Study on Preparation and Properties of Polyurethane-Epoxy Composite Emulsion Modified Emulsified Asphalt

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Abstract. In this study, epoxy resin modified waterborne polyurethane (EWPU) modified emulsified asphalt was successfully prepared by using self-made EWPU emulsion. Firstly, the optimal dosage range of EWPU was determined based on the evaporation residue content, remaining amount on sieve and storage stability of the modified emulsified asphalt. Secondly, the curing system of EWPU modified emulsified asphalt was investigated, and the optimal ratio of epoxy resin to curing agent and curing time were determined through mechanical property testing. Finally, the conventional properties of EWPU modified emulsified asphalt were characterized by using three indicators: penetration, softening point, and ductility. The microstructure and thermal properties were evaluated using fluorescence microscopy, infrared spectroscopy (IR), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). The results showed that the content of EWPU modifiers has a significant impact on the distribution of EWPU in emulsified asphalt. When the content of EWPU is changed from 5% to 20%, the EWPU modified asphalt emulsion has good storage stability. The mechanical performance test shows that the best performance is achieved when the ratio of epoxy resin to curing agent is 1:1 and the curing time is 24 hours. EWPU has a dual effect of physical and chemical modification in the process of emulsified asphalt modification. In addition, EWPU modification improves the thermal stability of emulsified asphalt.

Keywords: waterborne polyurethane; epoxy resin; emulsified asphalt; asphalt performance

1 Introduction

Emulsified asphalt, a dispersed liquid capable of maintaining fluidity at ambient temperatures, offers the advantage of being mixed, laid, and sprayed without necessitating elevated temperatures. Compared with hot asphalt, emulsified asphalt has significant advantages in terms of performance enhancement, environmental protection,

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energy conservation, and personnel health and safety. The performance of emulsified asphalt primarily relies on the technical properties of the residue after evaporation [1]. However, unmodified emulsified asphalt fails to meet the road construction requirements. Therefore, enhancing its pavement performance holds paramount importance. Currently, the primary approach for improving the performance of emulsified asphalt involves incorporating polymers. The modifiers commonly employed for preparing modified emulsified asphalt primarily include styrene-butadiene-styrene block copolymer (SBS), styrene-butadiene rubber (SBR), polyurethane (PU), and epoxy resin (EP). SBS modified emulsified asphalt has high mechanical strength, excellent resistance to low-temperature cracking, and superior stability under high temperatures. However, it also possesses some drawbacks such as increased viscosity, difficulties in emulsification, poor storage stability, and the need for specific equipment requirements for emulsification [2-4]. When SBR latex is used as modifier, the low temperature cracking resistance, elastic properties and viscosity of SBR latex modified emulsified asphalt are greatly improved. However, the high temperature performance of SBR modified emulsified asphalt is poor [5,6]. Epoxy resin (EP) has the advantages of chemical resistance, hydrolysis resistance, high mechanical strength and good thermal stability. However, epoxy resin modified emulsified asphalt needs to be used in combination with a curing agent. The disadvantages of epoxy resin modified emulsified asphalt are the low toughness, insufficient low-temperature resistance of its residues, and also its high cost.

Waterborne polyurethane (WPU) refers to a new type of environmentally-friendly material that involves the dispersion of polyurethane (PU) in water, which is completely consistent with the development concept of low-carbon environmental protection [7,8]. In recent years, research on WPU modified emulsified asphalt has received much attention. Wang [9] et al. prepared WPU modified emulsified asphalt as a waterproof coating. When the WPU reaches 1/4 of the quality of asphalt, the bonding strength is $\geq$ 0.18 MPa and the tensile strength is $\geq$ 0.68 MPa, which meets the requirements of waterproof coating. Cui et al. [10] studied the performance of WPU modified emulsified asphalt and its performance in the micro-surface, and mainly tested the bond strength and aging resistance of the binder. Compared with waterborne epoxy resin and WPU acrylic modified binder, the tensile strength of WPU modified binder after ultraviolet aging treatment was the highest, indicating that WPU modified emulsified asphalt has better aging resistance and weather resistance. Moreover, it was found that When WPU content is 50%, the tensile strength of the binder at 25 °C is increased by 300%, and the rutting depth of mixture is reduced to 1.06 mm.

