Influence of microcapsules on rheological properties of asphalt mortar

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Abstract: In order to study the effect of microcapsules on the rheological properties of asphalt mortar, microcapsules were prepared by interfacial polymerization and mixed with 70# matrix asphalt and SBS modified asphalt in different proportions to prepare two kinds of microcapsule asphalt mortars. The rheological properties of asphalt mortar were measured by dynamic shear rheological experiment and curved beam rheological experiment. The test results show that the microcapsules prepared by the interfacial polymerization method have a smooth surface, a high coating rate, and an average particle size of about 14.3μm; in the high temperature rheological properties, the rutting factor of the two asphalt mortars showed varying degrees of reduction with the increase of the microcapsule content. When the content of microcapsules is greater than 0.6%, the high temperature rheological properties of the microcapsule asphalt mortar do not change significantly; in the low temperature rheological properties, the creep stiffness modulus $S$ gradually decreases with the increase of the microcapsule content, and the creep rate $m$ gradually increases with the increase of the microcapsule content.

1. Introduction
At present, asphalt surface is the main surface form of high-grade highways in my country. Compared with other forms of pavement, it has better driving comfort, shorter structural construction period and better efficiency. With the increase of the service time of the pavement structure, the asphalt mixture will produce micro-cracks under the action of natural factors and traffic loads. With the development of the micro-cracks, the mechanical properties of the asphalt mixture will be greatly reduced, which will eventually cause serious damage to the road surface[1]. As an important raw material of asphalt mixture, asphalt has certain self-healing characteristics[2,3]. But under natural conditions, the self-healing ability of asphalt itself cannot completely repair the micro-cracks caused by fatigue damage. At this stage, common ways to improve the self-healing ability of materials are: microwave heating[4], electromagnetic heating[5], hollow fibers[6], and microcapsules[7-10].

Asphalt mixture is the most commonly used material in road engineering. While improving its self-healing performance, the road performance of the mixture cannot be ignored and must meet the requirements of use. Feng et al. studied the road performance of asphalt mixtures with microcapsules...
through comparative experiments. The water stability and high temperature stability of asphalt mixtures with microcapsules decreased slightly, and the low temperature crack resistance decreased significantly[11]. Zhang Lei incorporated the microcapsules into the asphalt mixture. With the increase of the amount of microcapsules, the low temperature crack resistance of the asphalt mixture has been improved, while the high temperature stability has been reduced[12]. Asphalt is an important part of asphalt mixture and an important factor in the road performance of the mixture. Zhang et al. prepared a polyurea resin microcapsule and found that the durability of the self-healing asphalt containing polyurea resin microcapsules was significantly improved compared with the base asphalt, and the low-temperature crack resistance was slightly decreased[13]. Liu et al. added microcapsules to asphalt materials and concluded that microcapsules can improve the low-temperature performance of asphalt and improve the adhesion of cold mixes[14]. Li et al. found through experiments that the microcapsule SBS modified asphalt exhibits better viscoelasticity, high temperature stability and rutting resistance than pure SBS modified asphalt[15]. Lu Shijie and others carried out research on self-healing asphalt pavement, and conducted related road performance tests on microcapsule asphalt pavement. The results showed that with the incorporation of microcapsules, the high-temperature stability of asphalt was improved and the low-temperature performance decreased[16]. Guo Yandong proved through experiments that mixing graphene microcapsules of different sizes into asphalt will not affect the penetration of asphalt, but when smaller graphene microcapsules are mixed with asphalt samples, the high temperature resistance of asphalt will be improved.

At this stage, the preparation of asphalt self-repairing microcapsules mostly adopts in-situ polymerization[17-19], and scholars have studied more on the promoting effect of microcapsules on the healing ability of asphalt materials. There is no unified conclusion about the influence of microcapsules on the road performance of asphalt mortar and asphalt mixture. In this paper, self-healing microcapsules (Self-healing microcapsule, SHM) with asphalt regenerant as the core material were prepared by the interfacial polymerization method, and the self-healing microcapsule (SHM) was mixed into the asphalt to prepare the self-healing asphalt mortar. Dynamic Shear Rheological test (DSR) and Bending Beam Rheological Test (BBR) were used to test the rheological properties of self-healing asphalt mortar. From the perspective of rheology, the effect of microcapsules on the rheological properties of asphalt was studied.

