longitudinal strains  $\epsilon_{yy}$ and in-plane shear strains  $\epsilon_{vv}$  at the surface and bottom of eRoad asphalt overlay, induced according to the above interface and rolling conditions.

Fig. 15.6A indicates that when under the free rolling, the longitudinal strain  $\epsilon_{yy}$ values are similar for WB and DB interface conditions while their amplitudes (in compression) are slightly lower than that for HB interface condition. The reason could be that the WB and DB interfaces allow relatively easier movements between the layers, dissipating thus more stresses induced during bending. However, this trend is reversed due to the tire braking, and importantly, significantly higher longitudinal  $\epsilon_{vv}$  strains in tension are induced at the pavement surface when the interface is weakly bonded or damaged. This is mainly related to the significant longitudinal stresses induced by the braking action. As a result, the longitudinal  $\epsilon_{vv}$  strains increase and these strains aggravate in further when the interfaces are not well bonded. Considering the fact that asphalt material performs very weak under tensile loading, the eRoad pavement surface could fall into the danger of severe tearing as well. Apart from that, Fig. 15.6B and C further denotes if WB or DB interfaces are present, significant in-plane shear strains  $\epsilon_{vv}$  would be induced at pavement surface by the tire braking, particularly around the lateral edge of the tire. To sum up, the simulations support that tire braking and accelerating actions could incur higher potential of surface-related distresses in eRoad, which could be further aggravated due to the loss of interface bonding.



**Figure 15.6** Strains at the (A–C) surface and (D–F) bottom of asphalt overlay in eRoad structure, under varying interfacial and rolling conditions. *Source*: Reprinted from F. Chen, C.F. Coronado, R. Balieu, N. Kringos, Structural performance of electrified roads: a computational analysis, J. Clean. Prod. 195 (2018) 1338–1349.

In Fig. 15.6D–F, similar trends are noted for the strain responses at the bottom of asphalt overlay. Significant strain increases at the bottom of asphalt overlay due to the vehicle braking are observed, which could adversely accelerate the degradation of interface bonding with CU, and vice versa. As a consequence, failure of the asphalt overlay, for example, bottom-up fatigue cracking, could be expected at a faster pace.

From all these, one can estimate that the interface bonding property could play a significant role in pavement performance of the integrated eRoad system. Significant loss of bonding at the CU-asphalt interface could notably intensify the tensile and shear actions in asphalt overlay, leading to the accelerated distresses and more frequent maintenance of the overlay.

## 15.2.2.3 Structural optimization by geometrical design

As indicated above, good bonding at different interfaces is helpful for improving the integrity of the whole structure and thus delaying the potential damage failures. In addition to that, it could be valuable to explore the other potential measures that could contribute for the same purpose. One of those could be to optimize the geometrical design, for example, using a fillet shape of the CU shoulder rather than the assumed sharp rectangular corner. To evaluate the effectiveness, a parametric sweep study is carried out by using the fillet shoulder with varying radius (r) of 3, 6, 9, and 12 cm. Load is applied at a critical location on eRoad pavement surface close to the CU. The induced von Mises stress and the damage scalar D distributions in the eRoad structure are shown in Fig. 15.7. One should also know that the higher the von Mises stress is, the easier the degradation of material could occur, in the form of a combination of plastic deformation and cracking. Meanwhile, the damage scalar D is a variable in the asphalt constitutive model, which varies from 0 to 1. When D = 0, no damage is indicated in asphalt continuum; when D = 1, the continuum is fully damaged or cracked.

From the comparisons in Fig. 15.7, one can note that by increasing the size of the fillet radius, the amplitudes of von Mises stress and damage scalar D decrease in a dramatic way. For the last case, that is, fillet shape with r = 12 cm, the maximum values of von Mises stress and damage scalar D are 0.69 MPa and  $1.1 \times 10^{-2}$ , respectively. These could still be higher than that of the tRoad, but has been significantly reduced by this geometrical optimization. Therefore, it can be concluded that an introduction of the fillet shape of CU shoulder can effectively reduce the damaging potential within eRoad structure, which can also be very easy to achieve in a practical fabrication. Apart from that, some further case studies in Ref. [9] demonstrate: (1) Using alternate materials with lower stiffness than the rigid cement concrete can be useful to decrease the high-stress fields induced within the structure and, so as to reduce permanent damages in asphalt. However, alternate materials should also not be too soft because, in the meantime, they need to protect the charging system and itself from damages by external loading. (2) The way of increasing the thickness of asphalt protection overlay should be considered carefully in an actual situation because its real effectiveness is not distinct in the simulation.



