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Effect of Magnesium Hydroxide Flame Retardant Treatment on the Properties of Corn Stalk Fiber

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Abstract: This study investigates the effect of magnesium hydroxide $Mg(OH)_2$ flame retardant treatment on corn stalk fiber and its impact on the properties of fiber asphalt when mixed at a 3% concentration with asphalt. The study examines the changes in fiber aspect ratio and microscopic morphology before and after flame retardant treatment, and explores the underlying mechanisms that influence the basic performance of fiber asphalt. The effects of flame retardant-treated corn stalk fibers on the asphalt binder were assessed using tests such as softening point, penetration, elongation, temperature scanning, and bending beam rheometer. The results indicate that as the concentration of magnesium hydroxide increases, the three main indicators of the fiber asphalt binder first increase and then decrease. The highest softening point (49.8°C) occurred at a concentration of 2%, the highest penetration (7.6mm) at 1%, and the highest elongation (12.7cm) at 1%. The high and low-temperature performance tests show that the fiber asphalt binder made with 1% magnesium hydroxide-treated corn stalk fibers achieves the best balance of both high and low-temperature properties.

Keywords: Corn stalk fiber; Magnesium hydroxide; Fiber asphalt; Asphalt properties; Microscopic imaging

1 Introduction

As a lightweight, energy-saving, and environmentally friendly material, straw has a broad application prospect in highway construction in recent years [1]. To improve the quality of asphalt pavements and reduce costs, many scholars have studied straw fibers.

Li et al. [2] conducted performance tests on corn stalk fiber asphalt mixtures and analyzed the interaction mechanism between fibers and asphalt from a microscopic perspective using scanning electron microscopy. The results show that corn stalk fibers have good physical properties and can effectively improve the pavement performance of asphalt mixtures. The adsorption of fibers and asphalt forms a three-dimensional network structure, which can prevent the development of cracks in asphalt pavements. Dong [3] conducted a study on the thermal stability limit of corn stalk fibers. The experiment found that, under the condition of 2 minutes of heating, the corn stalk fiber experienced significant weight loss in the temperature range of 190°C to 230°C. In the tests of corn stalk fiber asphalt binder, it was found that asphalt mixed with dried corn stalk fibers had significantly lower high-temperature performance than asphalt mixed with undried corn stalk fibers, and the higher the heating temperature during drying, the worse the high-temperature performance of the modified asphalt. Relevant studies [4, 5] indicate that straw fibers can enhance pavement performance, but these plant fibers are flammable and have poor heat resistance. When mixed with asphalt and used in fiber asphalt binder, they are exposed to high temperatures for extended periods. Asphalt mixtures are often mixed and produced at high temperatures, and no physical or chemical changes should occur during mixing. The fiber properties should not change during the high-temperature mixing, transportation, and paving processes, which could affect their performance [6–8]. Fibers with poor heat resistance will curl or clump when heated, affecting the overall performance of the asphalt mixture.

Therefore, to ensure that corn stalk fibers can be used in road applications and demonstrate their excellent technical performance in asphalt pavements, flame-retardant treatment is applied to the corn stalk fibers. After flame-retardant treatment, the heat resistance of the corn stalk fibers will change, and other pavement properties will also be affected. Hence, it is necessary to study the influence of flame-retardant treatment on the road performance of corn stalk fibers and further investigate the impact on the properties of the corn stalk fiber asphalt binder. This will provide a comprehensive understanding of the effect of flame retardants on the properties of corn stalk fibers and their asphalt binder, broadening the potential applications of plant fibers in asphalt roads. He et al. [9] introduced the development of flame retardants, the types of wood flame retardants, and various wood flame-retardant treatment processes in their article, which provides a reference for the selection of flame retardants and treatment methods in this study.

In terms of the innovation of this paper, existing research mainly focuses on physical treatments or alkaline solution treatments of corn stalk fibers for surface modification purposes. However, there has been no study on the flame-retardant treatment of corn stalk fibers using flame retardants. The research on the treatment of corn stalk fibers with magnesium hydroxide, a type of metal hydroxide, and its effects on the fibers themselves and the performance of fiber-asphalt mastic is innovative. Starting from the flame retardant mechanism of the flame retardant, and combining with microscopic images, this paper explains the impact of magnesium hydroxide concentration on the performance of fiber-asphalt mastic. This approach, which analyzes the influence at different levels-from the treatment solution, to the fibers themselves, and then to the fiber-asphalt mastic-is also a novel perspective.

This study uses magnesium hydroxide as a flame retardant for the treatment of corn stalk fibers. By comparing the impact of three flame retardants on the road performance of corn stalk fibers through fiber road performance tests, such as fiber acidity, water content, oil absorption, and heat resistance, the study further examines the changes in the properties of fiber asphalt binders mixed with flame-retardant-treated corn stalk fibers at fixed dosages. The study analyzes the variation trends of the binder's properties with different flame retardant concentrations. Finally, microscopic analysis is conducted to explore the effect of the flame retardant on the microscopic morphology of the corn stalk fiber, and the mechanisms by which flame-retardant-treated fibers influence the performance of fiber asphalt binder are investigated. This provides a reference for the application of corn stalk fibers in road engineering.

2 Materials and Experimental Preparation

2.1 Preparation of Fiber Samples

The corn stalk skin was sourced from Changchun City, Jilin Province. The corn stalk fibers were prepared by mechanical crushing and wet processing. The corn stalks were peeled and cut into small segments of approximately 10±2mm in length. The segments were soaked in water at room temperature for 4 hours. After soaking, 60g of the material was taken each time and placed into a multifunctional grinder for 1.5 minutes of crushing until all the material was broken down. Finally, the material was air-dried naturally and sieved using a screen with a 0.28mm aperture to obtain qualified corn stalk fibers [10]. The prepared corn stalk fibers are shown in Figure 1.

For the flame retardant treatment of the corn stalk fibers, magnesium hydroxide was selected as the flame retardant. Relevant literature on flame retardants for wood was reviewed and categorized, from which representative types were chosen. Considering factors such as cost, environmental impact, and availability, magnesium hydroxide was selected as the flame retardant. Magnesium hydroxide is a white powder that is poorly soluble in water, with advantages such as being environmentally friendly, highly efficient, and widely applicable, with distinct benefits compared to traditional flame retardants [11–13]. Magnesium hydroxide is shown in Figure 2.

The impregnation method was used to flame retardant treat the corn stalk fibers. For the control of magnesium hydroxide solution concentration, 5g, 10g, and 15g of magnesium hydroxide were respectively mixed with water to make the total solution weight 500g, preparing three concentrations of magnesium hydroxide flame retardant: 1%, 2%, and 3%. The preparation process is shown in Figure 3.