2. Experiment materials
Using 70# base asphalt and SBSI-C modified asphalt, its technical indicators are shown in Table 1. The ore powder is milled from limestone, and its technical indicators are shown in Table 2.

| Table 1. Technical indicators of asphalt |
|-----------------------------------------|
| Asphalt | Penetration at 25°C / (0.1mm) | Ductility /cm | Softening Point /°C | Density at 15°C / (g/cm³) | Penetration Index | Flash point /°C | Dynamic viscosity / (Pa·s) |
|---------|---------------------------------|---------------|--------------------|-----------------------------|------------------|----------------|----------------------------|
| 70#     | 68                              | 38            | 47.3               | 1.035                       | -0.315           | 290           | 210                        |
| SBSI-C  | 69                              | 35            | 72                 | 1.022                       | 0.112            | 290           | 1.85                       |

Note: The ductility test temperature of base asphalt is 10°C, the ductility test temperature of SBSI-C modified asphalt is 5°C; the dynamic viscosity test temperature of matrix asphalt is 60°C, and the kinematic viscosity test temperature of SBSI-C modified asphalt is 135°C.

| Table 2. Technical indicators of mineral powder |
|-----------------------------------------------|
| Pilot projects | Apparent density / (g/cm³) | Granularity <0.6mm | Granularity <0.15mm | Granularity <0.075mm | Hydrophilic coefficient |
|----------------|----------------------------|-------------------|-------------------|-------------------|---------------------|
| Test results   | 2.673                      | 100               | 91.2              | 76.4              | 0.71                |
Microcapsules (self-healing micro capsules, SHM) are prepared by interfacial polymerization. The preparation process is shown in Figure 1. First, Prepare a PVA emulsifier solution with a concentration of 0.4% as the water phase; Take a certain quality of oil-soluble reactive monomer isophorone diisocyanate (IPDI) and asphalt regenerant, and mix it as the oil phase; Mix the oil phase and the water phase is sheared at a high speed to form an oil-in-water emulsion; Stirring is carried out at a speed of 200r/min under the condition of a water bath at 70°C, and the catalyst dibutyltin dilaurate (DBTDL) is added dropwise at the same time, reacting for 3h, the SHM suspension is obtained; The suspension is centrifuged, filtered with suction, washed, and dried by oven blast to obtain powdery microcapsule SHM.

3. Experiment methods

3.1. Preparation method of microcapsule asphalt glue

SHM asphalt mortar is prepared by high-speed shearing method. The asphalt is heated to above 135°C, and then SHM and mineral powder are added in a certain proportion. Under heating conditions, a mechanical mixer is used to stir for 30 minutes at a speed of 1000r/min to obtain SHM asphalt. Among them, the powder-to-binder ratio is 1.0, and the powder-to-binder ratio is the ratio of the powder with a particle size of less than 0.075 mm to the content of the asphalt, and the mixed SHM is also a part of the powder. SHM is blended in the form of equal volume of replacement mineral powder, the mass of SHM blending is calculated according to formula (1), and the mass of replaced mineral powder is calculated according to formula (2).

\[ m_m = n \times m_i \]  
\[ m_m = \frac{n \times m_s \times \rho_m}{\rho_s} \]  

Formula: 
- \( m_m \) — the mass of microcapsules incorporated, g; 
- \( n \) — the percentage of microcapsules in microcapsule asphalt mortar, that is, the amount of microcapsules, %; 
- \( m_i \) — the total mass of microcapsule asphalt mortar, g; 
- \( m_s \) — the quantity of the mineral powder replaced by the microcapsule, g; 
- \( \rho_m \) — the density of mineral powder, g/cm³; 
- \( \rho_s \) — the density of the microcapsules, g/cm³.

The test is divided into two groups, A and B. Group A is SHM base asphalt mortar, with a powder-to-binder ratio of 1.0, and the SHM content is 0%, 0.2%, 0.4%, 0.6%, 0.8%; Group B is SHM modified asphalt mortar, with a powder-to-binder ratio of 1.0, and the amount of SHM is also set to 5 kinds of...
dosage, and the amount of SHM is 0%, 0.2%, 0.4%, 0.6%, 0.8%.

