



Preparation and Properties of Carbon Nanofiber Modified Emulsified Asphalt Based on Ultrasonication and Surfactant and the Impact of SBR and NH₄Cl

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Carbon nanofiber (CNF) is a nanomaterial with unique mechanical properties, which can improve the properties of composite materials effectively. Research focusses on the impact of CNF on asphalt, asphalt binders, and mixtures. Traditional emulsified asphalt presents a limited performance at both high and low temperatures. Emulsified asphalt with a better performance, is therefore required in engineering. Referring to research on CNF-asphalt, CNF is considered to improve the performance of emulsified asphalt. In this study, a preparation method for CNF modified emulsified asphalt with styrene-butadiene rubber (SBR) was proposed. Ultrasonication and surfactant were utilized to disperse the CNFs in water. The optimum dispersion surfactant percentages and ultrasonic energy density to disperse CNFs were determined through ultraviolet-visible spectra (UV-vis spectra). The modified emulsified asphalt was produced using CNFs suspension with SBR as a modifier, and the properties of the residue, with different percentages of CNFs, were tested. Gel permeation chromatography (GPC) was performed to analyze the molecular size distribution. The results indicated that CNFs significantly improved high-temperature performance of the residue but decreased low-temperature properties. The addition of SBR not only perfected storage stability but also improved low-temperature performance by introducing more small molecules.

Keywords: carbon nanofiber, dispersion, SBR, residue properties, GPC

INTRODUCTION

In the past few years, nanomaterials, especially carbon nanotube (CNT) and carbon nanofiber (CNF), have presented advantages in composite materials and has attracted extensive investigation. There are many studies on the application of CNT/CNF in civil engineering because of their extraordinary properties (Pacheco-Torgal and Jalali, 2011). For example, carbon nanofibers exhibit excellent mechanical properties with Young's modulus from 25 to 200 GPa (Lawrence et al., 2008) and tensile strength of up to 12 GPa (Mordkovich, 2003).

For CNF reinforced composite materials, it is vital to obtain a uniform dispersion of CNFs in the matrix material to take advantage of the excellent mechanical properties of CNFs. Compared to CNF, there are more studies on the dispersion of CNT that indicate the effectiveness of surfactant

and ultrasonication to disperse CNTs in water uniformly (O'Connell et al., 2002; Strano et al., 2003). Considering a similar surface structure between CNT and CNF, the dispersion principle and method for these two materials should therefore also be similar.

There are many studies that focus on CNF reinforced cement. A. Yazdanbakhsh and Zoi S. Metaxa provided a method to produce carbon nanofiber modified cement with surfactant and ultrasonication (Yazdanbakhsh et al., 2009, 2010; Metaxa et al., 2013): first, CNFs were dispersed through ultrasonication in water containing surfactant, then the suspension was added to cement instead of water, and participated in hydration. Konsta-Gdoutos MS and Shah et al. researched CNF-cement composites further. They found significant improvements in flexural strength, Young's modulus, flexural toughness, and fracture toughness (Gdoutos et al., 2016). Zhu et al. (2018) researched the effect of the interfacial transition zone on Young's modulus of CNF reinforced cement concrete. Moreover, there is also research that indicates that the addition of CNFs significantly influences resistance to corrosion, electrical conductivity, and resistivity sensibility to structural damage of the nanocomposites (Galao et al., 2014; Konsta-Gdoutos and Aza, 2014; Konsta-Gdoutos et al., 2017).

The success of CNFs-cement composites has motivated studies on CNFs modified asphalt. According to the production of CNFs-cement materials, to prepare a CNF-asphalt composite material, the CNFs can be dispersed in the solvent and then be added to the asphalt. Khattak et al. (2013a) dispersed CNFs in kerosene or acetone by ultrasonication and then added the mixture into the asphalt. Kerosene or acetone eventually evaporated by heating (Khattak et al., 2013a,b). Asphalt mixtures with nanofibers also exhibit excellent mechanical properties like enhanced stiffness, higher dynamic modulus (Khattak et al., 2013a), and higher resistance to cracking under repeated loads (Khattak et al., 2013b). Furthermore, Khattak et al. (2013a) also studied the impact of CNFs modified asphalt on asphalt binder rheology through dynamic shear rheometer (DSR). They found that visco-elastic response and fatigue life were improved, which indicated higher resistance to rutting and fatigue (Khattak et al., 2012). Goh's research demonstrated that CNFs improved moisture susceptibility performance (decrease the moisture damage potential) of the mixture in most cases, and hot-mix asphalt mixture exhibited the greatest tensile strength with 0.75 wt% nanofibers (Goh et al., 2011). Despite these achievements, there are still issues. Generally, CNFs are easy to disperse in water with the assistance of surfactant and ultrasonication (get suspension), hence, there is a relatively uniform dispersion of nanofibers in cement, of which the suspension is a composition. By contrast, it is more difficult to disperse CNFs in asphalt directly, since the ultrasonication fails to work in high viscosity and hot liquid. Although agents like kerosene facilitate dispersion, they must ultimately be removed. Additionally, incomplete removal can affect performance.

