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Study on flexural properties of coal gangue powder asphalt mixture under freeze-thaw cycles

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Abstract

In order to study the influence of freeze-thaw cycle on the flexural performance of asphalt pavement, semi-circular flexural test (SCB) is carried out. Coal gangue powder (accounting for 0%, 25%, 50%, 75% and 100% by weight of ore powder) is used to replace ore powder to improve the freeze-thaw resistance of asphalt. According to the test data, the load-displacement curve is drawn, the ultimate tensile stress ($\sigma_t$) is studied, and the variation law of different coal gangue powder replacement rates under different freeze-thaw cycles (0, 2, 4, and 6 times) is analyzed by ultimate tensile stress. The test results show that the best replacement rate of coal gangue powder is 50%, and $\sigma_t$ decreases with the increase of freeze-thaw cycles, so adding coal gangue powder can reduce the influence of freeze-thaw cycles on the bending performance of asphalt pavement. By analyzing the load-displacement curve, energy indexes such as $W_D$ (total work from initial failure to complete failure), $W_U$ (energy dissipation from initial failure to complete failure), $W_S$ (work from initial failure to specimen failure) and $\eta$ are obtained. In the freeze-thaw cycle, with the increase of cycle times, the energy consumption of $W_D$, $W_U$ and $W_S$ decreases, which aggravates the damage of asphalt mixture and reduces the flexural capacity of specimens. Among them, six freeze-thaw cycles have the lowest flexural capacity of specimens, indicating that freeze-thaw damage directly affects the flexural performance of asphalt mixture.

1. Introduction

Due to the low temperature and frequent snowfall in northern China in winter, the internal structure of asphalt concrete has been destroyed after many times of freeze-thaw. A large amount of snow and ice lead to the decrease in the anti-sliding coefficient of the road, which greatly reduces the safety of road traffic [1, 2]. The freeze-thaw cycles make water more likely to invade into the asphalt mixture, which can aggravate the internal erosion of water on the asphalt mixture [3, 4]. Wang et al [5] concluded that the number of freeze-thaw cycles have a significant effect on fracture toughness of the asphalt mixture by using J integral. Nang et al [6] analyzed the failure mechanism of asphalt mixture under different freeze-thaw cycles and concluded that the damage of asphalt mixture is more obvious when loading and freeze-thaw were acting at the same time. Due to the influence of external environmental factors, moisture in the asphalt mixture usually exists in liquid or solid state. When the temperature drops sharply and reaches the freezing point, the moisture begins to transform from liquid to solid, and the volume becomes larger. Such frost heaving stress not only expands the volume and number of voids [7, 8], but also causes irreversible damage to asphalt mixture [9]. When the temperature rises, the moisture will be converted into liquid state. As the frost heaving stress disappears, the mixture shrinks, and the water will stay in the expanded voids. With the increase of freeze-thaw cycles, the number of voids is more and more. In the end, under the action of load and other factors, voids are connected to form potholes and other diseases, which reduces the service life of the road.

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In winter snow and ice weather, water in freeze-thaw cycles can cause changes in molecular weight of hydrocarbons in bitumen components. When the temperature changes, large temperature stress will be generated, which will destroy the bond performance of asphalt. When the temperature decreases, the water in the pores of asphalt mixture freezes into ice, which reduces the bond force of asphalt. When the frost heaving force is greater than the bond force, the cracks will be accelerated. When temperatures rise, ice in the pores of the asphalt mixture melts into water, which destroys its toughness, shortening the service life of asphalt pavement.

