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Study on self-healing microcapsule containing rejuvenator for asphalt

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HIGHLIGHTS

- Optimum preparation conditions were explored.
- Molecular structure and thermal stability were studied by FTIR and TG respectively.
- Healing performance under conditions of low-temperature and fatigue load were investigated.
- Four evaluation indexes for healing efficiency were put forward.

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ABSTRACT

Asphalt materials inevitably suffer crack problem caused by age, temperature and load, but recently self-healing microcapsules containing rejuvenator have offered a promising way to solve this problem. In this paper, the microcapsules were prepared by in-situ polymerization method and optimum preparation conditions were carefully explored, and then the morphology, particle size, coating rate, thermal stability and molecular structure of microcapsules were comprehensively investigated. Moreover, the healing ability of asphalt containing microcapsules under conditions of low-temperature and fatigue load was fully studied. Results indicated that the microcapsules could survive during the asphalt melting process and showed good healing performance under conditions of low-temperature and fatigue load, but the healing efficiency increased first and then decreased with the additive amount of microcapsules, and the optimal additive amount of microcapsules was 0.3–0.5 wt% of the asphalt.

1. Introduction

Asphalt materials have been widely used in civil and material engineering, but they inevitably suffer crack problem caused by age, temperature and load. Although asphalt materials have a certain self-healing ability due to their viscoelasticity, this ability will decline dramatically under low-temperature environment due to the increase of the stiffness, leading to lots of micro cracks. Moreover, these micro cracks will develop to macro cracks under cyclic load, which lastly lead to the deterioration of material and structure performance [1,2]. This situation will become more serious once the asphalt ages. According to the research [3], million tons of the asphalt go out of use every year due to asphalt aging in pavement engineering. Although many measures have been taken to solve the crack problem of the asphalt, the repair effect of cracks is less than satisfactory due to the difficulty to detect micro crack in the early stage. Not until the cracks develop to visible cracks can remedial measure be done, resulting in a certain impact on traffic and poor repairing effect. Therefore, a new, active and preventive maintenance technology which can cut off incipient crack autonomously and repair age damage for asphalt materials is an urgent need.

Recently, self-healing microcapsules containing rejuvenator have offered a promising way to solve above mentioned problem. Microcapsule technology can be traced back to the 1930s, and in the early days, microcapsule technology was mostly used for protecting and controlling sensitive core components [4–6]. Later on, the self-healing ability of microcapsule began to get attention, and was gradually used for repairing the crack of polymers [7–11]. In consideration of the similarity between polymer and asphalt, some researchers tried to adopt self-healing microcapsule technology to solve the crack problem of the asphalt [12–18].

The self-healing mechanism of microcapsules for the asphalt is that when the crack develops to meet the microcapsule the shell materials of the microcapsule will be broken as to release the core material (rejuvenators) which can restore the properties of aging asphalt to be contact with asphalt around, and lastly close the cracks or limit its growth. So far, capsules containing rejuvenators can be divided into two types. (1) Porous sand core impregnated with oil

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and coated with a hard shell of filler and epoxy, with a size above 100 μm [12,13]. (2) An oil droplet surrounded by a hard polymeric shell, with a size under 100 μm [14–18]. The latter one is known as microcapsules, which has a better performance and can improve the durability of the asphalt and be applied for smart pavement [15].

However, most of researches on self-healing microcapsules were focused on the healing mechanism of microcapsules, there were few reports about the effect of preparation condition on the properties of microcapsules and the healing performance of microcapsules under conditions of low-temperature and fatigue load which are corresponding to thermal crack and fatigue crack respectively. The aims of this article are to (1) comprehensively investigate the effect of preparation conditions on the properties of microcapsules; (2) analyze and evaluate the healing performance of microcapsules for the asphalt under conditions of low-temperature and fatigue load.

2. Experiment

2.1. Materials

Self-healing microcapsules were prepared by in-situ polymerization method [19]. Raw materials include water, urea, formaldehyde, asphalt rejuvenator, emulsifier and modifier. Asphalt rejuvenator was obtained by blending lightweight oil containing a high content of aromatics with a chemical compound containing polar epoxy group (see Table 1). The SK-70 base asphalt was used in this study and its properties were shown in Table 2. Besides, some auxiliaries were used to adjust the pH value of the system and eliminate the foam.