Compared to the hot asphalt, considering that emulsified asphalt consists of asphalt and water, it is possible to produce emulsified asphalt with evenly dispersed CNFs by obtaining suspension first. Moreover, emulsified asphalt and relevant

materials such as cold recycled mixture and cement asphalt emulsion mortar are environmentally friendly, however, their poor properties limit further development (Ouyang et al., 2018, 2020). It is therefore necessary to improve the performance of cold recycled mixture and cement asphalt emulsion mortar, of which improving the properties of emulsified asphalt is essential.

This paper proposed a method to prepare CNF modified emulsified asphalt. According to present research, CNFs only enhance high temperature performance, so in this study, SBR was used as a modifier. Furthermore, the properties of the evaporation residue of the emulsified asphalt were investigated. Gel permeation chromatography (GPC) was performed to analyze the effect of SBR on the molecular size distribution and low-temperature properties.

PREPARATION OF CNFs MODIFIED EMULSIFIED ASPHALT

Materials

One 70# asphalt is selected as the base asphalt (**Table 1**). Carbon nanofibers are black powder (**Figure 1**) provided by Nanjing XFNANO Materials Tech Co., Ltd. All the CNFs are as-grown vapor grown carbon nanofibers (VGCNFs) produced at a high temperature with stable properties. The characteristic properties of CNFs are shown in **Table 2**. The surfactant is a kind of pale-yellow transparent liquid with a density of 0.98 g/ml provided by BASF Chemicals Co., Ltd (ID: GS8325).

TABLE 1 | Properties of asphalt.

Index	Result
Penetration (0.1 mm)	67
Softening Point (°C)	47.5
Ductility (10°C) (cm)	25
Rotational viscosity (135°C) (Pa·s)	0.48
PI (–)	–0.7



FIGURE 1 | CNFs used in this study.

TABLE 2 | Properties of carbon nanofibers (CNFs).

Commercial name	XFM60
Length (μm)	5–50
Diameter (nm)	200–600
Surface area (m^2/g)	>18
Purity (%)	>70
Density (g/cm^3)	2.1

Dispersion of CNFs

Emulsified asphalt consists of water and asphalt, so it is feasible to disperse CNFs in water and to then produce modified emulsified asphalt. The surfactant provided by BASF Chemicals Co., Ltd. was used in this study to improve the dispersion of CNFs and the stability of the suspension. VCX800 purchased from Sonics & Materials Co., Ltd. performed ultrasonication with a probe of a diameter of 25 mm. Ultrasonication caused a cavity (cavitation effect) and provided energy through a cavity burst to overcome the Van der Waals forces between nanofibers and disrupted CNFs aggregation. Surfactant attached on the surface of individual CNF enhanced the isolation. This process is exhibited in **Figure 2**. After ultrasonication, CNF aggregation was dispersed into individual CNF surrounded by surfactant molecules.

To obtain the optimum dispersion, ultrasonication energy density (KJ/L) and the amount of surfactant are significant factors (5), which should be determined in advance. In this study, the ultrasonic instrument was input energy with a constant power of 70 W. The energy density varied from 3000 KJ/L to 7000 KJ/L, and the weight ratio of CNFs to surfactant varied from 1:3 to 1:5.

UV-vis Spectra

Individual CNT is active in the UV-vis spectra and exhibits characteristic bands corresponding to additional absorption due