Figure 1. Corn straw fiber

Figure 2. Magnesium hydroxide

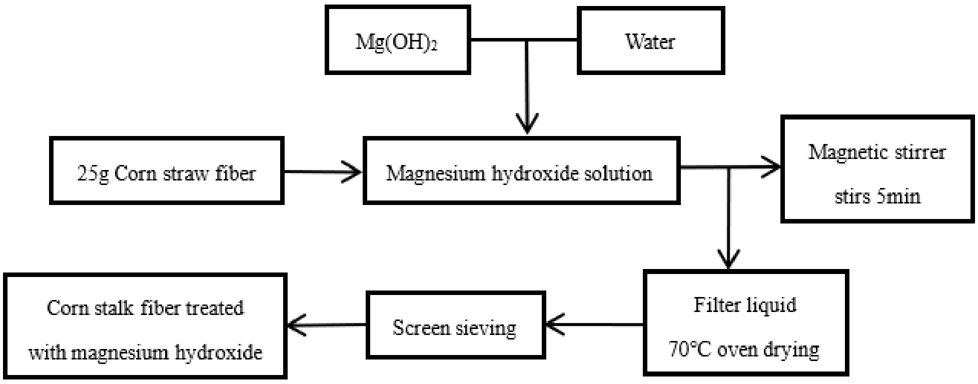


Figure 3. Production process of corn stalk fiber treated with magnesium hydroxide

Thus, four fiber samples were obtained from the experiment, as shown in Table 1. The magnesium hydroxide-treated corn stalk fibers are shown in Figure 4.

Table 1. Fiber sample required for testing

Fiber Type	Corn Stalk Fiber	Corn Stalk Fiber	Corn Stalk Fiber	Corn Stalk Fiber
Magnesium Hydroxide Concentration	0	1%	2%	3%

Figure 4. Magnesium hydroxide treated corn straw fiber

2.2 Base Asphalt

The asphalt used is No. 90 petroleum asphalt. The base asphalt underwent three index tests according to the *Highway Engineering Asphalt and Asphalt Mixture Test Procedures* (JTG E20–2011) [14]. The test results meet the specifications, as shown in Table 2.

Table 2. The index of basic properties of asphalt

Test Item	Test Result	Technical Requirement
Penetration (25°C, 100g, 5s)/mm	86	80-100
Softening Point (Ring and Ball Method)/°C	45.8	$\nless 45$
Ductility (5cm/min, 15°C)/cm	> 100	$\nless 100$

2.3 Preparation of Fiber Asphalt Binder

The fiber content was selected based on the summary and analysis of relevant literature [15, 16], choosing a fiber dosage of 3% for the fiber asphalt mixture.

The corn stalk fibers were placed in a 60°C oven for 1 hour to maintain a constant temperature, then cooled to room temperature in a desiccator. A sufficient amount of dried fibers was pre-weighed for use. The asphalt was heated in an oven at 160°C for 2 hours to ensure good fluidity. The heated asphalt was poured into a beaker and transferred to a 160°C oil bath. The oil bath provided constant-temperature heating, maintaining the fluidity of the asphalt for easy incorporation of fibers.

The corn stalk fibers were added gradually in small portions to ensure even distribution. A handheld stirrer was used during the addition, with both the stirrer rod rotating and the stirrer itself being manually rotated in the opposite direction to ensure the fibers were uniformly dispersed in the asphalt.

After all the fibers were added, the prepared fiber asphalt was placed in a 150°C oven for thermal insulation. During this process, a glass rod was used to stir intermittently to eliminate any bubbles in the asphalt.

Using the above method, four types of fiber asphalt binders were prepared:

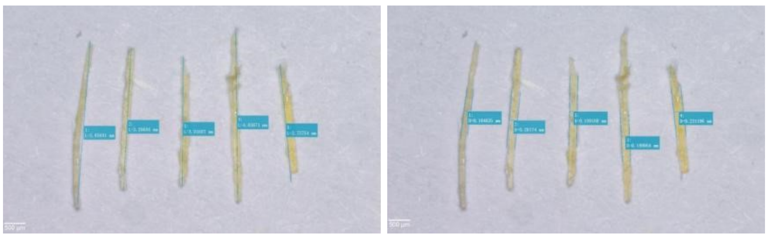
- Original corn stalk fiber asphalt binder,
- 1% magnesium hydroxide-treated corn stalk fiber asphalt binder,
- 2% magnesium hydroxide-treated corn stalk fiber asphalt binder,
- 3% magnesium hydroxide-treated corn stalk fiber asphalt binder.

3 Effect of Magnesium Hydroxide Flame Retardant Treatment on the Morphology of Corn Stalk Fibers

3.1 Aspect Ratio of Corn Stalk Fibers

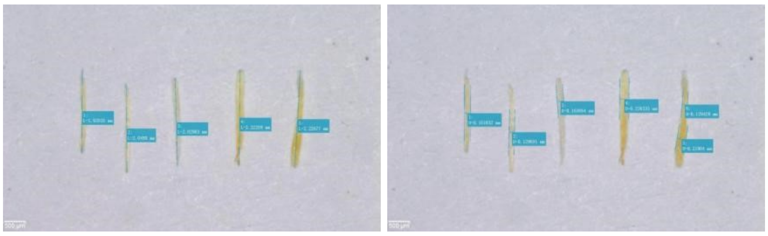
The aspect ratio of fibers refers to the ratio of the fiber length to its diameter. The greater the aspect ratio, the more evenly the fibers distribute in the asphalt, and the larger the specific surface area of the fibers, which can enhance the mechanical properties of the asphalt more effectively [17]. When fibers form a network structure in the asphalt, a good interface layer between the fibers and the asphalt is formed, significantly improving the asphalt's performance.

Both untreated and flame-retardant-treated corn stalk fibers were sampled using the five-point sampling method. Fifty fibers were randomly selected from five different locations, making a total of 250 fibers for each type. The selected fibers (five fibers per group) were placed on a white sheet of paper and observed using a Panasonic XG1600 model industrial camera with a digital microscope, combined with S-EYE2.0 software for fiber measurement. After measuring the length and diameter of each fiber, the average values were taken, and the aspect ratio was calculated using the average length and average diameter. The lens angles used for observation are shown in Figures 5 and 6.



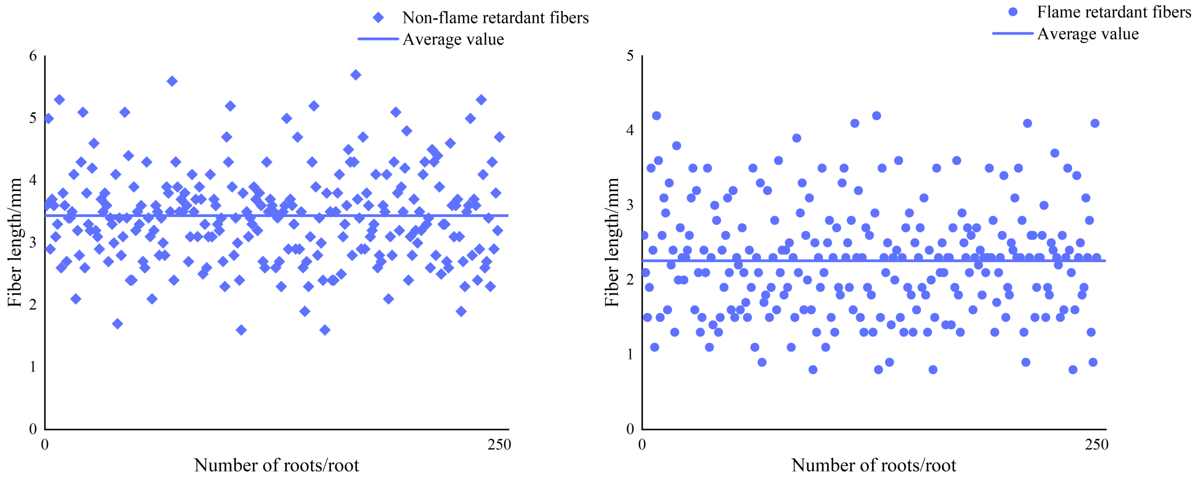
(a) (b)

