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# Construction and Building Materials

journal homepage: [www.elsevier.com/locate/conbuildmat](http://www.elsevier.com/locate/conbuildmat)

## Rheological and self-healing properties of asphalt binder containing microcapsules

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### HIGHLIGHTS

- UF microcapsule and self-healing asphalt containing microcapsule are prepared successfully.
- Microcapsules increase viscosity, high temperature stability and durability of asphalt.
- Microcapsules decrease penetration, ductility, and cracking resistance of asphalt.
- Microcapsules improve obviously self-healing property of asphalt.

### ARTICLE INFO

#### Article history:

Received 25 December 2017

Received in revised form 19 July 2018

Accepted 23 July 2018

#### Keywords:

Road engineering

Asphalt

Microcapsule

Rheological property

Self-healing ability

### ABSTRACT

The objective of this investigation is to prepare the microcapsule and study the rheological and self-healing properties of asphalt containing microcapsules. Urea formaldehyde resin (UF) self-healing microcapsule was prepared by in-situ polymerization method and the monomer properties of microcapsule were characterized. Penetration, softening point, ductility, dynamic shear rheology (DSR), bending beam rheology (BBR) and self-healing tests were conducted to study the rheological property and self-healing ability of asphalt containing microcapsules. The results show that UF microcapsule material has been successfully synthesized, and the microcapsules can withstand the high temperature during the construction of asphalt pavement to a certain extent. Compared with the properties of matrix asphalt, the ductility of self-healing asphalt containing UF microcapsules decreases obviously, the softening point and rutting factor increase slightly, the durability and self-healing ability increase obviously, the low temperature crack resistance decreases slightly.

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### 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 being 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 to the microcapsules in the matrix material, microcapsules will rupture

and repairing agent will be released to repair micro-cracks. 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 lots of researches in this area [13]. In 2001, White et al. [14] prepared self-healing microcapsules with urea-formaldehyde resin (UF) 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 dicyclopentadiene as the core, and found that the microcapsules had good elastic deformation ability. In 2013, Su [16,17] prepared microcapsules with the repair agent as the capsule core and with methanol modified melamine resin as the shell, and studied the thermal stability

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and mechanical properties of microcapsules and the interfacial stability between microcapsules and asphalt. The results showed that the particle size of microcapsules was in the range of 2.0–100.5  $\mu\text{m}$ , the microcapsules could withstand temperatures up to 200  $^{\circ}\text{C}$ , and the interfacial stability between microcapsule monomer and asphalt matrix was improved. Sun et al. [18] prepared microcapsules with melamine resin as capsule shell and with rejuvenator as core in 2015, and found that the self-healing ability and fatigue life of asphalt had been improved by adding microcapsules. Chung [19] in 2015 prepared microcapsules with urea-formaldehyde resin as the wall and with dimethyl phthalate (DMP) as the core, observed the healing process of cracks in asphalt by scanning electron microscopy (SEM) and proved that the asphalt containing microcapsules had good mechanical properties. In 2016, Micaelo [20] prepared microcapsules with sunflower oil encapsulated in calcium-alginate as the core and with epoxy-cement composite as the shell, and the self-healing ability of asphalt mixtures containing the microcapsules was studied. The results showed that the natural self-healing ability of asphalt mixtures could be enhanced with encapsulated rejuvenators. In 2016, Tabakovic [21] explored the potential use of compartmented alginate fibres as a new method of incorporating rejuvenators into asphalt pavement mixtures, and found that alginate fibres could survive during the production process of asphalt mixtures and increase the strength of the asphalt mastic mixture by 36%, and alginate fibres encapsulating rejuvenator had the potential to heal asphalt mixtures. In 2017, Al-Mansoori [22] prepared asphalt mixture containing microcapsules with vegetable oil as the core material and studied on the self-healing ability of it by crack-healing tests, the test results showed that cracked asphalt mixture with microcapsules recovered 52.9% of initial strength at 20  $^{\circ}\text{C}$  versus 14.0% of asphalt mixture without capsules. In 2018, Al-Mansoori T [23] studied the self-healing ability of asphalt mastic by the action of calcium-alginate capsules containing sunflower oil. It was proven that healing levels in the asphalt mastic samples with capsules were greater than samples without capsules.