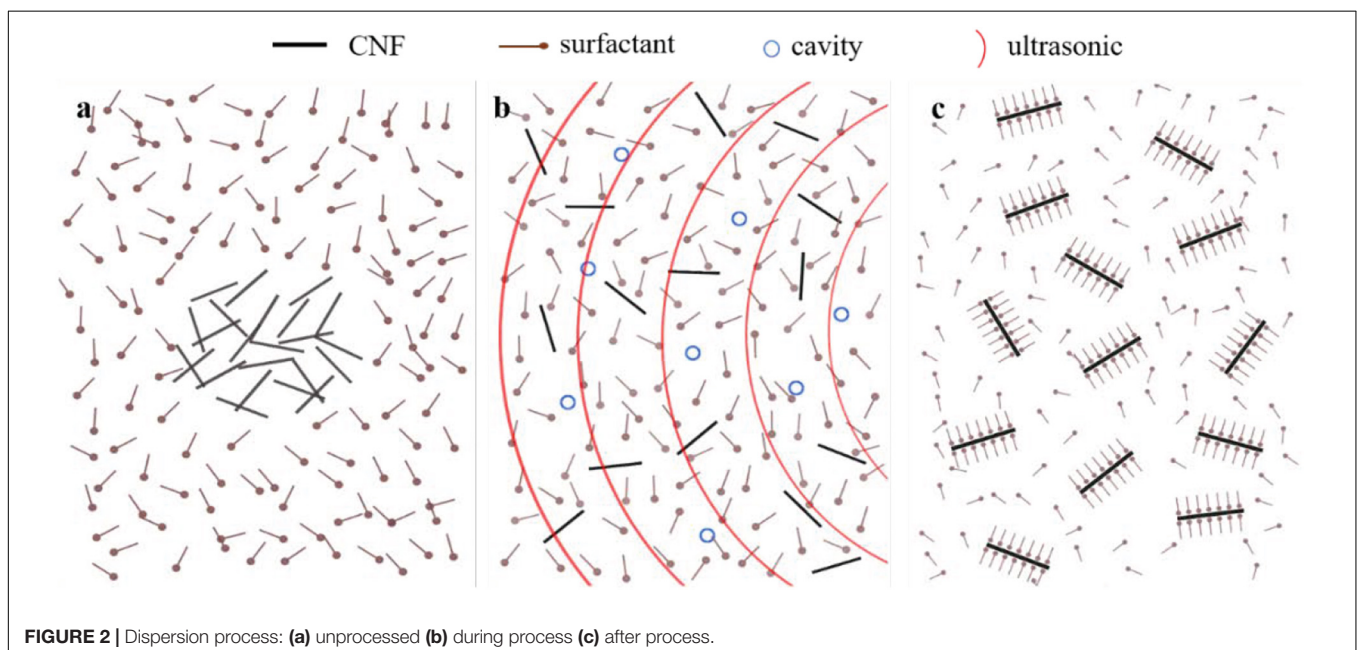
to van Hove singularities (Saito et al., 1992). Some researchers have obtained a strong peak of UV-vis spectra at the wavelength of approximately 250 nm (Chen et al., 1998; Grossiord et al., 2005; Yu et al., 2007; Cui et al., 2017). There is a certain similarity in the microstructure between CNF and CNT, so the UV-vis spectra can also be used to evaluate the dispersion of CNFs. The relationship between absorbance and solution concentration can be determined by Lambert-Beer law as follows (Formula 1), where A is the absorbance; T is the transmissivity; K is the molar absorptivity; b is the thickness of the absorption layer (cm); c is the concentration of light-absorbing substances (mol/L). A higher peak value at a particular wavelength indicates a higher concentration of individual CNF in suspension, presenting a better dispersion state.

$$A = \lg\left(\frac{1}{T}\right) = Kbc \quad (1)$$

There is a positive correlation between absorbance and solute concentration only when absorbance ranges from 0.2 to 0.8, according to the Lambert-Beer law. The suspension was therefore diluted by 50 times its volume before measuring to control the absorbance (**Figure 3**).

UV-vis spectra of all suspension with different energy density and surfactant content were performed with a sweep of wavelength from 200 to 500 nm. The results are demonstrated in **Figure 4**. Absorbance all appears within the expected range (0.2–0.8), so the results are valid.

There is an additional absorption peak for each group at a specific wavelength of 246 nm. The highest peak in **Figure 4** indicates that the optimal dispersion method is to disperse CNFs using ultrasonication at an energy density of 5000 KJ/L, accompanied by the addition of surfactant at a weight ratio of 1:5 to CNFs. It is noticeable that ultrasonication energy



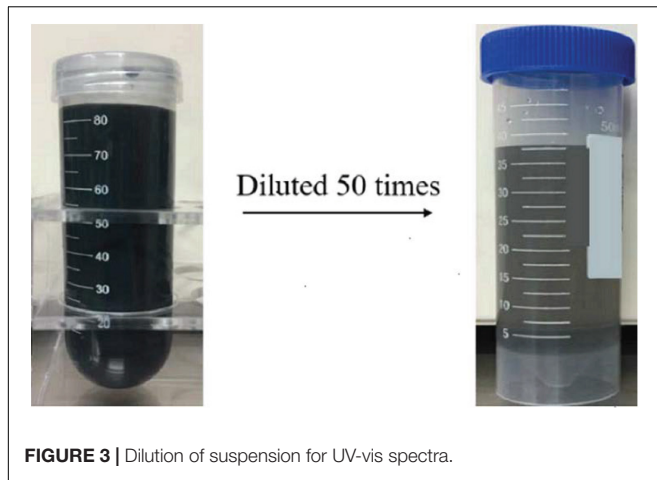


FIGURE 3 | Dilution of suspension for UV-vis spectra.

density and surfactant both impact disaggregation significantly. With the same surfactant content, the peak value of absorbance increases with the increase of energy density from 3000 to 5000 KJ/L and then decreases at a higher energy density. Similarly, under a constant energy density, more surfactant (weight ratio of CNFs to surfactant varies from 1:3 to 1:5) first

contributes to a higher peak, then the absorbance decreases at the ratio of 1:6.