In response to such problems, scholars have proposed to add fibers into the asphalt mixture to play the role of ‘reinforcement’ [10–12]. Chen et al [13] added basalt fiber to asphalt mixture, and obtained that the splitting strength and stiffness modulus of asphalt mixture with basalt fiber are higher than that of ordinary asphalt concrete before and after freeze-thaw cycles. And through the establishment of a damage evolution model, it was found that the internal freeze-thaw damage of asphalt mixture with fiber is uniform after freeze-thaw. Wu et al [14] mixed basalt fiber, polyester fiber, and lignin fiber into asphalt mixture, and found that the splitting strength of asphalt mixture with basalt fiber is slightly better than polyester fiber asphalt mixture, but the splitting strength of both is much greater than that of lignin fiber asphalt mixture through frozen experiment. However, from the economic point of view, polyester fiber is preferred. In recent years, with the continuous excavation of materials and the continuous enrichment of test content, some scholars have also tried to use coal gangue to improve the performance of asphalt mixture [15–18]. Coal gangue is a secondary resource in the process of coal mining, and its main components are SiO$_2$ and Al$_2$O$_3$. It was also found that coal gangue is ground into coal gangue powder by high-temperature calcination, and a large number of metal cations could react positively with the polar functional groups in the asphalt after being activated at high temperature [19]. At present, some research results have been achieved by using coal gangue powder as filler. The test shows that coal gangue powder has certain advantages in improving the low temperature crack resistance of asphalt mixture, but further research is needed to improve other properties of asphalt mixture. A large amount of coal gangue is produced in industrial production, piling up mountains and occupying a large area of land. It is easy to collapse in rainy season, blocking rivers and causing disasters. Under the action of rain, these harmful elements will dissolve and enter the soil, destroying soil nutrients and affecting the growth of vegetation. Spontaneous combustion of coal gangue is easy to catch fire, and produces a lot of harmful gases and explosive dust, which pollutes air and water sources and endangers human health [20]. Coal gangue powder can coexist stably with asphalt molecules, which basically eliminates the weakening of mechanical properties and environmental pollution caused by the escape of coal gangue powder asphalt mixture in actual use. Adding coal gangue powder to asphalt not only protects the environment, but also optimizes resources.

Under the condition of and humid environment, the failure process of asphalt mixture is not clear. Therefore, it is necessary to understand the destruction mechanism of asphalt mixture in freeze-thaw environment and propose corresponding effective measures. In the existing articles on the mechanism of freeze-thaw, few scholars use the energy method to analyze the energy dissipation of freeze-thaw on the ascending and descending sections of the loading curve of the specimens (figure 4). In this paper, base on the previous research results, from the point of view of materials, coal gangue powder is used instead of mineral powder to change the filling method. The ultimate tensile stress and energy index are used to explore the improvement measures and damage mechanism of asphalt mixture under freeze-thaw coupling environment. The load-displacement curve is constructed by energy index, and the improvement measures and failure mechanism of asphalt mixture under freeze-thaw cycle are obtained by analyzing the energy dissipation, and the influence of freeze-thaw cycle on asphalt bending performance is explored. Scanning electron microscope (SEM) is used to explore the micro-mechanism of coal gangue powder asphalt mixture. Through the analysis of the bending performance of asphalt mixture under freezing and thawing cycle, it is hoped that it can provide some guidance for the selection of asphalt mixture in freezing and thawing areas in winter.

2. Materials

The heavy oil on the 70th road asphalt was used, and the technical indexes are shown in table 1. Basalt, produced in Huainan, China was used as an aggregate. Its basic properties are listed in table 2. Mineral powder and coal gangue powder were used as fillers. The main ingredients of mineral powder and coal gangue powder are presented in table 3. The replacement rates that coal gangue powder replaces mineral powder by weight were 0%, 25%, 50%, 75% and 100%. At the same time, polyester fiber accounting for 0.4% of the asphalt mixture mass was incorporated into the mixture, and its properties are shown in table 4. All indexes of the above materials meet the specification requirements. The design gradation of the aggregate meets the gradation requirements of the AC-13 dense-graded asphalt mixture according to the Chinese Technical Specifications for Construction of Highway Asphalt Pavements [21], as shown in figure 1.
3. Specimen preparation and evaluation index