**Figure 15.7** Distributions of von Mises stresses (800 kPa, 0.1 s) and damage scalar *D* (800 kPa, 5000 s) within the optimized eRoad structure, using circular fillet shape CU shoulder and with a radius of (A) r = 3 cm, (B) r = 6 cm, (C) r = 9 cm, and (D) r = 12 cm. *Source*: Reprinted from F. Chen, R. Balieu, E. Córdoba, N. Kringos, Towards an understanding of the structural performance of future electrified roads: a finite element simulation study, Int. J. Pavement Eng. 20 (2019a) 204–215.

## 15.3 Road material's dielectric influence on eRoad system

As previously explained in Section 15.1.3, road materials' dielectric influence could potentially threaten the power transfer efficiency of an IPT system, which is discussed in detail in this section. In fact, the potential power losses in an IPT system during its charging process could come from several sources, which are (1) the conductive coils' Joule loss due to its resistance, (2) eddy current loss in ferrite cores, and (3) switching loss in high-frequency power electronics. Therefore, in order to ensure high-power transfer efficiency of an IPT system, it is important to minimize these losses as much as possible during the actual design. After integrating the IPT technology into the practical roads, another potential power loss source can be the electromagnetic (EM) power loss caused in the physical media that the highfrequency magnetic fluxes would pass through. To date, this latter one has received very little attention but could have a possible impact on the resulting functionality of the system. A common equation for assessing the power loss in a dielectric medium, caused by the presence of a sinusoidal electric field at frequency f, uses the equation following [11]:

$$\mathbf{P}_{loss} = 2\pi\epsilon \mathbf{0}\epsilon'' |\mathbf{E}|^2,\tag{15.1}$$

where  $P_{loss}$  is the absorbed power per unit volume (W/m<sup>2</sup>) as a time-average value for the periodic excitation;  $\epsilon'' = \epsilon'_r \tan \delta$  is the imaginary part of permittivity  $\epsilon_r$ , that is, dielectric loss factor; IEI (V/m) and *f* (Hz) are the magnitude and frequency of the electric fields;  $\epsilon_0$  is the vacuum permittivity (8.854  $\times 10^{-12}$  F/m).

From Eq. 15.1, it can be seen that this dielectric power loss is governed by (1) the frequency and amplitude of the electric fields and (2) the dielectric loss factor of the lossy medium. Based on this equation, an analytic model for prediction of this dielectric loss magnitude in an actual eRoad constructed with IPT technology was proposed by [12]. In that, frequency and amplitude of the electric fields induced by typical IPT charging systems were reviewed and characterizations of the dielectric properties of typical pavement materials, with and without moisture conditioning, were carried out. Thereafter, the dielectric power loss based on the model calculations were performed and presented in Fig. 15.8.

The result in Fig. 15.8 shows that the electric power losses caused by asphalt concrete (AC) and Portland cement concrete (PCC) materials (in both dry and wet states) are frequency-dependent, indicating a strongly super-linear relationship. This potential power loss caused by the studied materials is all lower than 0.1 W when the frequency controlled to be below 200 kHz (typical frequency range of an IPT system). For an



Figure 15.8 Prediction results of dielectric power loss within typical pavement construction materials.

*Source*: Reprinted from F. Chen, N. Taylor, R. Balieu, N. Kringos, Dynamic application of the inductive power transfer (IPT) systems in an electrified road: dielectric power loss due to pavement materials, Constr. Build. Mater. 147 (2017) 9-16.