Preparation of Emulsion Containing CNFs and Emulsification

Colloidal mill MD-1 from Jiaying Mide Machinery Co., Ltd. was utilized. In this study, the mass ratio of asphalt to emulsion (water and emulsifier) was 65:35. The emulsifier was SBT-50, provided by Westvaco Corporation. In this research, the emulsifier accounted for 3.9% of the mass of emulsified asphalt.

Carbon nanofibers content was determined by weight ratio to asphalt (0.1, 0.3, 0.5, and 1.0 wt%). First, CNFs were dispersed according to the optimal method above. The emulsifier was then dissolved into the suspension to obtain an emulsion. Hydrochloric acid was added to adjust pH value ($\text{pH} \approx 2$). Before production, the mill was heated, then materials were added to the colloid mill in this order: emulsion at 70°C was added and circulated for about half a minute, then hot asphalt at $135\text{--}145^\circ\text{C}$ was added with a constant speed to ensure uniform mix. For example, assuming the mass of emulsified asphalt is M , and the CNFs content is 0.1 wt%, the whole process is demonstrated in **Figure 5**.

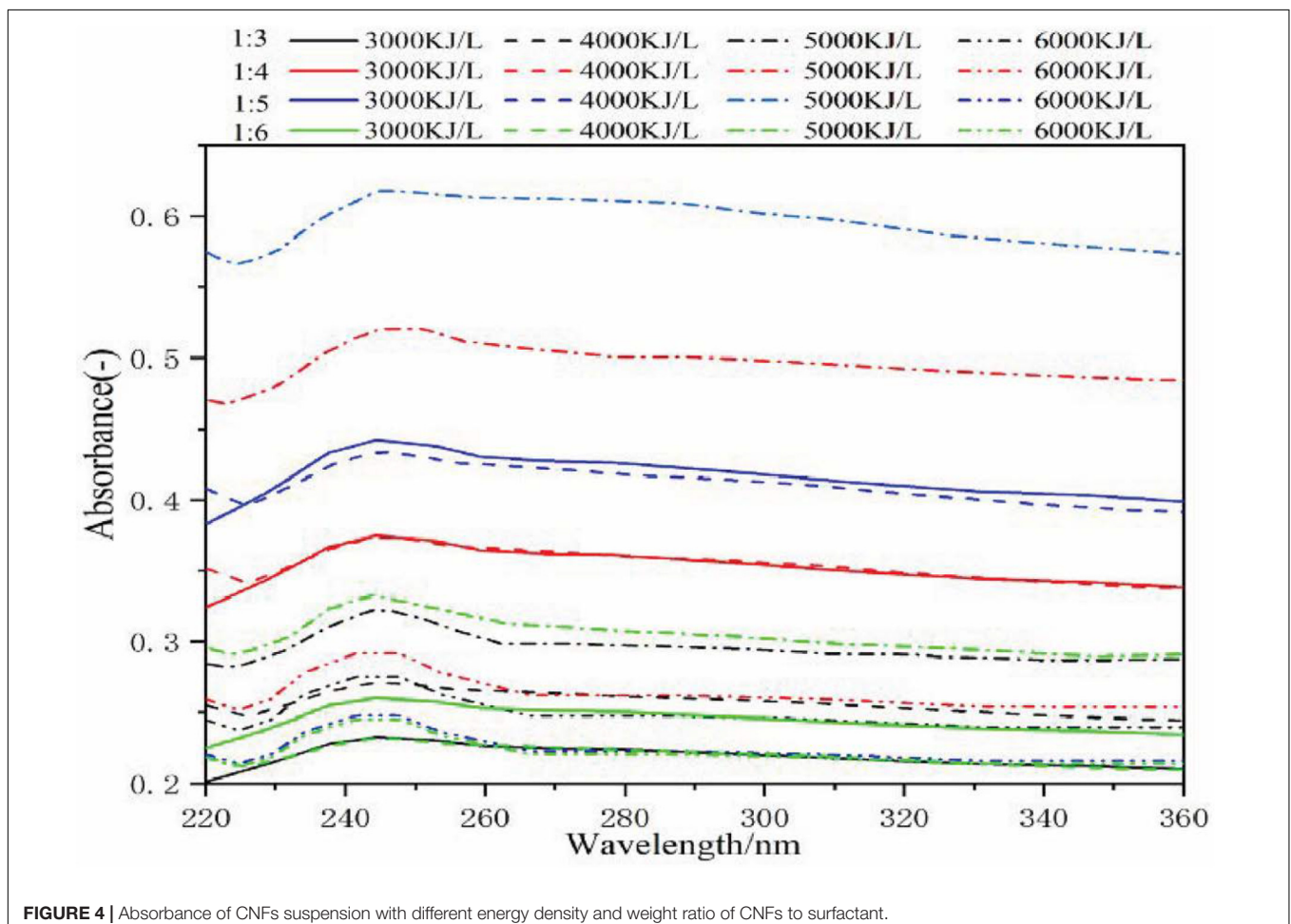


FIGURE 4 | Absorbance of CNFs suspension with different energy density and weight ratio of CNFs to surfactant.