Figure 5. Observation diagram of untreated corn stalk fiber



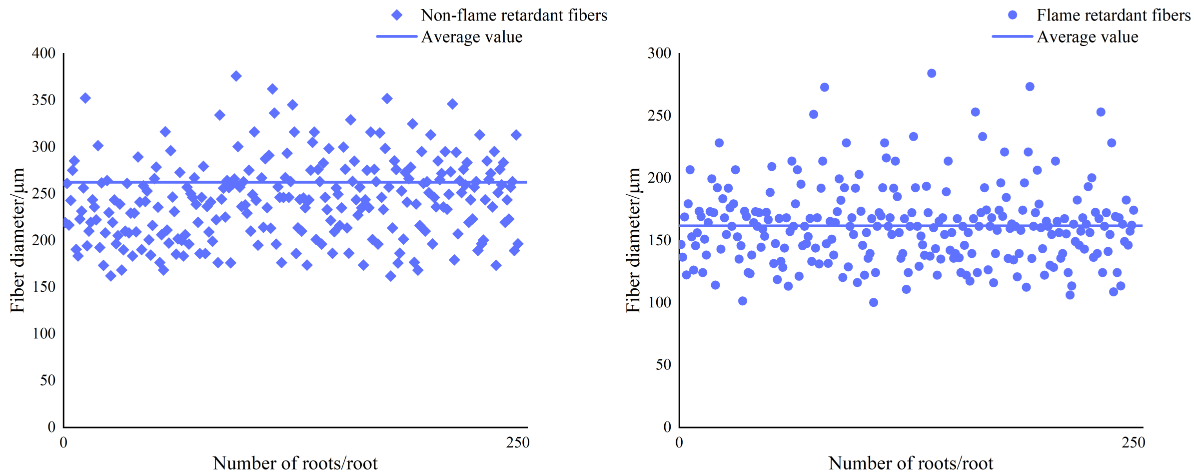
(a) (b)

Figure 6. Observation diagram of flame-retardant treated corn stalk fiber



(a) (b)

Figure 7. Fiber length data distribution



(a) (b)

Figure 8. Fiber diameter data distribution

The untreated corn stalk fibers had an average length of 3.5mm, an average diameter of 257.2μm, and an aspect ratio of 13.61.

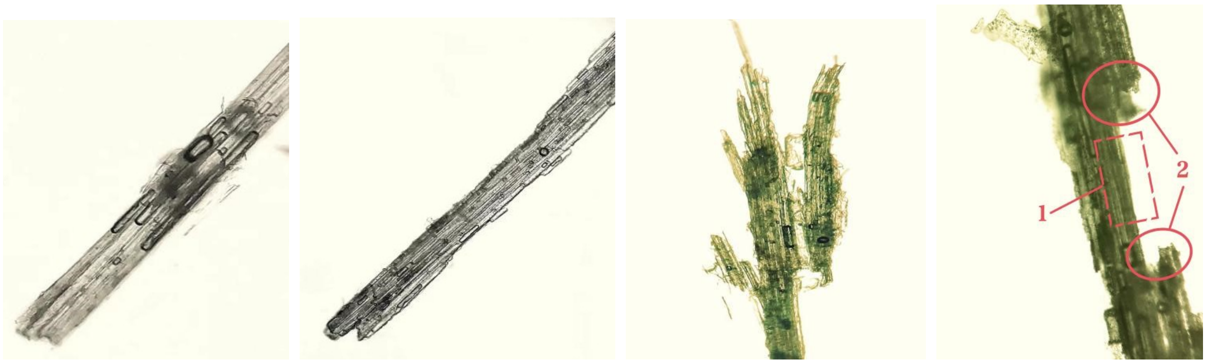
The flame-retardant-treated corn stalk fibers had an average length of 2.3mm, an average diameter of 161.5μm, and an aspect ratio of 14.24.

The distribution and average values of fiber length and diameter are shown in Figures 7 and 8.

3.2 Changes in the Microscopic Morphology of Fibers

The microscopic morphology of the fibers determines the amount of asphalt adsorption and the strength of the bonding. The surface adsorption of asphalt by the fibers helps to improve the stability of the asphalt. The rougher and more angular the surface of the fiber, the greater the number of bonding sites with the asphalt. This can significantly improve certain properties of the asphalt, thereby extending the service life of the pavement and enhancing pavement quality.

The corn stalk fibers before and after flame retardant treatment were observed under a polarizing microscope. Four fiber samples were randomly selected and observed, and representative images were captured. Four representative images are shown in Figure 9.



(a) (b) (c) (d)

Figure 9. Various forms of corn stalk fiber before and after flame retardant treatment

During the observation process, significant morphological changes in the corn stalk fibers before and after flame-retardant treatment were evident. Subgraph (a) of Figure 9 shows the untreated corn stalk fibers. From the image, it can be seen that the edges of the fibers are neat and smooth, and the fibers are relatively straight with a consistent width. The edges are parallel, and the ends are regularly cross-sectioned, indicating tight bonding between the fiber bundles.

As shown in subgraph (b) of Figure 9, after flame-retardant treatment, the edges of the corn stalk fibers become uneven, and the diameter distribution is irregular, showing signs of erosion by the magnesium hydroxide flame retardant. Compared to the side edges in (a), the edges in (b) exhibit more fiber cavity structures. The ends are no longer smooth but have irregular gaps, and the profile of the ends is uneven. The fiber bundle pattern is also more prominent compared to (a), indicating that not only the side edges but also the entire surface of the fiber has developed longitudinal grooves at varying depths, increasing the roughness of the fiber. The greater the fiber roughness, the larger the specific surface area of the fiber, which increases the contact area with the asphalt, thus improving certain properties of the fiber-asphalt binder.

Subgraph (c) of Figure 9 shows that after treatment with higher concentrations of magnesium hydroxide flame retardant, some fiber bundles begin to separate, leading to phenomena such as fiber splitting and fiber thinning, especially at the fiber ends. This phenomenon is especially apparent in fibers treated with 3% magnesium hydroxide. Pectin is the main binding substance between fiber bundles. After flame-retardant treatment, the separation between fibers occurs, suggesting that magnesium hydroxide may remove the pectin content from the fibers. The separation of fiber bundles also increases the specific surface area of the fibers, which enhances the adsorption of oil on the fiber surface. The protruding fiber bundles or cavity structures will also form a strong structural bond with the asphalt, leading to an increase in the proportion of structural asphalt, which has higher viscosity, stability, and lower temperature sensitivity. This improves certain properties of the fiber-asphalt binder.