IPT system, the amount of power loss is therefore very small in comparison to the transferred power (normally in kW). In other words, the calculated dielectric loss is unlikely to cause a dramatic decrease in power transfer efficiency of a current IPT system, which is actually a very positive finding. However, if large scale deployment of wireless IPT charging solutions becomes a reality in future, it is still very meaningful to try to minimize this energy loss; in the meantime, the assumptions of this study are based on the normal scenarios whereas some other special situations could also affect. Hence, some further considerations in the practice which, could contribute to the reduced power losses in road materials, are summarized as follows:

- 1. When considering the IPT system design, if possible, it could be helpful to reduce the working frequency and the amplitude of magnetic flux density (and thus the magnitude of electric fields).
- For the selections of eRoad pavement materials, the following advice can be useful:

   Because of the lower dielectric loss factor of asphalt concrete materials, the use of which should induce less power loss than that of cement concrete materials.
   Moisture appears to be a significant factor that may increase the dielectric loss dramatically; thus, to some extent, the adoption of a waterproof measure can be helpful.
   Different AC types may have different dielectric loss factors, for example, porous asphalt mixtures that possess higher moisture ingress potential, as well as new asphalt mixtures with alternative aggregates that have unknown permittivity behaviors, should better be excluded.
   Other environmental effects, such as accumulations of snow or ice in winter as well as infiltration of deicing material, could increase the dielectric loss and require further study.
- **3.** If road material possesses a higher magnetic permittivity, it may also induce a direct magnetic loss and influence transfer efficiency. This concern is raised up due to the fact there is an increasing trend of using industrial wastes or other recycled materials in pavement construction, whereas the EM properties of which may be unknown. Meanwhile, some other studies try to make pavement materials have some EM property, with the application in, for example, extending the service life of the pavement material by improving its self-healing potential by induction heating [13]. Hence, it is also quite important to be aware of the characteristics of different smart functions and, coordinate carefully during design of future smart road that caters to enabling these new functions.

## 15.4 Sustainability assessment of eRoad system

In comparison to the conventional vehicles, EVs can greatly reduce energy consumption and greenhouse gas (GHG) emissions, especially when the energy source is shifted from high GHG intensity power sources (e.g., oil, coal) to cleaner energy sources (e.g., wind and solar powers) [14–18]. However, most of the available environmental studies do not necessarily take the practical road infrastructure into their life-cycle assessment framework. In fact, many research studies have shown that road infrastructure produces a significant among of GHG emissions in its service life, for example, the studies by Butt [19] and Miliutenko [20]. In terms of an eRoad system, new infrastructural materials, new construction works, and extra

waste disposal can be involved, whereas the consequential life-cycle environmental impacts are unclear. The neglect of these impacts could risk the real effectiveness of using EVs to enhance the sustainability in road transport sector. Hence, a quantitative assessment of the life-cycle environmental impacts of typical eRoad systems was conducted by Balieu et al. [21], which is further compared to that of a tRoad. In that, a life-cycle assessment framework suitable for eRoad is established, assuming a functional unit of 1 km and a total service lifespan of 20 years. In a scenario case study, besides the IPT technology, two extra dynamic powering technologies are considered, which are the conductive rail solution and pantograph solution. The conductive rail solution is to supply the electric power to an EV from the rails located in the slot of a conduit mounted on roadway surface, via a movable arm. The pantograph solution is to transmit the energy from the overhead contact lines to the electric motor of a vehicle via a pantograph mounted on top of the truck. Important boundary conditions, in terms of such as material production, on-site construction, and long-term maintenance and rehabilitation operations, are carefully defined. An example of life cycle calculation result is presented in Fig. 15.9, in which the following important considerations are rationally taken into account:

- 1. In comparison to a tRoad, the major new impacts during material production, transportation and pavement construction processes of the eRoad come from the charging system side, which are (1) coils and cement concrete for IPT-based eRoad solution, (B) steel rails for the conductive rail solution, and (3) no change for pantagraph solution, because no pavement modification is needed for this solution.
- **2.** In order to ensure high efficiency of the power transfer, the frequency of the winter maintenance operations for the charging solutions of IPT and conductive rail is doubled in comparison to that of a tRoad.



**Figure 15.9** Life-cycle environmental impacts (CO<sub>2</sub> emission) of different road systems. *Source*: Reprinted from R. Balieu, F. Chen, N. Kringos, Life cycle sustainability assessment of electrified road systems. Road Mater. Pavement Des. 20 (2019) S19–S33.