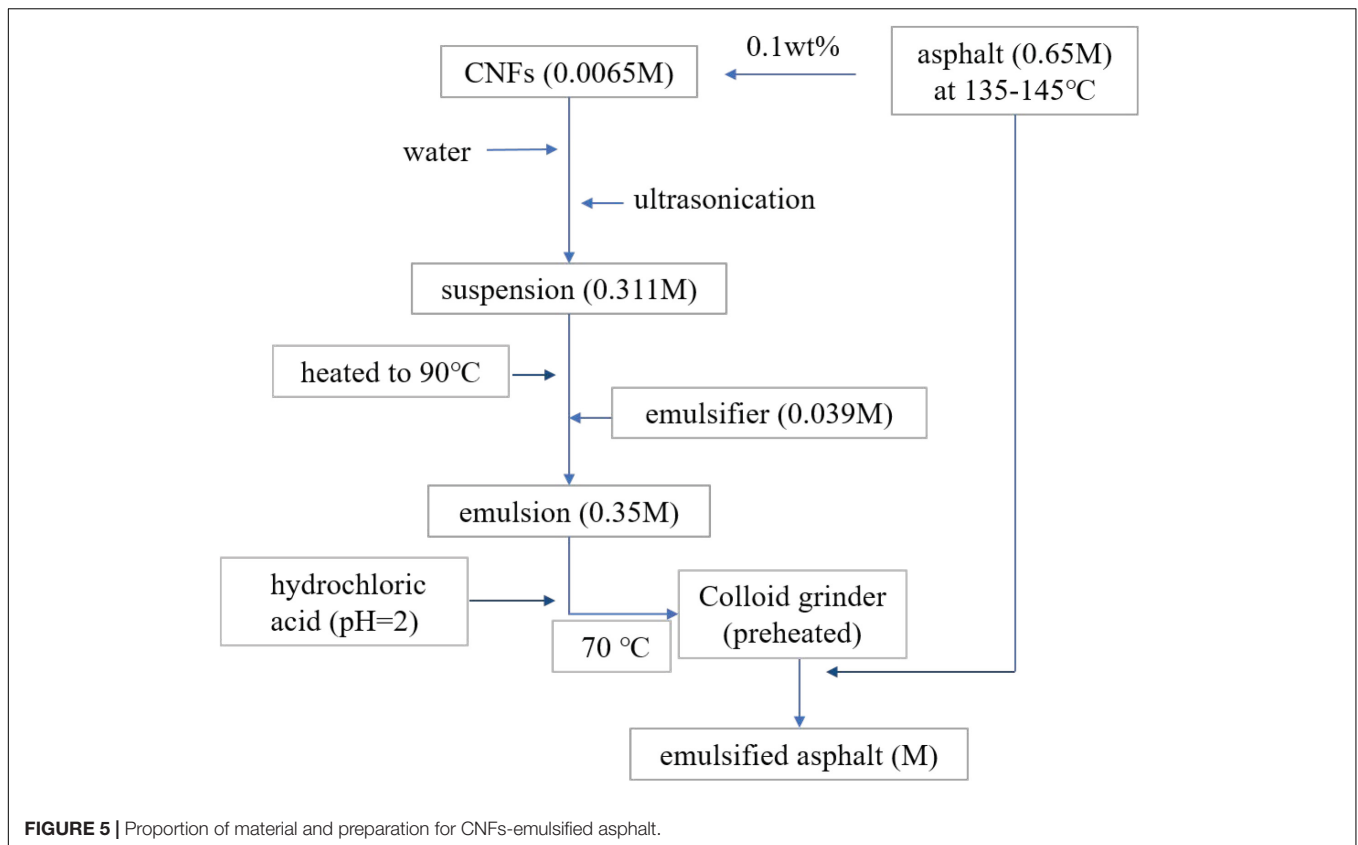


TABLE 3 | Experimental group setting (0.1 wt% CNFs for example).

ID	CNFs content (wt%)	NH ₄ Cl content (wt%)	SBR content (wt%)
Control	–	–	–
0.1C	0.1	–	–
0.1C1N	0.1	1.0	–
0.1C3N	0.1	3.0	–
0.1C1N1S	0.1	1.0	1.0
0.1C3N1S	0.1	3.0	1.0

After production, ammonium chloride (NH₄Cl) was added as a stabilizer. NH₄Cl content varied as 0, 1, and 3 wt% (weight ratio to asphalt). 1 wt% SBR latex (weight ratio to emulsified asphalt) was added as a modifier. **Table 3** shows the ID of groups with 0.1 wt% CNFs. The ID of groups with 0.3, 0.5, and 1.0 wt% CNFs are similar.