3.1. Specimen preparation

The semi-circle specimens were cut from the standard Marshall specimens by using a high-precision cutting machine in this test. As shown in figures 2 and 3, the standard Marshall specimens were cut into two semicircles along the diameter, and then each semicircle was divided into two parts along the height. The diameter of each semicircle is 101.6 mm and the height is 31.75 mm. According to the content of gangue powder (0%, 25%, 50%, 75% and 100%), the semi-circular specimens were divided into five groups, then the specimens were divided into 4 groups according to the number of freeze-thaw cycles (0, 2, 4, 6). A freeze-thaw cycle was defined as: the freezing temperature was $-18 \, ^\circ C \pm 2 \, ^\circ C$ for 16h ± 1h. After freezing, the specimens were quickly placed in a constant temperature water bath with a temperature of $60 \, ^\circ C \pm 0.5 \, ^\circ C$ for 24h ± 1h [22]. Semi-circular

| Table 1. Major Technical Indexes Of Asphalt. |
|---------------------------------------------|
| Indicator                                     | No.70 (A) quality indicators | Measured values |
| Penetration (25 °C 100g 5s)                  | 60 ~80                        | 65               |
| Density (15 °C) g cm$^{-3}$                  | 1.035                         |
| Penetration Index PI                         | $-1.5 \pm 1.0$                |
| Ductility (10 °C 5 cm min$^{-1}$) /cm ≥      | 20                            |
| Ductility (15 °C 5 cm min$^{-1}$) /cm ≥      | 100                           |
| Softening Point (°C) ≥                       | 46                            |
| Solubility (%)                               | 99.5                          |
| Flash Point (°C)                             | 260                           |
| Water absorption rate (%)                    | 0.3                           |
| Polishing value                             | 48.1                          |

| Table 2. The main technical indicators of basalt. |
|-----------------------------------------------|
| Apparent density (g cm$^{-3}$)                | 2.910                         |
| Crushed value (%)                            | 10.8                          |
| Los Angeles wear loss (%)                    | 15.7                          |
| Adhesion of Asphalt (level)                  | 5                             |
| Water absorption rate (%)                    | 0.3                           |
| Polishing value                              | 48.1                          |

| Table 3. Main ingredients of mineral powder and coal gangue powder. |
|---------------------------------------------------------------|
| Ingredient                     | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | MgO     | CaO     | K$_2$O | Na$_2$O | H$_2$O | Others |
| Mineral powder                | 23.6    | 11.8        | 0.3         | 2.3     | 50.23   | 0.5    | 0.01    | 1.5    | 9.76   |
| Coal gangue powder            | 50.42   | 46.11       | 0.56        | 0.11    | 0.29    | —      | —       | —      | 2.51   |

| Table 4. Main indicators of polyester fiber. |
|---------------------------------------------|
| Project                                    | Performance | Pavement requirements |
| Length (mm)                                | 12 ± 2      | ≥3                     |
| Diameter (μm)                              | 20 ± 2.5    | 20 ± 5                |
| Strength (MPa)                             | 500 ~680    | ≥500                  |
| Elongation at break (%)                    | 15 ~35      | ≥15                   |
| Young modulus (GPa)                        | >13.5       | —                     |
| Proportion                                 | 1.36        | 1.36 ± 0.05           |
| Melting point (°C)                         | 259         | ≥258                  |

3. Specimen preparation and evaluation index
bending test (SCB) was carried out by universal testing machine. A semi-circular specimen of appropriate size was placed at a certain distance from the fulcrum. The load was applied along the diameter direction at the midpoint of the upper surface of the specimen, and the load was controlled by the opening displacement of the pre-cut slit. When the test load reaches the peak value and drops below 0.1kN, the test terminates. Load and deformation in the loading process were recorded and load-displacement curves were drawn. SCB test was based on energy judgment and ultimate tensile stress was taken as strength index. At the same time, the total work from initial failure to complete failure, energy dissipation from initial failure to complete failure, work from initial failure to specimen failure and $h$ were obtained by using loading data as energy indexes, and the semi-circular bending test (SCB) was used to characterize the flexural performance of asphalt mixture.

**3.2. Evaluation index**

It can be seen from figure 4 that load-displacement curve is divided into zones. $F_{\text{max}}$ is the work performed up to the maximum load. $\Delta F_{\text{max}}$ is the displacement at the maximum load. $\Delta R$ is the displacement corresponding to the load down to 0.1kN. $W_U$ and $W_S$ are the areas enclosed by the ascending and descending sections of the loading curve respectively, which are used to evaluate the damage mechanism of freeze-thaw cycles [23, 24].