However, the concentration of the flame retardant treatment does not always correlate positively with the performance of the fiber-asphalt binder. Related studies have shown [18, 19] that high-concentration alkaline solutions can reduce the lignin content in fibers, weaken the strength of the cell walls, and cause the fiber bundles to disperse to some extent, damaging the supporting structure of the fiber. During processing, fiber cells undergo delamination, reducing the interfacial strength between the cells, which leads to a decrease in fiber strength. Similar situations can be observed in images like subgraph (d) of Figure 9. Subgraph (d) of Figure 9 shows corn stalk fibers treated with 3% magnesium hydroxide, with large block-like gaps in the middle of the fibers. As shown in position 1 of subgraph (d) of Figure 9, the longitudinal cross-section profile is regular, aligned with the fiber bundle direction. The magnesium hydroxide treatment has removed the pectin content from the fibers, causing a decrease in the bonding between the fiber bundles and a weakening of the interfacial strength. The fiber bundles are no longer tightly connected, resulting in defects in the overall mechanical properties of the fibers, such as reduced toughness. When the fibers are subjected to twisting or shear forces, stress tends to concentrate at the weakest bonding interface, leading to longitudinal splitting, and the fiber bundles on both sides will separate. After longitudinal splitting, the fiber breaks into two thinner parts, and when subjected to tension or transverse shearing, the thinnest part is likely to experience transverse fractures. If both ends of the longitudinal split also experience transverse fractures, the situation shown in position 2 of subgraph (d) of Figure 9 occurs, resulting in block-like detachment in the middle of the fiber. The fiber becomes incomplete, and its supporting structure is damaged, which indeed causes a decrease in the mechanical properties of the fiber and weakens the strength. Consequently, some properties of the resulting fiber-asphalt binder will also decrease.

In summary, changes in fiber morphology can affect the performance of the fiber-asphalt binder. Using an appropriate concentration of magnesium hydroxide flame retardant for the treatment of corn stalk fibers can improve the surface roughness of the fibers, enhancing the bond with the asphalt interface. However, excessively high concentrations of magnesium hydroxide flame retardant can have adverse effects on the fibers, which in turn affects the performance of the fiber-asphalt binder.

4 Effects of Magnesium Hydroxide on Basic Properties of Fiber-Asphalt Binder

To evaluate the preliminary improvement effects of magnesium hydroxide flame retardant on the fiber-asphalt binder made from corn stalk fibers, it is necessary to study the road performance of the fiber-asphalt binder made from fibers treated with different concentrations of magnesium hydroxide flame retardant, as well as the fiber-asphalt binder made from untreated corn stalk fibers. Asphalt is a viscoelastic material, and the three main empirical indicators commonly used to evaluate its basic properties are penetration, softening point, and ductility. Ductility, softening point, and penetration tests were conducted on the fiber-asphalt binder, with parallel tests carried out on the binder made from untreated corn stalk fibers. By measuring these three indicators, the changes in the properties of the fiber-asphalt binder made from corn stalk fibers treated with different concentrations of magnesium hydroxide flame retardant can be analyzed. All test methods followed the requirements of JTG E20—2011. This study explores the effect of different concentrations of magnesium hydroxide on the road performance of the fiber-asphalt binder and investigates how the magnesium hydroxide flame retardant treatment of corn stalk fibers affects the basic properties of the binder.

4.1 Flame Retardant Mechanism of Magnesium Hydroxide

Magnesium hydroxide absorbs a large amount of heat when heated, which helps reduce the temperature of flammable materials below their ignition point. It begins to decompose at around 340°C, producing magnesium oxide and water. This decomposition absorbs heat from the surface of the burning material, contributing to the flame retardant effect. At the same time, a large amount of water vapor is released, which dilutes the oxygen on the material's surface. The magnesium oxide formed during decomposition adheres to the surface of the flammable material, further preventing combustion [20]. Throughout the entire flame-retardant process, magnesium hydroxide not only generates no harmful substances but also absorbs a significant amount of harmful gases and smoke produced by the fire.

When preparing corn stalk fiber-asphalt mastic, the temperature does not reach the decomposition point of magnesium hydroxide. In this case, magnesium hydroxide primarily exerts its flame retardant effect through physical barriers and chemical actions. Physical Barrier Effect: Magnesium hydroxide particles are absorbed into the fibers, forming a physical barrier that prevents the transfer of heat and oxygen. When flames come into contact with the material, the magnesium hydroxide particles reflect and scatter heat, reducing the heat transferred to the interior of the material, thus delaying or preventing the spread of combustion. Chemical Action: When fibers are mixed with asphalt, magnesium hydroxide can react with the acidic substances in the asphalt, generating water and other compounds. These reaction products dilute the concentration of combustible gases and oxygen, reducing the likelihood of combustion. Additionally, magnesium hydroxide can react with the active groups in the asphalt to form a protective layer that further inhibits combustion. In addition to these two mechanisms, magnesium hydroxide also has hygroscopic properties, meaning it can absorb moisture from the material's surface. In high-temperature environments, this helps lower the surface temperature of the material, reducing the formation of combustible gases and further contributing to its flame retardant effect.

4.2 Softening Point

Asphalt is a temperature-sensitive non-Newtonian fluid, meaning that its strength decreases with increasing temperature. Fiber-asphalt binders, which exhibit similar properties, will have better high-temperature stability with a higher softening point. Therefore, the softening point can be used as an indicator to assess the high-temperature stability of fiber-asphalt binders [21]. Softening point tests were conducted on the five types of fiber-asphalt binders. The results and trends, averaged from multiple tests, are shown in Figure 10.

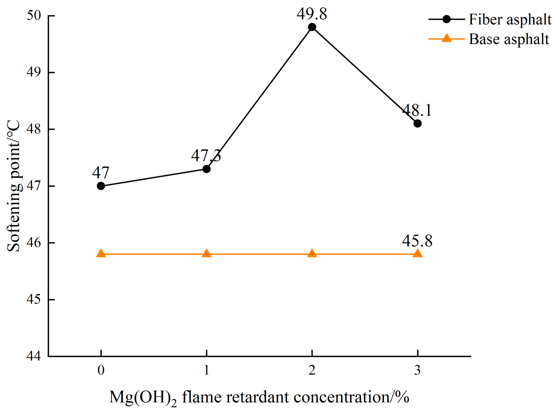


Figure 10. Softening point test results

From Figure 10, it can be seen that the softening point of the matrix asphalt is 45.8°C. After adding untreated corn stalk fibers, the softening point increases to 47°C, which is a 2.6% increase compared to the matrix asphalt. This indicates that the addition of fibers improves the thermal stability of the asphalt. After treating the corn stalk fibers with magnesium hydroxide, the softening point of the resulting fiber-asphalt binder fluctuates. The softening point of the 1% magnesium hydroxide-treated corn stalk fiber-asphalt binder is 47.3°C, which is slightly higher than the original corn stalk fiber-asphalt binder. The softening point of the 2% magnesium hydroxide-treated corn stalk fiber-asphalt binder is 49.8°C, which represents a 6% increase over the original corn stalk fiber-asphalt binder and an 8.7% increase over the matrix asphalt. This shows that the treatment with 2% magnesium hydroxide significantly improves the softening point of the asphalt. The softening point of the 3% magnesium hydroxide-treated corn stalk fiber-asphalt binder is 48.1°C, which is a 2.3% increase compared to the original corn stalk fiber-asphalt binder and a 5% increase compared to the matrix asphalt. However, it is 3.4% lower than the binder treated with 2% magnesium hydroxide. Thus, although the 3% magnesium hydroxide treatment also improves the softening point, the effect is less pronounced than that of the 2% concentration.