The preceding studies show that the use of microcapsules in asphalt materials is feasible and beneficial. After the addition of microcapsules, when micro-cracks happen next to the microcapsules, rejuvenator is released from ruptured microcapsules to the cracks to increase the rate of self-healing. The most current researches focused on the microcapsule itself, such as the preparation methods, core material, shell materials and property of microcapsule. The researches on microcapsule modified asphalt are limited and they mainly focus on the repair mechanism, penetration, softening point, ductility and fatigue life of asphalt. The dispersion of microcapsules in asphalt and the anti-aging property, high temperature shear resistance, low temperature cracking resistance and self-healing ability of microcapsule modified asphalt are rare.

The objective of this paper is to evaluate the effect of microcapsules on the property of asphalt. The self-healing microcapsules were prepared with UF as the shell and with the repair agent as the core by in-situ polymerization method and the properties of the microcapsules were characterized. Then, the microcapsule self-healing asphalt was prepared and the effect of microcapsule on the rheological property and healing ability of asphalt material were studied.

## 2. Materials

### 2.1. Microcapsules

The ZS-1 asphalt rejuvenator was selected as the core material of microcapsules. Urea-formaldehyde resin (UF) was selected as

the shell material. Self-healing microcapsules were prepared by in-situ polymerization method, and the preparation process is as follows.

#### (1) Synthesis of urea – Formaldehyde resin prepolymer

The mass ratio of urea to formaldehyde was selected as 1:2. The solution consisting of 15 g of urea and 40.5 g of formaldehyde solution were mixed and stirred in the beaker to make the urea be dissolved completely and the mass fraction of formaldehyde solution was 37%. Then, the NaOH solution was added to the beaker to adjust the pH of solution to 8.0–9.0. And the solution in the beaker was poured into the three-necked flask. The water bath temperature was adjusted to 70–80  $^{\circ}\text{C}$ , the solution was stirred for 1 h at 400–450 r/min, and finally clear and transparent prepolymer solution of shell material was obtained.

#### (2) Emulsification of core material

The specific method of emulsification was as follows: Firstly, added 200 ml deionized water and 2 g emulsifier into the beaker, then stirred with a glass rod evenly. Secondly, added 25 g ZS-1 asphalt rejuvenator into the beaker, adjusted the water bath temperature to 50  $^{\circ}\text{C}$  and stirred at 1000–2000 r/min for 20–30 min with electric stirrer to prepared oil-in-water emulsion solution. In addition, 1–2 drops of defoamer n-octanol was needed during the emulsification process.

#### (3) Preparation of self-healing microcapsules

The prepolymer solution prepared in step (1) was weighed and added into the beaker with a plastic dropper, and the solution was stirred at a rate of 800 r/min for 10 min. While the water bath temperature rose from ambient temperature to 50  $^{\circ}\text{C}$  slowly, catalyst  $\text{NH}_4\text{Cl}$  was added in batches to maintain the PH between 3.0 and 4.0, and deionized water was added into the solution to make sure that the solution was not very sticky. Then, curing agent resorcinol was added into the solution and the solution was stirred at 800 r/min for 1.5 h to make the microcapsules cured. After stirring, NaOH solution was added into the solution to adjust the pH to 7. Then the solution was cooled down to ambient temperature and stood still for 1 h. Finally, the core material in the upper part of the solution was adsorbed with a plastic dropper, the emulsion of the remained part was washed with deionized water and poured into a suction filter. After the filtration was completed, the product was baked in an oven at 60  $^{\circ}\text{C}$  for 3 h. Fig. 1 shows the prepared UF microcapsule sample.

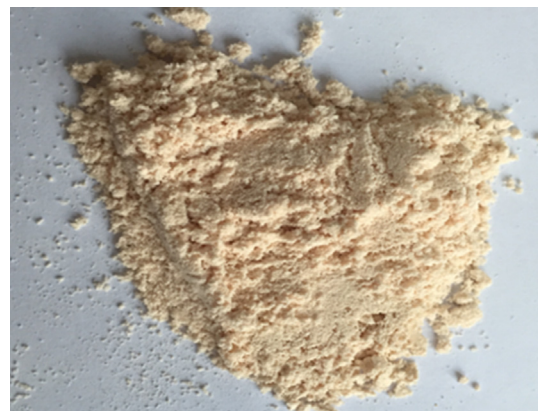


Fig. 1. UF microcapsule sample.

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