![Image](AC-13 Aggregate gradation curve.)

**Figure 1.** AC-13 Aggregate gradation curve.

![Image](Loading model diagram(S/D = 0.8))

**Figure 2.** Loading model diagram ($S/D = 0.8$).
Asphalt mixture is a kind of viscoelastic material. In the low temperature crack resistance test, when the temperature reaches a certain value, the mixture is brittle failure. However, during the freeze-thaw test, with the increase of the number of freeze-thaw cycles, the energy dissipation in the descending section of the loading curve is rarely studied. Current research shows that asphalt mixture has certain self-healing performance under the action of temperature, load and other factors. By analyzing the energy dissipation in the descending section of the loading curve, it is possible to fully understand the life cycle of asphalt mixture under bitter cycles of freeze-thaw.

The tensile stress ($\sigma_t$) is calculated by:

$$\sigma_t = \frac{4.888 \times F_{\text{max}}}{W \times D}$$

$\sigma_t$ is tensile stress/MPa; $W$ is the thickness of the specimen mm$^{-1}$ ($W = 31.75$); $D$ is the specimen diameter mm$^{-1}$ ($D = 101.6$).

$W_U$ and $W_S$ are calculated by [25]:

$$W_i = \int Fd_s$$

$W_i$ is the area enclosed by the force and vertical displacement in figure 4. ($I$ are $U$ and $S$)/MPa; $F$ and $s$ are the forces loaded at different points and the corresponding vertical displacement/N mm.

$W_D$ is calculated by:

$$W_{Dn} = W_U + W_S$$

$D_n$ is calculated by:

$$D_n = \frac{W_{Dn} - W_{In}}{W_{D0}}$$

$I$ is $D, U$ and $S$; $0$ means the number of freeze-thaw cycles is 0; $n$ is for the number of freeze-thaw cycles.

4. Analysis of test results

4.1. Optimum coal gangue powder replacement rate

With the increase of the replacement rate of coal gangue powder, $\sigma_t$ of the specimen first increases and then decreases. The asphalt mixture with 50% coal gangue powder replacement rate achieves the highest ultimate tensile stress. Figure 5 shows that the $\sigma_t$ of the specimen gradually decreases with the increase in the number of freeze-thaw cycles, and its downward trend of the ultimate tensile stress gradually tends to be gentle. The exponential model of $e^{(a+bx+c/n)}$ is used to fit, and $R^2 = 0.998$ was obtained, which indicates that the damage degree of specimens tend to be stable after several freeze-thaw cycles. The finding of freeze-thaw cycles on specimens in the paper is consistent with the research results of Li et al [23, 26, 27]. Therefore, 50% replacement rate of coal gangue powder can effectively improve the ultimate tensile stress of specimens in freeze-thaw.
composite environment, increase the tensile strength of asphalt, and improve the bearing capacity and strength reserve of materials. The performance of asphalt is improved, the bond between coarse and fine aggregates is stronger, and the deformation of specimens is more obvious when they are destroyed. The ductility of the material is obviously improved, and it has strong bearing capacity and resistance. Under the action of bearing capacity, it absorbs more kinetic energy. The above analysis shows that 50% substitution rate of coal gangue powder improves the influence of freeze-thaw cycle on the bending performance of asphalt mixture, and effectively improves the durability of samples in freeze-thaw composite environment.