Overall, the softening point of the fiber-asphalt binder increases first and then decreases with increasing magnesium hydroxide concentration, with the 2% concentration resulting in the highest softening point. At a concentration of 3%, the softening point does not continue to increase but rather decreases. All four types of fiber-asphalt binders have a higher softening point than the matrix asphalt. On the one hand, the addition of fibers itself plays a reinforcing role in the asphalt, forming a three-dimensional network structure of asphalt colloid when the fibers interlock. This improves the high-temperature stability of the fiber-asphalt binder compared to the matrix asphalt. On the other hand, the magnesium hydroxide treatment of corn stalk fibers is effective, enhancing the fibers' ability to withstand high temperatures and allowing them to exhibit their tensile strength, good toughness, and other performance advantages, which contribute to the high-temperature stability of the fiber-asphalt binder.

4.3 Penetration

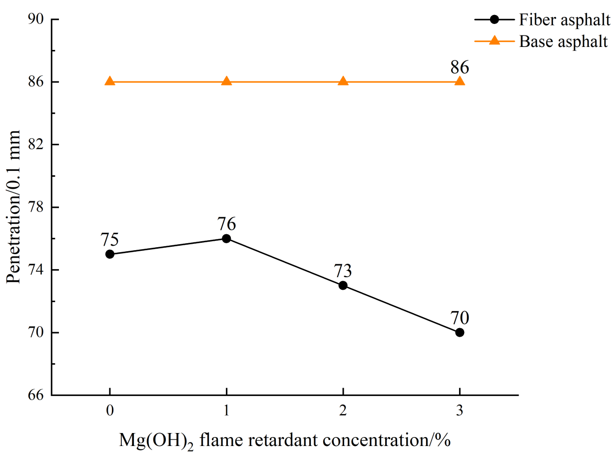


Figure 11. Softening point test results

The penetration of asphalt is used to measure the soft-hardness and viscosity of the asphalt, reflecting its relative viscosity under certain conditions. It is used to determine the deformation ability and flowability of the asphalt [22]. Penetration tests were conducted on the five types of fiber-asphalt binders, and the results and trends, averaged from multiple tests, are shown in Figure 11.

From Figure 11, it can be seen that the penetration of the matrix asphalt is 86, while the penetration of the original corn stalk fiber-asphalt binder is 75, a decrease of 12.8%, indicating that the addition of fibers increases the overall viscosity of the asphalt. With different concentrations of magnesium hydroxide used to treat the corn stalk fibers, the penetration of the resulting fiber-asphalt binders also changes. For the 1% magnesium hydroxide-treated corn stalk fiber-asphalt binder, the penetration is 76, which is an increase of 1.2% compared to the original corn stalk fiber-asphalt binder. The penetration of the 2% magnesium hydroxide-treated corn stalk fiber-asphalt binder is 73, a decrease of 2.7% compared to the original corn stalk fiber-asphalt binder and a decrease of 15.1% compared to the matrix asphalt. In this case, the penetration of the fiber-asphalt binder is lower than both the matrix asphalt and the original corn stalk fiber-asphalt binder. The 3% magnesium hydroxide-treated fiber-asphalt binder has the lowest penetration at 70, a decrease of 6.7% compared to the original corn stalk fiber-asphalt binder and a decrease of 18.6% compared to the matrix asphalt.

From the data analysis, it can be summarized that the penetration of the fiber-asphalt binder increases slightly at 1% concentration of magnesium hydroxide and then decreases continuously. The penetration of all four fiber-asphalt binders is lower than that of the matrix asphalt. On one hand, the fibers in the binder are randomly distributed and interlocked, and the fibers adsorb free oil, reducing the flowability of the asphalt and increasing its viscosity [23]. On the other hand, after the magnesium hydroxide treatment, the surface roughness of the corn stalk fibers increases, and the branched fiber tips are able to adsorb the asphalt more firmly. This increases the overall viscosity of the fiber-asphalt binder, leading to a further decrease in the penetration.

4.4 Ductility

Ductility is an indicator of the plastic deformation ability of asphalt materials, which can well reflect the low-temperature properties of the material. The higher the low-temperature ductility value, the better the low-temperature properties of the material [24]. To better characterize the low-temperature performance of the fiber-asphalt binder using ductility, the test temperature was set at 5°C. The ductility tests were performed on the five types of fiber-asphalt binders, and the results and trends, averaged from multiple tests, are shown in Figure 12.

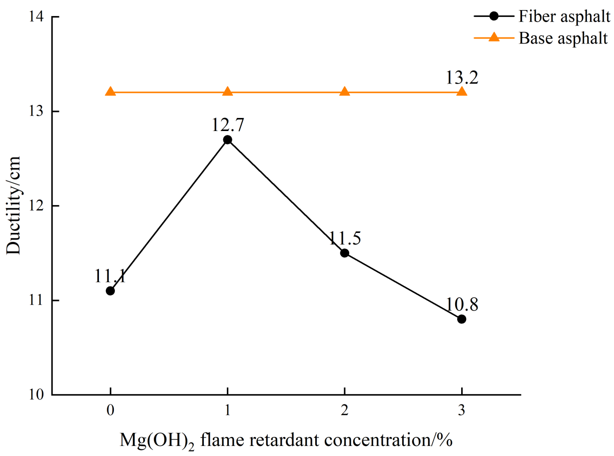


Figure 12. Ductility test results

From Figure 12, it can be observed that the ductility of the asphalt with added corn stalk fibers is smaller than that of the matrix asphalt, which has a ductility of 13.2cm. The ductility of the fiber-asphalt binder made from untreated corn stalk fibers is 11.1cm, a significant decrease of 15.9% compared to the matrix asphalt. For the 1% magnesium hydroxide-treated corn stalk fiber-asphalt binder, the ductility is 12.7cm, which is much closer to the matrix asphalt, showing a decrease of 3.8%. Additionally, at this concentration, the ductility of the fiber-asphalt binder shows a significant jump, increasing by 14.4% compared to the untreated fiber-asphalt binder. However, as the concentration of magnesium hydroxide increases, the ductility of the fiber-asphalt binder gradually decreases. The ductility of the 2% magnesium hydroxide-treated corn stalk fiber-asphalt binder is 11.5cm, which is an increase of 3.6% compared to the untreated fiber-asphalt binder but a decrease of 12.9% compared to the matrix asphalt. At a concentration of 3%, the ductility of the fiber-asphalt binder is the lowest among all four concentrations, decreasing to 10.8 cm, which is a reduction of 2.7% compared to the untreated fiber-asphalt binder and 18.2% compared to the matrix asphalt.

In summary, as the concentration of magnesium hydroxide increases, the ductility of the fiber-asphalt binder first increases significantly at 1%, then decreases with increasing concentration, reaching its lowest value at 3%. The matrix asphalt has the highest ductility among the five types of binders. On one hand, the addition of fibers enhances the resistance to deformation at high temperatures, but at low temperatures, the asphalt itself becomes harder and more brittle. The three-dimensional network structure in the fiber-asphalt binder is not efficient in dispersing stress, which leads to reduced plasticity and makes the material more prone to fractures during stretching [25]. Therefore, the addition of fibers reduces the low-temperature performance of the asphalt. On the other hand, the change in ductility with varying concentrations of magnesium hydroxide treatment indicates that the treatment alters the morphology of the fibers, which in turn affects the performance of the fiber-asphalt binder. The higher the concentration of the flame retardant, the more significant the change in fiber morphology.