**3.** Finite element predictions [22] show that higher damage potentials can be induced in eRoad systems than that of a tRoad, especially the IPT-based solution. This entails that an increase in the frequency of long-term maintenance activities can be expected and, thus, the consequential effect to eRoads' life-cycle environmental performance should also be considered in the assessment. Therefore, the frequency of rehabilitation operations of the IPT-based eRoad, mainly referring to the replacement of the top asphalt layer, is assumed to be three times higher than that of a tRoad.

According to the calculations in Fig. 15.9, one can note that all of the three eRoad transport systems produce higher life-cycle  $CO_2$  emission than that of a tRoad. In that, the IPT-based eRoad is the most significant, which produces 166% more  $CO_2$  than that of a tRoad. Further comparisons at different life-cycle phases indicate that the major increase in the impacts come from the increased rehabilitation activities. These life cycle analysis results emphasize the importance of ensuring the good performance of the eRoad pavement system, as it would mitigate the long-term maintenance demand and thus the environmental impacts at large. Moreover, these results can help also decision-makers in industrial organizations and governmental agencies adopt the more sustainable and cost-effective practice for the eRoads, contributing to the improved sustainability of road transport sector through electrification.

## 15.5 Conclusions and recommendations

Many different disciplines are currently pursuing the development of knowledge and technology needed to enable some new functions directly from a practical road, which is also known as making our roads become smart or multifunctional. Given their dramatic potentials in enhancing the sustainability of our society as a whole, in the long-term run, the importance of these new developments would increase and thus technologies should be ready for allowing to do so. The dynamic application of the IPT technology in an eRoad, which aims for wireless charging of EV in-motion, could be a typical case of those new functional road solutions. In this study, the main concern of research lies in the important missing interfaces between disciplines for a sustainable implementation of the eRoad system into the practice from the road infrastructure's perspective. Emergent technical challenges induced by such an integration, mainly referring to the long-term service performance of both pavement structure and charging system, have been scrutinized. In that, knowledge and analysis tools from different engineering disciplines have been crossly applied for solving the practical issues of the integration. Some useful scientific findings as well as recommendations have been documented, which can serve as a basis of continuation in this new and interesting research domain.

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## WOODHEAD PUBLISHING SERIES IN CIVIL AND STRUCTURAL ENGINEERING

The pavement infrastructure plays an important role in the sustainability of urban systems. It is crucial covering an area of about 100,000 km2 whose construction, maintenance, and/or rehabilitation consume around 100 million tons of different types of asphalt binder annually. This high consumption of nonrenewable resources is also associated with the pollution of water reserves, reduced biodiversity at extraction sites, and high greenhouse gas emissions in a notorious unsustainable pattern. Unfortunately, over the last decades research concerning the pavement infrastructure has merited scarce attention by the scientific community. For example, a recent Scopus survey illustrates that publications listed under the subfield of pavements only represent 2% of the total field of civil engineering. This is a clear sign that pavements have not been one of the major research efforts of civil engineering researchers. The same survey also shows that the first publications related to sustainability in the field of civil engineering appeared 34 years but those under the subfield of pavements started to dedicate their research to sustainability issues around 16 years. While civil engineering publications related to sustainability are now being produced in an exponential manner in the subfield of pavements, they have not yet even reached 8%. This is why the book *Eco-efficient Pavement Construction Materials* will help to fulfill a major gap in the literature and also help to boost research in this important subfield, especially in the development of eco-efficient pavement materials to address resource efficiency, and to tackle climate change effects as well as to provide multifunctional features such as self-healing and electricity production.

This book is divided into four key parts. Part 1 focuses on cold and hot recycling of asphalt pavements including waste recycling. Part 2 reviews cool pavements and reflective coatings for high albedo pavements as well as the influence of the ageing process on the performance of cool coatings. Part 3 looks at self-healing materials and technologies. Part 4 brings the book to a close with several chapters on pavements with energy harvesting potential and pavements that deliver electric power to electric vehicles when driving.

The book will be a standard reference resource for civil engineers and all researchers working in the field of pavement materials.

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