4.2. Analysis of erosion mechanism under frozen condition based on energy index
As shown in figure 6, the indexes of $W_D$, $W_U$, and $W_S$ all decrease with the increase of the number of freeze-thaw cycles. Firstly, $W_D$ is the sum of the work done by the specimens from the beginning of loading to complete
failure. When the freeze-thaw cycles are 2, 4 and 6 times, \( W_D \) decreases by 49\%, 65.2\% and 78.4\% respectively compared with the 0 freeze-thaw cycles. The energy index \( W_U \) is used to evaluate the energy dissipation process of the specimen from the initial failure to the complete failure. When the freeze-thaw cycles are 2 times, 4 times and 6 times, \( W_U \) is 51.67\%, 51.21\% and 50.29\% lower than that of the zero freeze-thaw cycles respectively. With the increase of freeze-thaw cycles, the decline range is 50.29\%~51.67\%. For \( W_S \), it is the work done from the begining of loading to the destruction of the specimen, which can well reflect the ability of the specimen to resist destruction. When the freeze-thaw cycles are 2 times, 4 times and 6 times, compared with the zero freeze-thaw cycles, \( W_S \) decreases by 43.6\%, 61.82\% and 76.75\% respectively, and with the increase of freeze-thaw cycles, \( W_S \) decreases by 43.16\%~76.75\%. Therefore, we can conclude that the specimens are highly sensitive to freeze-thaw cycles. When the number of freeze-thaw cycles reaches 4 and 6, the decrease range tends to be stable compared with that of 2 freeze-thaw cycles respectively, and the degree of decline is significantly less than that of 2 freeze-thaw cycles, which supports the above-mentioned conclusion drawn by the \( \delta \).

Through the above analysis, it can be concluded that the energy dissipation process of the ascending and descending sections of the specimen loading curve follows a certain law, that is, the freeze-thaw cycles does not disturb this law at low temperature. With the increase of the number of freeze-thaw cycles, the three indexes of \( W_D, W_U \) and \( W_S \) have the same trend of decreasing amplitude, so it can be concluded that \( W_D, W_U \) and \( W_S \) have a certain degree of synchronization. The influence of the number of freeze-thaw cycles on the three indicators is mainly reflected in the decreasing rate of three indicators, that is, the number of freeze-thaw cycles mainly affect the ability of the specimen to resist freezing damage.

### 4.3. Macroscopic analysis of erosion degree of SCB specimens

Macroscopic pictures of specimen after freeze-thaw cycles and loading failure are shown in figure 7. When the number of freeze-thaw cycles reaches 2, as shown in figure 7(a), the top surface of the semi-circular specimen starts to appear asphalt film damage, resulting in individual aggregates spalling. When the number of freeze-thaw cycles is 4 times, as shown in figure 7(b), it can be seen from the side of the semi-circular specimen that the asphalt film between the coarse aggregates is more seriously damaged and there is a gully phenomenon, but due to the embedded force and the cohesive force inside the aggregate, the spalling has not yet occured. This is because the coarse aggregates have larger edges and corners, which is easy to form voids in the process of compaction. As the number of cycles increases, the voids gradually increase, stress concentration is easy to form, which becomes the weak link of stress. In addition, the surface area of coarse aggregate is large than that of the fine aggregates. During the freeze-thaw cycles, the probability of micro cracks in the coarse aggregates is much greater than that of fine aggregates, which causes the asphalt film on the surface of the coarse aggregates to be destroyed first. With the continuous increase of the loading force, the asphalt on the surface of the coarse aggregate is forced to agglomerate and transfer under the action of kinetic energy, thereby reducing the adhesion of asphalt. Zhai \textit{et al}\[28\] Studied the anti-reflective cracking performance of asphalt mixtures under the freeze-thaw action, and found that freeze-thaw effect attenuated the anti reflective cracking performance of large-size aggregates, which is consistent with the above pictures.
When the number of freeze-thaw cycles reaches 6 times, as shown in figure 7(c), the polyester fiber on the top of the semi-circular specimen begins to precipitate and agglomerate, the asphalt film on the top is seriously damaged, forming more cavities, and some fine aggregates on the surface appear peeling off. In short, the damage degree of the specimen is further aggravated. After 6 cycles of freeze-thaw, the macroscopic situation of the internal damage of the specimen after loading failure is shown in figure 7(d). The asphalt on the surface of the coarse aggregate at the crack of the mixture is seriously lost, and the adhesion of the asphalt is greatly reduced. The coarse aggregates show its original color, and the asphalt is bonded with some fine aggregates and fillers to form a mass, so the adhesion performance of the asphalt is reduced to a large extent.