3.2. High temperature rheological performance test of SHM asphalt mortar
The Kinexus dynamic shear rheometer was used for the dynamic shear test, using 25mm parallel plates, the plate spacing was 1mm, and the frequency was 10rad/s. The test temperature of SHM base asphalt mortar is 70°C, 76°C, 82°C, 88°C; the test temperature of SHM modified asphalt mortar is 88°C, 94°C, 100°C, 106°C. The complex modulus $G^*$ and phase angle $\delta$ of asphalt are measured by dynamic shear rheology test, and the rutting factor $G^*/\sin\delta$ is calculated as the evaluation index of high temperature performance.

3.3. Low-temperature rheological performance test of SHM asphalt mortar
The CANNON-thermoelectric bending beam rheometer was used to measure the stiffness modulus ($S$) and creep rate ($m$) of SHM asphalt mortar under different SHM content to evaluate its low temperature performance. The trabecular size is 125mm×6.25mm×12.5mm. The test temperature is -12°C, -18°C, -24°C, and the temperature control error does not exceed ±0.1°C, and the loading time is 60s.

4. Results & Discussion

4.1. Technical properties of microcapsules
From the microcapsule images and particle size distribution diagrams in Figures 2 and 3, it can be seen that the prepared SHM has a smooth surface, a high coating rate, and a relatively uniform particle size distribution, with an average particle size of approximately 14.3 μm.
4.2. High temperature rheological properties of microcapsule asphalt mortar

In order to explore the modification effect of SHM on the high-temperature rheological properties of asphalt mortar, this paper uses DSR to determine the complex modulus $G^*$ and phase angle $\delta$ of SHM asphalt mortar, and calculates the rutting factor $G*/\sin\delta$ to evaluate the SHM asphalt mortar high temperature performance. In the test, two asphalts, 70# base asphalt and SBS modified asphalt, were used to prepare SHM asphalt mortar. The powder-binder ratio was 1.0, and the SHM content was 0%, 0.2%, 0.4%, 0.6% and 0.8%. The test results are shown in Table 3.

Rutting factor is used to characterize the resistance to permanent deformation of asphalt materials at high temperatures. The larger the value, the stronger the rutting resistance of the asphalt and the better the high temperature performance of the asphalt. It can be seen from Figure 4: (1) At the same amount of SHM, the rutting factor $G*/\sin\delta$ of SHM asphalt mortar gradually decreases with the increase of temperature, indicating that temperature is an important factor affecting the high-temperature stability of asphalt mortar; (2) At the same temperature, the rutting factor $G*/\sin\delta$ of SHM base asphalt mortar shows a decreasing trend with the increase of SHM content. Compared with the base asphalt mortar without SHM, the rutting factor $G*/\sin\delta$ under the condition of 0.8% SHM at 70°C, 76°C, 82°C, and 88°C decreased respectively by 24.0%, 27.5%, 31.4%, 26.9%; (3) At the same temperature, the rutting factor $G*/\sin\delta$ of SHM modified asphalt mortar shows a decreasing trend with the increase of SHM content. When the SHM content exceeds 0.6%, the rutting factor $G*/\sin\delta$ of the asphalt mortar no longer changes significantly. Compared with the modified asphalt mortar without SHM, the rutting factor $G*/\sin\delta$ under the condition of 0.8% SHM at 88°C, 94°C, 100°C and 106°C decreased respectively by 30.5%, 28.7%, 28.2%, 28.0%; (4) Comparing SHM modified asphalt mortar and SHM base asphalt mortar, the rutting factor $G*/\sin\delta$ of SHM modified asphalt mortar is higher at 88°C and different SHM content than SHM base asphalt mortar.

