Investigation of self-healing properties of dense-graded asphalt mixture containing microcapsule

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Abstract: To study the self-healing performance of the asphalt mixture with microcapsules and its influencing factors, this paper uses high-pressure water penetration test to evaluate the self-healing performance of the microcapsule asphalt mixture, and studies the influence of microcapsule content, mixture properties (mixture type, maximum nominal particle size, porosity, asphalt type and amount) and use environment (self-healing temperature and time, moisture) on the self-healing performance. The results showed that as the amount of microcapsules increased, the self-healing effect increased first and then decreased. The optimal amount of microcapsules was determined to be 6%. In terms of the properties of the mixture, the self-healing effect of the suspension dense gradation is better than that of the skeleton dense type, and the matrix asphalt is better than the modified asphalt. The smaller the nominal maximum particle size and porosity are, the better the self-healing effect is. In external conditions, the temperature is conducive to the self-healing of the mixture within a certain range. The self-healing performance increases nonlinearly with the healing time, and the moisture is not conducive to the self-healing of the asphalt mixture cracks.

1. Introduction
After cracks appear in the asphalt mixture, the asphalt and the mixture have a certain self-healing ability at higher temperatures. Through the penetration and diffusion of asphalt on both sides of the crack, the crack can slowly heal automatically [1-2]. However, the healing process is extremely slow at ambient temperature, which not only requires a higher ambient temperature, but also requires no repeated action of traffic loads during the healing process [3]. The self-healing ability of asphalt alone is not enough to allow cracks to be automatically repaired in time. Microencap technology is a self-repair method that uses external substances as a replenishment method [4-6]. The mixture can improve the self-healing ability after being mixed with microcapsules, and automatically heal in time after the cracks are produced, inhibit the development of cracks, increase the service life of the pavement, and save the maintenance and repair costs of the pavement.

White [7] first proposed the concept of microcapsules in 2001, and proposed the principle of healing: external stress breaks the wall of the microcapsule, releasing the inner capsule core healing agent, and the healing agent reacts with external materials to make the damage self-healing. Garcia [8] proposed to apply microcapsules to asphalt pavements. Under the stress of the crack tip, the microcapsules are pierced to allow the internal healing agent to permeate out and stimulate the flow of aging asphalt materials to heal the cracks. ARRASA [9] used maltene as the core material and industrial cellulose (CE)
and polymer (C) as the wall to prepare microcapsules for the self-healing of asphalt mixture cracks. In this study, the stability and self-healing effect were studied using an optical microscope. Chai et al. [10] tested the self-made microcapsule asphalt mixture by splitting test and fatigue strength test, and found that adding microcapsules reduced the splitting strength by 11.3%, while the fatigue performance of the mixture increased by about 20%. Ji et al. [11] developed a test device for the self-healing performance of densely-mixed asphalt mixtures based on high-pressure water seepage, and evaluated the self-healing performance by using the ratio of the water seepage rate before and after the healing of the specimen. It is found that the evaluation results are consistent with the traditional intermittent fatigue test, split healing strength ratio and other test results. Jiang et al. [12] pointed out that the meso-distribution characteristics of asphalt mortar are closely related to the self-healing behavior of asphalt mixtures, and revealed the influence of gradation type, maximum nominal particle size, mineral clearance ratio (VMA) and asphalt saturation (VFA) on the micro-distribution characteristics of asphalt mortar. Huang et al. [13] found that the self-healing effect of asphalt mixture is related to healing time, asphalt dosage, strain size and porosity.

The factors that affect the self-healing performance of the microcapsule asphalt mixture are multifaceted, including the influence of the microcapsule content and the nature of the asphalt mixture, as well as the environmental conditions. Related research has rarely comprehensively investigated the comprehensive influence of capsules, mixtures and environmental conditions on the mixtures. To address the research gap, this paper uses high-pressure water penetration test to evaluate the self-healing performance of the microcapsule asphalt mixture, and studies the influence of microcapsule content, mixture properties (mixture type, maximum nominal particle size, porosity, asphalt type and amount) and use environment (self-healing temperature and time, moisture) on the self-healing performance. The research results will provide a powerful reference for the optimization design of microcapsule asphalt mixture.