EXPERIMENTS

Storage Stability

Storage stability (1 day) at room temperature was investigated to estimate the feasibility of this preparation method. Emulsified asphalt was settled in the storage stability tube for 1 day, then the asphalt content of the 50 ml sample from the top and the 50 ml sample from the bottom were calculated, respectively. Their difference presents storage stability. In most situations, the

emulsified asphalt is used immediately after production, so the stability of 1 day is enough to prove engineering suitability.

Basic Properties of the Modified Emulsified Asphalt

In this study, to investigate the influence of CNFs and SBR, traditional experiments were performed. The emulsified asphalt was heated to remove moisture primarily. In this process, the temperature slowly increased and was below 180°C to avoid asphalt aging. Then the penetration (25°C), softening point, rotational viscosity (135°C), and ductility (5°C) were tested according to experiment specifications. Each experiment was performed three times, and the average was calculated as the final result.

Gel Permeation Chromatography (GPC) Test

Emulsified asphalt consists of components with various molecular weights, that significantly influence the properties of asphalt. In this study, the GPC test was performed to analyze the molecular weight distribution and to investigate the impact on properties. Tetrahydrofuran (THF) was selected as the solvent to dissolve the 20 mg emulsified asphalt sample. A combination of three columns was used for separating components with various molecular weights. The solution passed through the columns at a flow rate of 1 mL/min.

RESULTS AND DISCUSSION

Storage Stability and Properties

Experiment results of modified emulsified asphalt with 0.1 wt% CNFs are exhibited in **Table 4**.

Negative influence on stability is introduced by CNFs: the sample of 0.1 C presents unqualified stability that is much more than the specification requirements (<1%). However, NH₄Cl significantly improves storage stability. After adding the stabilizer, the CNFs modified emulsified asphalt exhibits excellent stability (less than 1%). The ability of group 0.1C1N1S is almost the same as group 0.1C3N1S, which indicates that the 1.0wt%

stabilizer is economical. It can be thus be concluded that SBR seldomly impacts stability.

Compared to the control group, the addition of CNF improves the high-temperature properties of the residue by an increase in the softening point and rotational viscosity and a decrease in penetration. Nevertheless, on the other hand, CNFs also worsen performance under low temperatures. There is no ductility value for groups 0.1C, 0.1C1N, and 0.1C3N because specimens fractured immediately under tension at 5°C. This undesirable impact can be changed by SBR; samples with SBR show significantly higher ductility, which presents better low-temperature performance. Further, SBR also slightly improves high-temperature properties.

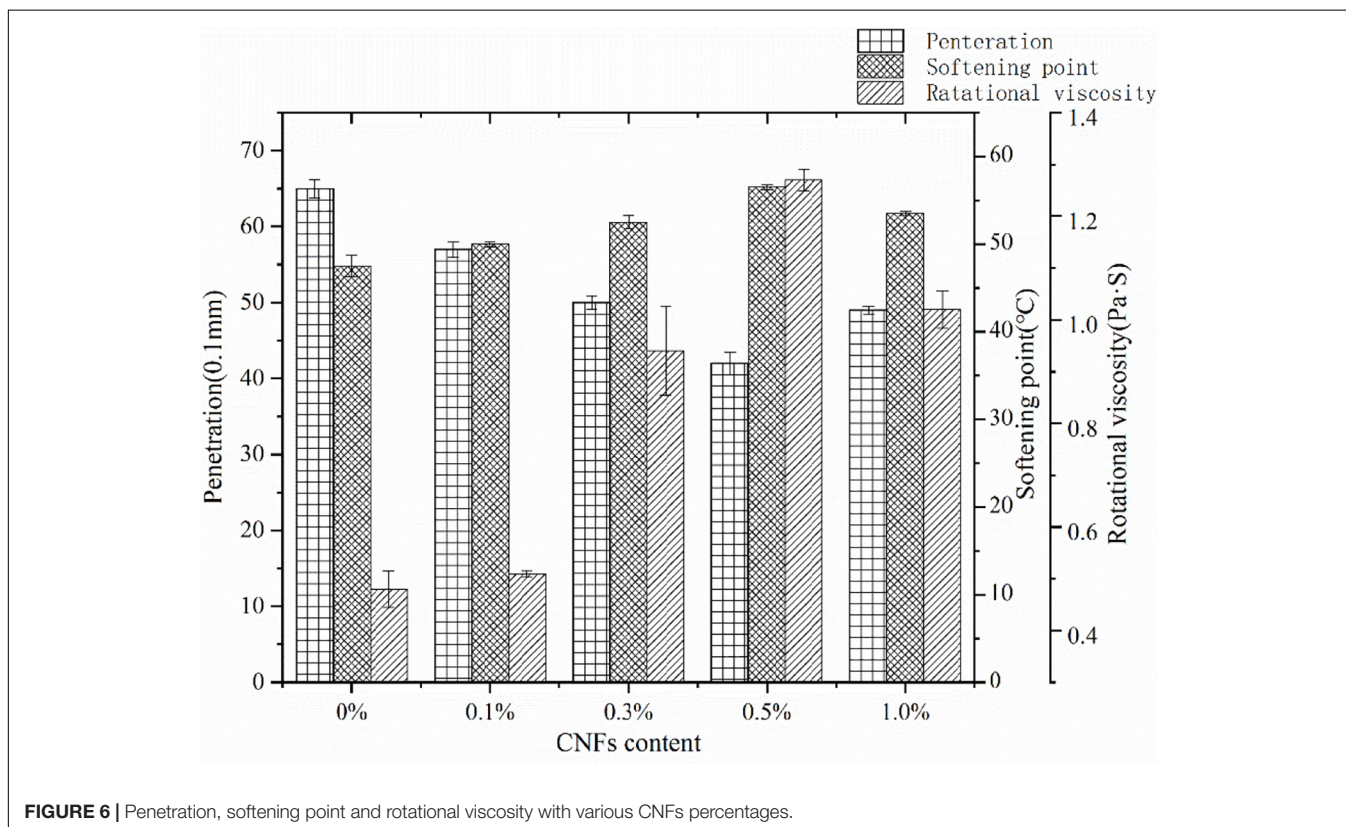
TABLE 4 | Properties and storage stability of modified emulsified asphalt (0.1 wt% CNFs).