5 The Effect of Magnesium Hydroxide on the High and Low-Temperature Performance of Fiber Asphalt

Asphalt is a viscoelastic material that is highly sensitive to temperature, with more complex temperature sensitivity compared to other types of viscoelastic materials. The high-temperature performance of asphalt plays a crucial role in resisting high-temperature rutting deformation during the service of asphalt pavements [26]. At low temperatures, the viscosity of the asphalt increases, and its flowability decreases. If the temperature drops too low and lasts for a long period, the asphalt's elastomeric properties degrade, leading to increased brittleness. At high temperatures, the viscosity of the asphalt decreases, flowability increases, and elasticity reduces, leading to more plastic deformation, which may result in rutting and other pavement distresses. Asphalt is a viscoelastic mixture under normal conditions.

Basic asphalt binder performance tests have limitations in evaluating the high and low-temperature performance of fiber-asphalt binders made from magnesium hydroxide-treated corn stalk fibers. Therefore, further investigation using Dynamic Shear Rheometer (DSR) temperature sweep tests and Bending Beam Rheometer (BBR) tests is necessary to assess the high and low-temperature performance of fiber-asphalt binders. The impact of different concentrations of magnesium hydroxide-treated corn stalk fibers on the high and low-temperature properties of the fiber-asphalt binder is studied under the same fiber content.

5.1 High-Temperature Performance

A DSR was used to conduct a temperature sweep test on the fiber-asphalt binder, studying the impact of flame retardant treatment on the high-temperature rheological properties of the binder. The test parameters include the complex shear modulus (G*) and phase angle (δ). The temperature scan starts at 46°C with an interval of 6°C for each group.

The complex modulus of the fiber-asphalt binder made from fibers treated with various concentrations of flame retardant is shown in Figure 13.

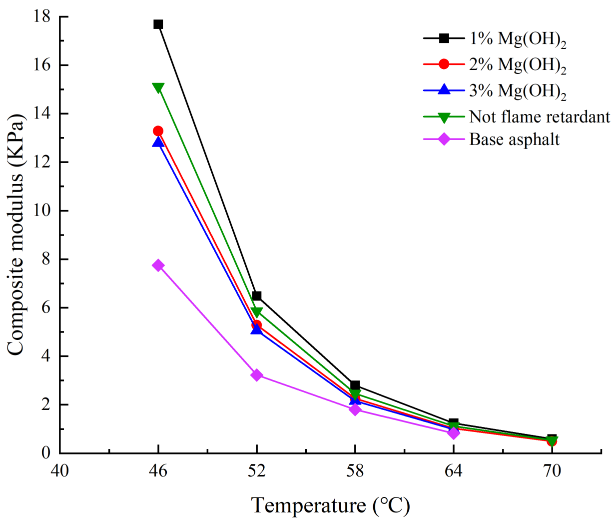


Figure 13. Effect of magnesium hydroxide on composite modulus of fiber asphalt

From the figure, it can be observed that the complex modulus of fiber-asphalt binders made from magnesium hydroxide-treated fibers follows a regular trend. When fibers are treated with a 1% magnesium hydroxide concentration, the complex modulus of the resulting fiber-asphalt binder is the highest. The binder made from untreated fibers comes second, while the binders made from fibers treated with 2% and 3% magnesium hydroxide fall into third and fourth places, respectively. The matrix asphalt has the lowest complex modulus. Additionally, both the matrix asphalt and the fiber-asphalt binder made with 3% magnesium hydroxide treatment do not show a complex modulus or phase angle above 70°C, as the temperature scan range does not reach this temperature. After the fibers undergo flame retardant treatment, the high-temperature deformation resistance of the asphalt binder is improved to a certain extent. The treatment at 1% concentration is the most effective. As the concentration of the flame retardant increases, the complex modulus of the fiber-asphalt binder decreases. This is because higher concentrations of flame retardant can damage the fibers' structural integrity, reducing their reinforcing effect in the asphalt, which ultimately lowers the overall high-temperature deformation resistance of the binder. The complex modulus of the binder made from untreated fibers is higher than that of binders made with 2% and 3% flame retardant-treated fibers because untreated fibers are generally larger, thicker, and longer. These fibers are better able to resist high-temperature deformation, leading to superior high-temperature performance.

The phase angle of the fiber-asphalt binder made from fibers treated with different concentrations of flame retardant is shown in Figure 14.

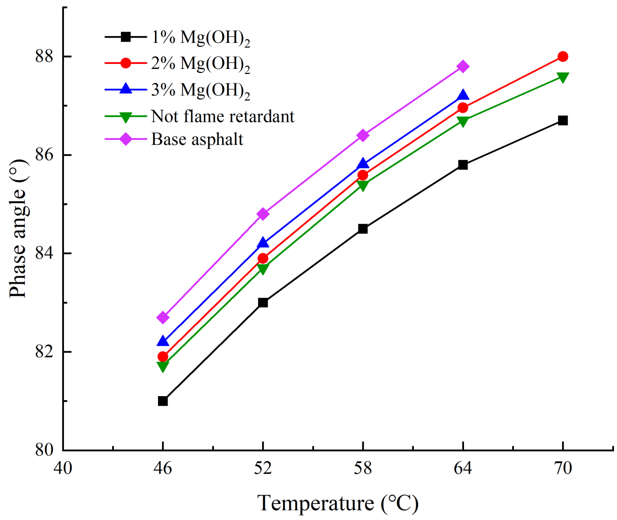


Figure 14. Effect of magnesium hydroxide on phase angle of fiber asphalt

The relationship between the viscosity and elasticity of asphalt can measure its ability to resist permanent deformation and fatigue cracking under load. When asphalt has a greater elastic deformation capacity to withstand more load, its ability to resist rutting during the service life of the pavement is improved. Similarly, when asphalt shows significant flexibility and elasticity, its ability to resist fine cracking during the service life is stronger [27]. The phase angle (δ) is a relative indicator of the recoverable and non-recoverable deformation of materials under repeated shear, representing the ratio of viscous to elastic components. A larger δ value indicates that the asphalt contains more viscous components, and its elastic-plastic properties are poorer.

As the temperature increases, the phase angle of asphalt mastic also increases. The larger the phase angle, the weaker the recoverable ability; conversely, the smaller the phase angle, the greater the recoverable ability. The phase angle of fiber asphalt made with 1% magnesium hydroxide flame retardant treatment is the smallest, followed by untreated fiber asphalt, and the phase angles of fiber asphalts made with 2% and 3% flame retardant treatment are ranked third and second, respectively. The phase angle of the base asphalt is the largest. A larger phase angle means that under load and environmental factors, permanent deformation is more likely to occur, while the inclusion of fibers reduces the phase angle of the asphalt mastic, indicating an increase in the elastic component of the asphalt, which contributes to improving the high-temperature deformation resistance of the asphalt mastic. The 1% flame retardant concentration is the most effective in enhancing the high-temperature performance of fiber asphalt mastic. At high temperatures, smaller fibers in the asphalt do not have a strong recovery ability compared to larger fibers. Smaller fibers adhere to less asphalt, have a weaker reinforcing effect in the asphalt, and cause less hindrance to the asphalt's flow. The strength and toughness of the fibers in the asphalt are not as effective as that of larger fibers. The phase angle reflects the ratio of the elastic to viscous components in fiber asphalt mastic. The larger the phase angle, the more viscous the component is, and the more likely permanent deformation is to occur. The phase angle increases with rising temperature, and the proportion of viscous components increases, causing the fiber asphalt mastic to gradually transition from an elastic to a viscous state, making it more prone to permanent deformation.