2.2. Test methods

a) Morphology and particle size

PH50-3A43L-PL optical-photography microscope and Image Analysis Software (Image Pro Plus) were used to observe the morphology and analyze the particle size of the microcapsules.

b) Coating rate

Coating rate which represents the synthetic efficiency can be expressed by Eq. (1):

$$CR = \frac{M_C}{M_T} \tag{1}$$

where CR is the Coating Rate of microcapsules; M_C is the mass of core-material which was successfully capsuled in the microcapsule; M_T is the total mass of capsule-core added in the test.

c) Thermal stability and molecular structure

TG (Thermogravimetric Analysis) and FTIR (Fourier Transform Infrared Spectroscopy) were utilized to determine thermal stability and molecular structure of microcapsules respectively.

Table 1
The physical properties and chemical components of the rejuvenator.

	Index	Rejuvenator
Physical properties	Flash point (°C)	>220
	Viscosity changing ratio after TFOT aging	1.6
	Weight loss after TFOT aging (%)	1.7
Chemical components	Saturates content (%)	14.3
	Aromatics content (%)	61.1
	Resins content (%)	15.8
	Asphaltenes content (%)	8.7

Table 2
Properties of SK-70 base asphalt.

Properties	Values	Specification limit
Penetration (25 °C, 100 g, 5 s) (0.1 mm)	67.3	60–80
Ductility (5 cm/min, 10 °C) (cm)	23.1	⩾20
Ductility (5 cm/min, 15 °C) (cm)	110	⩾100
Softening point (°C)	46.5	⩾45
Density (g/cm^3)	1.023	–

d) Healing performance

Most of studies [14–16] on the healing performance of microcapsule were more confined to evaluate the performance of microcapsule itself, but not the performance of asphalt containing microcapsule. The technology used in these studies, such as indentation techniques, may be not easy to realize in engineering. Therefore, we decided to modify normal test for the asphalt to evaluate the healing performance of microcapsules.

Ductility test according to ASTM D113-07 was used to investigate the healing performance of microcapsules under low-temperature condition. Test temperature was 5 °C and tensile speed was 1 cm/min. Before the test, the samples will have a pre-cut crack about 4 mm depth and then heal for 4 hours [20], as shown in Fig. 1. The low-temperature healing efficiency can be expressed by Eq. (2):

$$HE = \frac{L_{heal}}{L_{original}} \tag{2}$$

where HE is the healing efficiency, L_{heal} is the ductility of the sample with pre-cut crack after healing for 4 hours, and $L_{original}$ is the ductility of intact sample.

Asphalt fatigue test according to ASTM D7175-15 was adopted to investigate healing performance under fatigue-load condition. The test temperature was 20 °C, and stress control mode (0.3 MPa) was adopted and the loading frequency was 10 Hz. The loading process can divided into two stages. In the first load stage, the load will stop until the modulus decrease to 60% of the initial modulus. Then the asphalt had 0.5 hour healing time [21]. After the end of heal stage, second load stage begin, and the test finally end until the modulus decrease to about 60% of the initial modulus again. A typical test result is shown in Fig. 2. Based on the change of the modulus before and after the healing stage, we proposed two healing indexes: HI_1 and HI_2.

$$HI_1 = \frac{G^*_{heal}}{G^*_{initial}} \tag{3}$$

$$HI_2 = G^*_{heal} - G^*_{damage} \tag{4}$$

where the $G^*_{initial}$ is the initial modulus of the asphalt, G^*_{damage} is the modulus of the asphalt at the end of first load stage, and G^*_{heal} is the modulus of the asphalt after healing for 0.5 h.

Moreover, based on the change of strain energy during the two load stages, we put forward another healing index HI_3, which can be calculated according to Eqs. (5)–(8)

$$E_i = \sigma_i \cdot \varepsilon_i \tag{5}$$

$$E_{first} = \sum_1^{n_1} E_i \tag{6}$$

$$E_{second} = \sum_1^{n_2} E_i \tag{7}$$

$$HI_3 = \frac{E_{first}}{E_{second}} \tag{8}$$