ID	Properties				Storage stability (%)
	Penetration/0.1mm	Softening point/°C	Rotational Viscosity/Pa·s	Ductility/cm	
Control	65	47.5	0.48	5.0	0.5
0.1C	57	50.0	0.51	–	3.2
0.1C1N	56	50.5	0.50	–	0.6
0.1C3N	57	50.0	0.52	–	0.5
0.1C1N1S	52	52	0.55	9.5	0.6
0.1C3N1S	51	51.5	0.55	9.9	0.5

Effect of CNFs Content

To investigate the effect of CNFs content on the properties of the residue, samples with 0.3, 0.5, and 1.0wt% CNFs underwent the same experiments. The results are shown in **Figure 6**.

It can be seen from **Figure 6** that with the increase of CNFs percentages from 0 to 0.5wt%, the penetration of residual asphalt decreased remarkably, while the softening point and rotational viscosity increased, which indicates an improvement in temperature performance. However, a further increase of CNFs percentages (to 1.0wt%) induces a higher penetration value, lower softening point, and lower rotational viscosity than those of 0.5wt%. Compared to the control group, the residue asphalt with 0.5wt% CNFs exhibits the best high-temperature performance, with penetration decreasing by 22.4%,



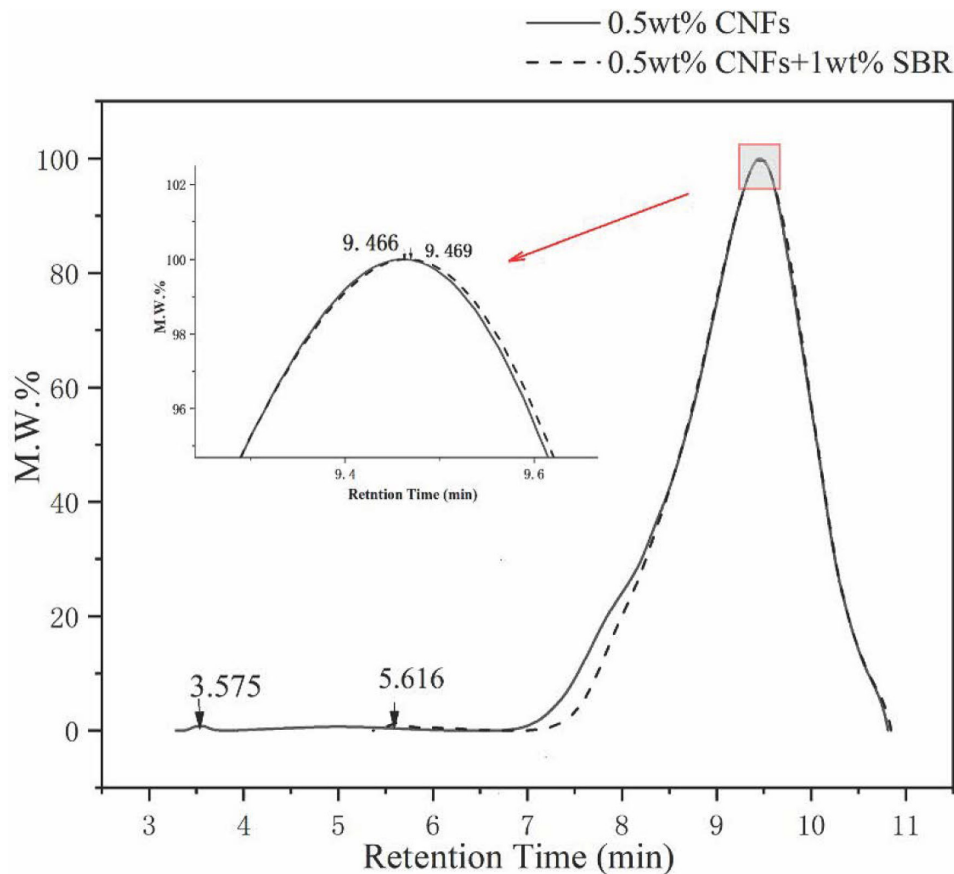


FIGURE 7 | GPC original results of modified emulsified asphalt.

the softening point increasing by 18.9%, and the rotational viscosity increasing by 164.6%.

This phenomenon may be explained by the network of CNFs. Individual CNF interconnects to form a micro-network to improve high-temperature properties. More CNFs facilitate and strengthen the network structure, which stiffens the asphalt and results in better high-temperature performance. However, excessive CNFs may interfere with network formation, and the high-temperature properties worsen to some extent.

GPC Results and the Effect of SBR

Gel permeation chromatography results of the emulsified asphalt with 0.5wt% CNFs were selected to analyze the particle size distribution and the effect of SBR. **Figure 7** shows the initial results of GPC, and relevant data are presented in **Table 5**.

In the GPC test, smaller molecules stay in columns for longer, so the peak appears later. **Figure 7** shows that before adding SBR, two peaks appear at the time of 3.575 and 9.466 min, respectively. The addition of SBR causes two peaks to shift to the right, appearing at 5.616 and 9.469 min, which indicates the proportion of smaller molecule increases.

Table 5 exhibits three primary data from GPC: Mn and Mw are average molecular weight in terms of number and

TABLE 5 | GPC data of samples with and without SBR.

Data	0.5 wt% CNF	0.5 wt% CNFs + 1 wt% SBR
Number Average Molecular Weight (Mn)	730	698
Weight Average Molecular Weight (Mw)	15469	2833
Mw/Mn	21.18	4.06