5.2 Low-Temperature Performance

Asphalt mastic, as an important component of asphalt mixtures, plays a significant role in low-temperature cracking of asphalt pavements [28]. In freezing regions, due to very low winter temperatures, asphalt materials become brittle and hard, thereby reducing their stress relaxation ability. Under these conditions, the tensile stress inside the asphalt pavement materials can exceed their ultimate tensile strength due to the combined effects of traffic loads and low temperatures, leading to low-temperature cracking of the pavement. Improving the low-temperature crack resistance of asphalt mixtures requires improving the low-temperature performance of asphalt mastic [29]. To study the effect of various flame retardants on the low-temperature rheological performance of fiber asphalt mastic, low-temperature BBR tests were conducted on various fiber asphalt mastics. The test indices were the creep stiffness (S) and the creep rate (m). The creep rate of asphalt reflects its flexibility from different angles. A higher creep rate indicates better low-temperature performance. The greater the brittleness of the asphalt, the more likely it is to develop fine cracks during the service life of the pavement, making it difficult to guarantee the overall quality. Therefore, it is preferable to choose an asphalt with lower creep stiffness to extend the service life of the pavement. The test temperatures selected were -6°C, -12°C, and -18°C, with the results shown in Table 3.

Table 3. BBR test results of different kinds of fiber asphalt mortar

Fiber No.	Fiber Type	Flame Retardant Type	Concentration (‰)	Creep Stiffness (MPa)			Creep Rate		
				Temperature (°C)					
				-6	-12	-18	-6	-12	-18
1	No Fiber	No Flame Retardant	0	49	127	224	0.557	0.468	0.362
2	Corn Stalk Fiber	No Flame Retardant	0	58	146	276	0.521	0.411	0.330
3			1	53	135	257	0.544	0.453	0.349
4	Corn Stalk Fiber	Magnesium Hydroxide	2	55	141	268	0.541	0.432	0.342
5			3	57	145	274	0.53	0.417	0.337

The trend of creep stiffness of each fiber asphalt mastic at different temperatures is shown in Figure 15.

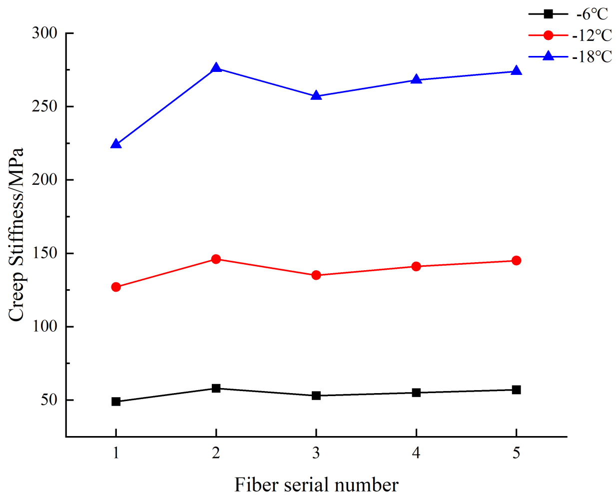


Figure 15. Creep stiffness of each fiber asphalt mortar at different temperatures

Creep stiffness reflects the flexibility of asphalt. The smaller the creep stiffness, the better the asphalt's performance in resisting low-temperature cracking. As shown in the figure, the creep stiffness of the asphalt mastic increases as the temperature decreases. As the temperature decreases, the difference in creep stiffness of fiber asphalt mastics made from fibers treated with magnesium hydroxide flame retardant gradually increases. The creep stiffness of the matrix asphalt is the smallest, while the creep stiffness of the fiber asphalt mastic made from untreated corn stalk fiber is the largest. This indicates that the incorporation of fibers weakens the low-temperature crack resistance of the asphalt mastic.

Corn stalk fibers themselves are not sensitive to temperature and maintain a certain toughness at low temperatures, which helps improve the crack resistance of asphalt and resist some tensile stress. Therefore, even though the creep stiffness of the fiber asphalt mastic is numerically larger at low temperatures, the fibers still alleviate some factors contributing to asphalt low-temperature cracking.

The creep stiffness of the fiber asphalt mastic made from corn stalk fibers treated with magnesium hydroxide flame retardant fluctuates between the maximum and minimum values. Specifically, as the concentration increases, the creep stiffness increases, and the growth range becomes larger as the temperature decreases. Under low-temperature conditions, asphalt becomes brittle overall, and flame-retardant treatment causes the fibers to generally become smaller. The reduction in fiber length and width results in more even stress distribution in the asphalt, making it less likely to produce large stress concentrations. Compared to larger fibers, these smaller fibers can better resist low-temperature cracking. As the flame retardant concentration increases, the surface of the fiber becomes more uneven, with more groove-like structures, which increases the bonding area between the fiber and the asphalt. This results in a larger overall structure in the asphalt, causing the asphalt's flexibility to decrease and making it harder and more brittle at low temperatures. This is reflected in the data, showing that as the flame retardant concentration increases, the creep stiffness of the fiber asphalt increases.

The trend of the creep rate of each fiber asphalt mastic at different temperatures is shown in Figure 16.

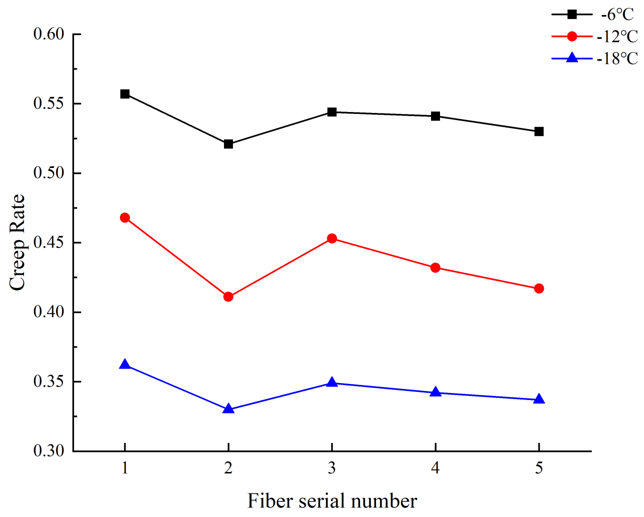


Figure 16. Creep rate of each fiber asphalt mortar at different temperatures

Creep rate can reflect the stress relaxation capability of asphalt. The larger the creep rate, the better the low-temperature cracking resistance. From the figure, it can be seen that as the temperature decreases, the creep rate of the asphalt mastic becomes smaller. In contrast to creep stiffness, the matrix asphalt has the largest creep rate, while the fiber asphalt mastic made from untreated corn stalk fibers has the smallest creep rate. The creep rate of fiber asphalt mastic made from corn stalk fibers treated with magnesium hydroxide flame retardant fluctuates between these two extremes. Specifically, as the flame retardant concentration increases, the creep rate decreases. The addition of fibers forms an irregular network in the asphalt, which hinders the internal stress relaxation of the asphalt. The smaller the fibers, the smaller the hindrance. As the flame retardant concentration increases, the fiber morphology becomes rougher, with an increased specific surface area and more bonding sites with the asphalt. This increases the hindrance to stress distribution in the asphalt, making it more likely to accumulate stress at the fiber interfaces, leading to potential cracking at these points.