4.4. Multiple factor analysis

According to the σι of the specimen under different variables, SPSS is used to carry out multiple factor significance analysis. When the freeze-thaw cycle is not carried out, there is no damage inside the test piece after the asphalt mixture is soaked in water, and there is good cohesion between asphalt and aggregate. Under the freeze-thaw cycle, the asphalt film begins to break, and the cohesion between asphalt and aggregate decreases. The analysis results are shown in table 5, and the significant difference coefficients (Sig) are all less than 0.05.
which indicates that the amount of coal gangue powder, the number of freeze-thaw cycles have an influence on the $\delta_i$ of the specimens. The statistic F value shows that the number of freeze-thaw cycles has the greatest impact on asphalt mixture, and coal gangue powder has the lowest influence, which is consistent with the analysis results of Wu et al [10].

4.5. Micro-analysis

Put the prepared asphalt in a low-temperature container with liquid nitrogen and cool it to $-165$ °C for about 30 min, so that all samples become brittle and easily broken. Cut the two ends of the fracture of the specimen with a cutting machine, and the thickness was less than 1 cm, and sprayed gold on its surface to improve the conductivity of asphalt. Put the cross-section specimen in the fixture of SEM test to observe its microscopic morphology. 50% coal gangue powder and 50% mineral powder are used as filler, which can effectively improve the frost resistance of asphalt mixture, and the microscopic pictures of the combined action of the two are shown in figures 8(a) and (b). As displayed in figure 8(a), the specific surface area of coal gangue powder is larger than that of mineral powder [29], and the surface of coal gangue powder is rough and easy to form folds, which can absorb more asphalt and generate Van der Waals force to lock the asphalt molecules [30], thus forming a thick asphalt film and enhancing the asphalt binding force to a certain extent. Secondly, the surface of coal gangue powder is porous, which improves the adsorption capacity of asphalt, while the surface of mineral powder is relatively smooth [10], and the asphalt molecules are easy to slide, resulting in the decrease of adhesion. Meanwhile, it can be seen that the mineral powder and the coal gangue powder are tightly embedded with each other, and the coal gangue powder with a smaller specific surface area is adsorbed around the mineral powder. When 50% coal gangue powder and 50% mineral powder are used as filler, the Gibbs energy between coal gangue powder and mineral powder can be effectively enhanced [31]. In this way, it not only avoid the phenomenon that larger voids are easily formed in the asphalt mixture, but also avoid the high content of coal gangue powder, resulting in the reducing of shear force and bearing capacity. Organic combination of the coal gangue powder and mineral powder between asphalt and produce force, so that the bonding of asphalt mixture is firmer, dense asphalt film is formed on the asphalt surface, and the bending resistance of asphalt mixture is improved.

5. Conclusions

(1) The addition of coal gangue powder could effectively improve the ultimate tensile stress of specimens, enhance the binding force and adsorption force of asphalt, and the asphalt mixture with 50% coal gangue powder replacement rate could reach the highest ultimate tensile stress. Therefore, the optimum proportion of coal gangue powder in asphalt mixture is 50%. Choosing the appropriate replacement rate of coal gangue powder can improve the tensile strength of asphalt mixture, and reduce the harm of freeze-thaw cycle to asphalt mixture.

(2) In the freeze-thaw process, the energy dissipation process in the ascending and descending sections loading curve followed a certain rule and was not affected by freeze-thaw cycles. But the energy dissipation rate of
the specimen is affected by the number of freeze-thaw cycles, and the ultimate tensile stress decreases with the increase of the number of freeze-thaw cycles. Adding appropriate amount of coal gangue powder can reduce energy dissipation and the influence of freeze-thaw cycles.

(3) The freeze-thaw cycles intensified the internal erosion of the asphalt mixture by water and the number of freeze-thaw cycles had a great influence on the durability of asphalt. The addition of coal gangue powder alleviates the erosion of the sample by water and improves the durability of the sample in the freeze-thaw environment. Adding appropriate amount of coal gangue powder can improve the bending performance of asphalt mixture and reduce the erosion of freeze-thaw cycle to asphalt mixture.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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