| Microcapsule content % | $G*(sin\delta)$ of SHM base asphalt mortar /kPa | $G*(sin\delta)$ of SHM modified asphalt mortar /kPa |
|------------------------|-----------------------------------------------|-------------------------------------------------|
|                        | $70^\circ C$ | $76^\circ C$ | $82^\circ C$ | $88^\circ C$ | $88^\circ C$ | $94^\circ C$ | $100^\circ C$ | $106^\circ C$ |
| 0                      | 5.0833      | 2.6131      | 1.4461      | 0.7525      | 5.3543      | 3.4298      | 2.1792      | 1.3500      |
| 0.2                    | 4.8713      | 2.4015      | 1.2520      | 0.6960      | 4.4761      | 2.7599      | 1.7801      | 1.1140      |
| 0.4                    | 4.7987      | 2.3003      | 1.2225      | 0.6686      | 4.0107      | 2.6111      | 1.6704      | 1.0382      |
| 0.6                    | 4.2125      | 2.0636      | 1.0672      | 0.5955      | 3.7160      | 2.4207      | 1.5517      | 0.9644      |
| 0.8                    | 3.8655      | 1.8953      | 0.9914      | 0.5501      | 3.7221      | 2.4451      | 1.5652      | 0.9715      |

The test results show that: as the temperature of the asphalt mortar increases, the rutting factor of the asphalt mortar decreases, and the anti-deformation ability of the asphalt mortar weakens; the modified asphalt mortar has better anti-deformation ability than the base asphalt mortar; With the increase of SHM content, the rutting factor of asphalt mortar decreases, that is, the increase of SHM content will weaken the high temperature permanent anti-deformation ability of asphalt mortar. The reason is analyzed: the content of microcapsules increases, the mineral powder content decreases, and the mineral powder in the asphalt because of its large specific surface area, it can be combined with asphalt to form structural asphalt, so that the formed structural asphalt is reduced, and the bonding effect of structural asphalt and free asphalt is weakened. At the same time, the mixed SHM is a spherical oily substance with a smooth surface, which is different from the morphology of the mineral powder. The SHM has a smooth surface and has a weak adsorption effect on asphalt. In addition, the SHM oily substance will act in the asphalt mortar in terms of lubrication, it weakens the anti-deformation ability of the asphalt mortar, which is manifested by the decrease of high temperature stability.
4.3. Low-temperature rheological properties of microcapsule asphalt mortar

In the bending beam rheological test, the creep stiffness modulus $S$ and the creep rate $m$ are used to characterize the low-temperature crack resistance of the mixture. Creep stiffness modulus $S$ is a characterization of asphalt resistance to constant load, reflecting the ability of asphalt to resist permanent deformation; creep rate $m$ is a characterization of asphalt stiffness change after load, it reflects the time sensitivity of asphalt stiffness and stress relaxation performance. The experiment uses 70# base asphalt and SBS modified asphalt to prepare SHM asphalt mortar, the powder-binder ratio is 1.0, and the SHM content is 0%, 0.2%, 0.4%, 0.6% and 0.8%. The experimental results are shown in Table 4. As shown in 5.

| Microcapsule content % | Creep rate $m$ | Creep stiffness modulus $S$/MPa |
|-----------------------|---------------|-------------------------------|
|                       | -24°C | -18°C | -12°C | -24°C | -18°C | -12°C | -24°C | -18°C | -12°C |
| 0                     | 0.089 | 0.244 | 0.476 | 1760  | 1139  | 476   |       |       |       |
| 0.2                   | 0.153 | 0.270 | 0.479 | 1670  | 1130  | 455   |       |       |       |
| 0.4                   | 0.155 | 0.282 | 0.494 | 1480  | 974   | 449   |       |       |       |
| 0.6                   | 0.222 | 0.348 | 0.521 | 885   | 353   | 154   |       |       |       |
| 0.8                   | 0.226 | 0.349 | 0.530 | 527   | 275   | 85.7  |       |       |       |

Table 5. Results of BBR test of SHM modified asphalt mortar

| Microcapsule content % | Creep rate $m$ | Creep stiffness modulus $S$/MPa |
|-----------------------|---------------|-------------------------------|
|                       | -24°C | -18°C | -12°C | -24°C | -18°C | -12°C |
| 0                     | 0.191 | 0.316 | 0.478 | 871   | 375   | 190   |
| 0.2                   | 0.201 | 0.327 | 0.481 | 659   | 320   | 139   |
| 0.4                   | 0.207 | 0.330 | 0.489 | 550   | 305   | 120   |
| 0.6                   | 0.216 | 0.358 | 0.499 | 382   | 170   | 77.5  |
| 0.8                   | 0.248 | 0.376 | 0.546 | 359   | 113   | 61.3  |