6 Conclusion and Outlook

6.1 Conclusion

Based on the experimental data and microscopic analysis presented earlier, the effects of different concentrations of magnesium hydroxide flame retardant-treated corn stalk fibers on the basic properties of fiber asphalt mastic can be summarized as follows:

(1) Corn Stalk Fiber Morphology Changes with Different Concentrations of Magnesium Hydroxide Flame Retardant Treatment

The fiber morphology changes with different concentrations of magnesium hydroxide flame retardant treatment, which leads to different effects on the performance of the fiber asphalt mastic. The size, length, width, and aspect ratio of the fibers changed after the flame retardant treatment. The flame-retardant-treated corn stalk fibers are finer and shorter than untreated fibers, with a larger aspect ratio than untreated fibers. This leads to different performance changes in the asphalt after fiber incorporation. Microscopic observations show that untreated corn stalk fibers have regular, smooth profiles, and the fiber bundles are tightly connected without loosening or separation. Magnesium hydroxide flame retardant treatment removes wax, grease, and pectin components from the corn stalk fibers, resulting in irregular fiber profiles with finer splitting and increased roughness. The fibers have more contact with the asphalt, adsorbing more asphalt and forming a strong structural asphalt with protruding fiber bundles or cavities. However, if the flame retardant concentration is too high, the fibers may experience splitting and separation, resulting in block-like peeling. The interfacial bonding between fiber bundles weakens, and the fiber structure is damaged, which reduces fiber strength and negatively affects the performance of the fiber asphalt mastic.

(2) Best High-Temperature Performance at 2% Magnesium Hydroxide Flame Retardant Concentration

At a 2% magnesium hydroxide flame retardant concentration, the fiber asphalt mastic exhibits the best high-temperature performance. As the concentration of magnesium hydroxide flame retardant treatment on corn stalk fibers increases, the softening point of the fiber asphalt mastic increases from 1% to 2%, then decreases at 3%. The softening point reflects the high-temperature performance of asphalt. After flame retardant treatment, the morphology of the corn stalk fibers changes, the surface becomes rougher, and the cavity structure is exposed. The fiber ends split, enhancing the adhesion and interlocking between the fibers and asphalt, which increases the high-temperature stability of the fiber asphalt mastic. At 3%, the concentration is too high, and the treatment causes some damage to the fiber structure. Although the contact area between the fiber and asphalt increases, the fiber strength decreases, leading to a decrease in the softening point compared to the 2% treatment, though still higher than that of the untreated corn stalk fiber asphalt mastic.

(3) Best Low-Temperature Performance at 1% Magnesium Hydroxide Flame Retardant Concentration

The penetration and ductility of the fiber asphalt mastic decrease sequentially from 1% to 3% magnesium hydroxide flame retardant concentration. As the flame retardant concentration increases, the treated corn stalk fibers become more irregular in shape, with rougher surfaces. The fibers adsorb more asphalt and form more structural asphalt, which weakens the overall flowability and increases the viscosity of the mastic, thus improving its resistance to deformation. At 3%, although the fiber mechanical strength decreases, it does not weaken the increase in viscosity and the improvement in flowability. It can be inferred that at low temperatures or room temperature, the adhesion between the fibers and asphalt plays a major role, which causes the fiber asphalt mastic to still maintain higher hardness, resulting in a decrease in both penetration and ductility. When the flame retardant concentration is between 0 and 1%, both penetration and ductility increase to varying degrees. This is likely due to the fiber size change during the flame retardant treatment process, which breaks the fibers into smaller pieces. Finer fibers are more effective in dispersing stress in the asphalt, improving the flowability and plasticity of the fiber asphalt mastic, thus enhancing its low-temperature performance.

(4) High and Low-Temperature Performance of Fiber Asphalt Mastic Treated with Magnesium Hydroxide Flame Retardant

From the temperature sweep test and low-temperature bending beam test, the influence of magnesium hydroxide on the high and low-temperature performance of fiber asphalt can be further investigated. Overall, the fiber asphalt mastic made from fibers treated with magnesium hydroxide at a 1% concentration shows the best high and low-temperature performance compared to mastic treated with 0%, 1%, 2%, and 3% concentrations. At this concentration, the fiber asphalt mastic achieves the best balance between high and low-temperature performance. The 1% magnesium hydroxide flame retardant treatment does not over-process the fibers. The wax, grease, and pectin components are effectively removed, and the surface roughness of the fibers is increased, improving adhesion between the fibers and asphalt. At the same time, the fiber bundles are not overly separated, and no significant block-like defects occur, so the fiber strength is maintained. This allows the fibers to effectively perform their role in enhancing the toughness and interlocking effect in the asphalt. At high temperatures, this provides a strong anti-deformation capability, while at low temperatures, it helps disperse stress in the asphalt and alleviates local cracking.

6.2 Outlook

Through the treatment of corn stalk fibers with magnesium hydroxide as a flame retardant, the performance of the resulting corn stalk fiber-asphalt mastic has been further improved, opening up new prospects for the application of corn stalk fibers in asphalt pavements. As a material for road surface layers, flame-retarded corn stalk fibers can be applied in a wider range of engineering scenarios, such as highways, urban expressways, and airport runways, thereby enhancing the durability, environmental sustainability, and cost-effectiveness of road projects. This also expands the application scope and improves the performance of corn stalk fiber asphalt.

The study of the effects of different concentrations of flame retardants on the performance of corn stalk fiber-asphalt mastic provides important parameters for practical engineering applications. By targeting road material performance goals, it is possible to select the most appropriate concentration of flame retardants. When the magnesium hydroxide treatment concentration is 2%, the high-temperature performance of the corn stalk fiber-asphalt mastic is optimal, making it suitable for regions with long high-temperature seasons. The asphalt pavement can maintain good adhesion and flexibility, avoiding issues such as softening, flowing, or deformation in high-temperature working environments. When the magnesium hydroxide flame retardant concentration is 1%, the low-temperature performance of the corn stalk fiber-asphalt mastic is best, making it ideal for areas with long low-temperature seasons. In cold environments, the asphalt pavement can still maintain sufficient elasticity and toughness, preventing cracking or damage caused by sudden temperature drops and effectively reducing road damage due to low temperatures. Selecting the appropriate concentration of magnesium hydroxide flame retardant based on the actual usage scenarios of asphalt roads can help extend the service life of the constructed pavements.

Looking ahead, future environmental requirements will play a significant role in the development of the asphalt fiber industry. With increasing environmental awareness and the improvement of environmental regulations, the asphalt fiber industry is expected to move toward a greener and more environmentally friendly direction. Manufacturers will invest more in environmental protection, adopt cleaner production technologies, reduce energy consumption and emissions, and enhance the environmental performance of products. The use of magnesium hydroxide flame retardant treatment for corn stalk fibers aligns perfectly with these trends, and the resulting corn stalk fiber-asphalt mastic is expected to have even broader application prospects in the future.

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Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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