weight statistics, respectively, and their ratio Mw/Mn presents the distribution width of molecules. The addition of SBR reduces Mn and Mw significantly, which means an increase in the proportion of small molecules. Moreover, the Mw/Mn value of the sample with SBR is far less than that without SBR, meaning a more concentrated distribution. It can be concluded that after adding SBR to the emulsified asphalt, more molecules are small molecules that distribute in a smaller range.

This conclusion is consistent with the results of ductility and storage stability. More small and flexible molecules enhance the elasticity of the residue asphalt, so the ductility under low temperature increases. Since smaller molecules settle more slowly, the stability is thus improved. On the other hand, even though SBR enhances the performance at a

low temperature, it seldomly influences the high temperature properties like penetration and the softening point, because the high temperature performance strongly depends on the network of CNFs but the SBR fail to interfere with the network.

CONCLUSION

A method to produce CNFs modified emulsified asphalt with NH_4Cl and SBR was proposed based on ultrasonication. The performance of CNFs modified emulsified asphalt was then evaluated. A GPC test was performed to investigate the effect of SBR on molecule distribution. The following conclusions were obtained:

- (1) A combination of ultrasonication and surfactant is effective to disperse CNFs. To achieve uniform dispersion, the optimum weight ratio of surfactant to CNFs is 1:5, and the optimum energy density is 5000KJ/L.
- (2) A method to produce CNF modified emulsified asphalt was proposed by dispersing CNFs in water with surfactant and ultrasonication in advance. The emulsifier was then dissolved into the suspension. Hot asphalt and emulsion were mixed in a colloidal mill to produce modified emulsified asphalt.
- (3) Storage stability of CNF modified emulsified asphalt cannot meet the requirements, but the addition of NH_4Cl improves storage stability effectively, and SBR will not influence the stability.
- (4) The addition of CNFs increases the high-temperature performance and decreases the low-temperature properties

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of the residue. Residue with a CNFs percentage of 0.5 wt% exhibit the best high temperature performance (penetration decreased by 22.4%, softening point increased by 18.9%, and rotational viscosity increased by 164.6%).

- (5) GPC results indicate that SBR improves low-temperature ductility of residue asphalt significantly by increasing the proportion of small molecules.

DATA AVAILABILITY STATEMENT

All datasets generated for this study are included in the article/supplementary material.

AUTHOR CONTRIBUTIONS

JF, FL, and XZ conceived the presented study idea. XL and YA performed the experiments and collected the data. XL wrote the manuscript with support from FL. All authors discussed the results and contributed to the final manuscript.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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