2. Materials and methods

2.1 Raw materials

The microcapsules were prepared by adsorption method [14], as shown in Figure 1. The microcapsules were composed of capsule core, capsule wall and wrapping material. The capsule core was an asphalt regenerant, the capsule wall was porous expanded perlite, and the wrapping material was diluted with epoxy resin, epoxy curing agent, epoxy and liquid dispersant. The preparation steps are as follows: (1) Put the porous expanded perlite into the asphalt regenerant to fully soak, and then let it stand for 24 hours. (2) Add the wrapping material to the container in proportion, and stir with a glass rod for 2-3 min to a uniform state; (3) Put the porous expanded perlite after absorbing the regenerant into the wrapping material and stir for 2-3 min; (4) Add the solid dispersing material (cement) into the container, mix and uniformly form microcapsule particles; (5) Use a 0.3mm screen to disperse the microcapsule particles, spray water and cure to obtain the microcapsules. The mass fractions of wrapping materials are 40.5%, 13.1%, and 46.4% respectively.

Figure 1. Microcapsules
70#, 90# matrix asphalt and SBS modified asphalt were used in the test, and the performance is shown in Table 1. Limestone is also used in the asphalt mixture. The test shows that the crush value and the polish value are 15.8% and 43.5%, respectively.

### Table 1. Experimental Results of Asphalt

| Asphalt type | Penetration /0.1mm | Ductility /cm | Viscosity/(Pa.s) | Softening Point/℃ | Rotating film heating test |
|--------------|-------------------|--------------|------------------|-------------------|----------------------------|
| 70# matrix   | 67                | 107.9        | 259.5            | 46.0              | -0.50                      |
| 90# matrix   | 90                | 116.0        | 279              | 47.5              | -0.06                      |
| SBS modified | 65                | 32.8         | 2.6              | 87.5              | 0.23                       |

**Remarks:** The test temperature for the ductility of the matrix and SBS modified asphalt are 10°C and 5°C, respectively. The viscosity of the base asphalt is 60°C dynamic viscosity, and the viscosity of SBS modified asphalt is 135°C Brookfield viscosity.

2.2 mixtures

Five kinds of asphalt mixtures are used for testing and analysis, namely AC–10 (matrix 90#), AC-13 (matrix 90#), AC-13(SBS modified), and SMA-10 (SBS modified) and SMA-13(SBS modified). The technical indicators of the mixture are shown in Table 2.

### Table 2. Marshall Parameters of asphalt mixtures.

| Mixture   | Asphalt type | Optimal asphalt content (%) | Bulk relative density | Volume of air voids (%) | Voids in mineral aggregate (%) | Void filled in asphalt (%) | Marshall stability (kN) |
|-----------|--------------|-----------------------------|-----------------------|-------------------------|--------------------------------|---------------------------|------------------------|
| AC–10     | matrix 90#   | 5.4                         | 2.369                 | 3.3                     | 16.2                           | 79.6                      | 9.8                    |
| AC–13     | matrix 70#   | 4.8                         | 2.422                 | 3.5                     | 13.5                           | 73.7                      | 8.8                    |
|           | matrix 90#   | 4.9                         | 2.429                 | 3.5                     | 14.0                           | 74.8                      | 8.9                    |
|           | SBS modified | 5.2                         | 2.438                 | 3.6                     | 13.9                           | 74.1                      | 12.4                   |
| SMA–10    | SBS modified | 6.2                         | 2.316                 | 3.6                     | 18.1                           | 80.0                      | 14.9                   |
| SMA–13    | SBS modified | 6.0                         | 2.317                 | 3.8                     | 17.8                           | 78.7                      | 13.5                   |

3. Development of test method for assessing self-healing properties of dense-graded asphalt mixtures

3.1 Testing instrument and process

Water can seep through cracks in the asphalt mixture, and the seepage rate depends on the size of the cracks. The self-healing behaviour of the asphalt mixture changes the crack size, thereby influencing the seepage rate. Based on this principle, a device for testing the self-healing abilities of dense-graded asphalt mixtures based on high-pressure seepage was developed, as shown in Figure 2. In Figure 2, 1 indicates the flow meter, 2 the water injection pipe, 3 the pressure sensor, 4 the air pump, 5 the valve, 6 the water valve, 7 the intake pipe, 8 the fixed screw, 9 the outlet, 10 backplane, 11 the catchment funnel, 12 the display, 13 the dedicated tank, and 21 the rubber gasket. The inner diameter, outer diameter, and height of the dedicated tank are 102.5 mm, 104 mm, and 300 mm, respectively. The air pump provides a pressure of 0–1000 kPa.