It can be seen from Figures 5 and 6 that: (1) At the same amount of SHM, as the temperature decreases, the creep rate $m$ of SHM asphalt mortar gradually decreases, and the creep stiffness modulus $S$ gradually increases; (2) At the same temperature, with the increase of SHM content, the creep rate $m$ of SHM base asphalt mortar shows an increasing trend, and the creep stiffness modulus $S$ shows a decreasing trend. At -12°C, -18°C, -24°C, compared with 0%SHM self-healing asphalt mortar, the $m$ of 0.8%SHM self-healing asphalt mortar increased respectively by 11.3% and 43.0%, 153.9%; $S$ is reduced by 82.0%, 75.9%, 70.1% respectively when compared with 0% content; (3) At the same temperature, with the increase of SHM content, the creep rate $m$ of SHM modified asphalt mortar shows an increasing trend, and the creep stiffness modulus $S$ shows a decreasing trend. Under the conditions of -12°C, -18°C
and -24°C, \( m \) increased by 14.2%, 19.0%, and 29.8% at 0.8% SHM dosage compared with 0% dosage; at -12°C, -18°C, -24°C, \( S \) with 0.8% SHM dosage was reduced by 67.7%, 69.9%, and 58.8% respectively compared with 0% dosage; (4) Comparing SHM modified asphalt mortar and SHM base asphalt mortar, the creep rate \( m \) of SHM modified asphalt mortar is basically higher than that of SHM base asphalt mortar. The creep stiffness modulus \( S \) of SHM modified asphalt mortar are lower than SHM base asphalt mortar.

The test results show that the temperature of the asphalt mortar decreases, the creep rate \( m \) of the asphalt mortar decreases, the creep stiffness modulus \( S \) increases, and the low-temperature crack resistance of the asphalt mortar weakens; the modified asphalt mortar has better low-temperature crack resistance than the base asphalt mortar; under the same temperature conditions, the creep rate \( m \) of the asphalt mortar containing SHM is higher than that of the asphalt mortar without SHM, and with the increase of the amount of SHM, the creep performance of the SHM asphalt mortar gradually increases, indicating that the regenerant released by SHM supplements the light components in the asphalt mortar and enhances the deformation recovery ability of the asphalt mortar. It proves that the SHM prepared in this paper has a regeneration effect on asphalt and improves the low temperature performance of the asphalt mortar.

![Figure 5](image5.png)

**Figure 5.** The influence of SHM on the low-temperature rheological properties of 70# matrix asphalt mortar

![Figure 6](image6.png)

**Figure 6.** Influence of SHM on low-temperature rheological properties of SBS modified asphalt mortar

5. Conclusions

In this paper, road self-healing microcapsule SHM was prepared by interfacial polymerization, and the
high-temperature rheological properties and low-temperature rheological properties of two asphalt mortars were studied through DSR and BBR, and the following conclusions were summarized:

1) The road self-healing microcapsule SHM with the core as an asphalt regenerant was prepared by the interfacial polymerization method. The prepared SHM has a smooth surface, a high coverage rate, and an average particle size of approximately 14.3 μm.

2) As the temperature of SHM base asphalt mortar and SHM modified asphalt mortar increases, the resistance to high temperature deformation of asphalt mortar decreases; under the same temperature condition, with the increase of SHM content, the rutting factor decreases by 24%~31%, the high temperature rheology of asphalt mortar gradually deteriorates.

3) As the temperature decreases, the low-temperature rheological properties of the SHM base asphalt mortar and SHM modified asphalt mortar decrease; under the same temperature conditions, with the increase of the SHM content, the creep stiffness modulus $S$ gradually decreases and the creep rate $m$ gradually increases, indicating that the incorporation of SHM improves the low temperature performance of the asphalt mortar.

Acknowledgments
This paper was supported by the Science and Technology Plan Project (No. 2020H08) of Zhejiang Provincial Highway and Transportation Management Center.

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