The test process for assessing the self-healing properties of a dense-graded asphalt mixture is as follows:

- Step 1: Cylindrical specimens of 101.6 mm in diameter and 63.5 mm in height are prepared.
- Step 2: A microcrack is induced along the height direction via the splitting method within the temperature range 2–7 °C.
- Step 3: vaseline is spread on the side of the cylindrical specimen, which is then placed in the dedicated tank. The butter seal on the edge of the specimen is checked and scraped of excess butter;
Step 4: The tank is filled with 5000 ml water, and then the water valve is closed and the initial flow value $Q_1$ is recorded.

Step 5: The air pump is connected, the air valve is turned to inflate the tank, and the inlet valve is closed until the value on the display reaches 700 kPa;

Step 6: The screw on the bottom of the closed tank is opened and the test time $t$ is recorded synchronously. The final flow value $Q_2$ is recorded and used to calculate the seepage rate $v$ by Equation (1).

Step 7: The seepage rate $v_1$ is tested before healing. The tested specimen is placed in an oven for self-healing at 45 °C for 12 h. The seepage rate $v_2$ of the specimen is then tested after self-healing.

Step 8: The ratio of $v_e$ is calculated to evaluate the self-healing properties of the dense-graded asphalt mixture as shown in Equation (2). Larger values of $v_e$ correspond to better self-healing properties.

$$v = \frac{Q_1 - Q_2}{t}$$

$$v_e = \frac{v_1}{v_2}$$

where: $Q_1$ and $Q_2$ are the initial and final flow values, respectively (mL), $t$ is the test time (min), $v_1$ and $v_2$ are the seepage rates before and after healing, respectively (mL/min), and $v_e$ is the ratio of the seepage rate.

Figure 2. Structure diagram of water seepage tester

3.2 Validation of application of the seepage test to evaluate the self-healing properties of dense-graded asphalt mixtures

Different dosages of 2, 4, 6, 8, 10, and 12% microcapsules were incorporated into the AC–13 (matrix asphalt 90) mixture. The $v_e$ and intermittent fatigue lifetimes of these mixtures were tested, and the results are presented in Figure 3. The results show that the $v_e$ and intermittent fatigue life are gradually increased with increasing microcapsule content for microcapsule dosages of $\leq$6%, but the opposite trend is observed for microcapsule contents $>6$%. This indicates that the $v_e$ and intermittent fatigue life are consistent in evaluating the self-healing properties of the dense-graded asphalt mixtures, and proves that $v_e$ can be used for self-healing capability characterisation. The intermittent fatigue life is typically tested with expensive equipment such as universal testing machines (UTM), with the disadvantages of complex operation, long testing periods, and highly variable results. In comparison, the seepage-rate test is simple to operate, uses simple equipment, requires a short test period, and yields small variability.
4. Factors influencing self-healing properties of dense-graded asphalt mixtures containing microcapsules

4.1 Influence of microcapsule dosage
Microcapsules were incorporated into the AC–13 (matrix asphalt 90) mixture at dosages of 2, 4, 6, 8, 10, and 12. The results of $v_e$ were measured, and the results are shown in Figure 4. The results show that the $v_e$ results of AC–13 mixtures containing 2–10% microcapsules exceed that of the microcapsule-free asphalt mixture, indicating that the microcapsules promote self-healing for dosages of 2–10%. At microcapsule dosages of <6%, $v_e$ is increased rapidly with increasing microcapsule dosage and is decreased for dosages >6%, showing that the self-healing effect is maximised at the microcapsule dosage of 6%.

4.2 Influence of type and parameters of asphalt mixture
Tests were conducted to reveal the effects on the self-healing properties of asphalt mixtures containing microcapsule for different factors, such as the mixture type, nominal maximum aggregate size, volume of air voids, and content and type of asphalt.

4.2.1 Influence of mixture type
The results of $v_e$ for the four types of mixtures with different microcapsule dosages are shown in Figure 5. The results show that the $v_e$ for AC–10 with microcapsule dosages of 0 and 6% are 1.8 and 4.5 times higher, respectively, than those of SMA–10; whereas, the $v_e$ for AC–13 with microcapsule dosages of 0
and 6% are 1.8 and 4.0 times higher, respectively, than those of SMA–13. These findings demonstrate that the self-healing properties of AC are considerably better than those of SMA.

4.2.2 Influence of nominal maximum aggregate size
The results of $v_e$ for the four types of mixtures with different nominal maximum aggregate sizes are shown in Figure 6. The nominal maximum aggregate sizes of AC–10, AC–13, AC–16, and AC–20 are 9.5 mm, 13.2 mm, 16 mm, and 19 mm, respectively. The results show that the $v_e$ is decreased with increasing nominal maximum aggregate size, indicating that the aggregate size has a small effect on the self-healing capability.

4.2.3 Influence of air void volume
Specimens of AC–13 and SMA–13 with different volumes of air voids were produced experimentally. The results of $v_e$ for the different void volumes are shown in Figure 7. The results show that the $v_e$ is gradually decreased with increasing air void volume, indicating that the self-healing effects of the dense-graded asphalt mixtures are weakened with increases in the volume of air voids.

4.2.4 Influence of asphalt type
The results of $v_e$ of AC–13 with three different asphalt types were measured as shown in Figure 8. The result demonstrate that the grade of the matrix asphalt has only a slight effect on the self-healing capability, whereas the self-healing of SBS-modified asphalt was considerably worse than that of either grade of matrix asphalt.

4.2.5 Influence of asphalt content
The asphalt content influenced the self-healing effect. AC–13 with four asphalt contents of 4.5, 4.9, 5.3, and 5.7% were prepared, and the $v_e$ were measured as shown in Figure 9. As the asphalt content is increased from 4.5% to 5.7%, the $v_e$ of AC–13 with 6% microcapsules increases from 5.3 to 20.9. This shows that the self-healing effect is improved with increasing asphalt content.
4.3 Influence of environmental conditions

The environmental conditions include three aspects, namely healing temperature, curing period, and water. The microcapsule-containing AC–13 (matrix asphalt 90) with microcapsule dosages of 0 and 6% were selected for testing to investigate the influences of the above three aspects on the self-healing properties of the asphalt mixtures.

4.3.1 Influence of healing temperature

Asphalt is a temperature-sensitive material that softens with increasing temperature. Microcracks in asphalt can be repaired through the permeation and diffusion of the asphalt mixture for pavement of a temperature reaching a certain value. Therefore, temperature is a critical factor for the self-healing behaviour of asphalt. In order to investigate the influence of temperature on the self-healing properties, the specimens were cured for several hours at different temperatures of 15 °C, 30 °C, and 45 °C. The ve of the cured specimens were tested as shown in Figure 10. The results show that the ve of the asphalt mixture containing 6% microcapsules is increased with increasing temperature. This demonstrates that the self-healing capability is increased with temperature.

4.3.2 Influence of curing time

The process of self-healing for asphalt mixtures occurs gradually, and the curing period has an important influence on the self-healing properties. The specimens were cured for different periods, the seepage rates of which were measured as shown in Figure 11. The results show that the ve is gradually increased.
for increasing curing times. It is shown that the self-healing increases nonlinearly with increased curing time, with a sharp initial increase (e.g. curing times of 4–6 hours) before slowing after 12 hours.

4.3.3 Influence of water
In cracked asphalt pavement, environmental water penetrates the inner pavement through the cracks. Water negatively affects the self-healing process. In order to investigate the influence of water on the self-healing capability, the specimens were cured in a water bath at 45 °C. The results of $\nu_e$ for the soaked and dry specimens were tested as shown in Figure 12. The $\nu_e$ of the soaked and dry AC–13 specimens with 6% microcapsules are 2.9 and 22.0, respectively, indicating that the self-healing effect is decreased by 78.8% in the presence of water.

5. Conclusion
(1) As the content of microcapsules increases, the self-healing effect tends to increase first and then decrease. The self-healing performance of the asphalt mixture reaches the best when the content of microcapsules is 6%.

(2) The article reveals the influence law of mixture type, maximum nominal particle size, porosity, asphalt type and asphalt content on self-healing performance. The self-healing effect of the suspension dense gradation is better than that of the skeleton dense type, and the matrix asphalt is better than the modified asphalt. The smaller the nominal maximum particle size and porosity are, the better the self-healing effect is.

(3) The effects of healing temperature, healing time and moisture on self-healing performance are
revealed. The temperature is conducive to the self-healing of the mixture within a certain range. The self-healing performance increases nonlinearly with the healing time, and the moisture is not conducive to the self-healing of the asphalt mixture cracks.

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