Although WPU material exhibits excellent performance, it also possesses certain limitations, including poor water resistance, poor high temperature stability, and weak mechanical properties [11,12]. To address these drawbacks and enhance the overall performance of PU while expanding its application scope, further modifications are necessary. Epoxy resin contains a large number of epoxy groups, hydroxyl groups, and benzene rings. The presence of these structures gives epoxy resin unique advantages in terms of mechanical properties, thermal stability, corrosion resistance, and hydrolysis resistance. Additionally, the hydroxyl group in the molecular chain allows it to directly participate in the synthesis reaction of WPU and connect to the molecular chain segment of PU, thereby improving its mechanical properties and water resistance.

To obtain a bridge deck waterproofing bonding material with high bonding strength and flexibility, Yang et al. [13] prepared waterborne epoxy resin-PU emulsified asphalt that can be cured quickly at room temperature. The results showed that WPU can effectively improve the flexibility of high content waterborne epoxy resin emulsified asphalt, leading to the modified emulsified asphalt has excellent high and low temperature bonding properties, impact resistance and water stability, avoiding pavement defects such as pushing and shoving, and overcrowding, etc., which can effectively reduce the cost of maintenance, and can improve the quality of the bridge pavement structure and prolong its service life. He et al. prepared polyurethane/epoxy modified asphalt for steel bridge deck pavement, and the results showed that polyurethane/epoxy modified asphalt could effectively improve the road performance of epoxy asphalt [14]. Yang et al. [15] studied the tensile strength and flexibility of waterborne epoxy resin, WPU and their composite modified emulsified asphalt binders. The results show that waterborne epoxy can improve the high-temperature tensile strength of modified emulsified asphalt, while WPU can optimize the low-temperature elongation at break of modified emulsified asphalt and improve its flexibility.

Due to the poor compatibility between PU and asphalt, the modification effect of a single WPU modifier on asphalt is not ideal. In addition, the toughness of water-borne epoxy modified asphalt is poor. To overcome the above problems, this work uses self-made epoxy modified waterborne polyurethane (EWPU) composite emulsion to modify emulsified asphalt. The optimal dosage, curing system, and modification technology for EWPU composite emulsion modified emulsified asphalt were studied. The conventional properties of the modified asphalt were evaluated through testing the three key parameters: penetration, softening point, and ductility. Furthermore, fluorescence microscopy (FM), TGA and DSC tests were conducted to investigate the thermodynamic stability and microscopic distribution of EWPU modifiers. The research results lay the foundation for the application of EWPU as a new type of emulsified asphalt modifier.

2 Materials and Methods

2.1 Raw Material

70# A grade asphalt, Qilu Petrochemical Co, LTD (China); Epoxy modified WPU (EWPU) composite emulsion, Self-made in our lab; asphalt emulsifier, Jiangxi Yitong material Co., LTD (China); Curing agent, Guangdong Heima new material technology Co., LTD (China); Hydrochloric acid, AR, Sinopharm Chemical Reagent Co., Ltd (China).

2.2 Preparation of EWPU

In this study, the EWPU composite emulsion with the epoxy resin content of 5%, 7% and 10% synthesized by ourselves was used as modifier: EWPU5, EWPU7 and EWPU10. The detailed synthesis process, please see reference [16].

2.3 Preparation of Emulsified Asphalt

Emulsified asphalt was prepared using colloid grinding method with an oil-water ratio of 6:4. The emulsifier was dissolved by stirring in water at 60 °C to obtain soap solution (the amount of emulsifier was 2%). After the pH value of the soap lye was adjusted to 2~3 with hydrochloric acid, and it was added to the colloid mill. Then the asphalt heated to 135 °C is slowly added into the colloid mill for high-speed shear emulsification. The emulsified asphalt product is obtained after continuous emulsification for 3 min.

2.4 Preparation of EWPU Modified Emulsified Asphalt

The emulsified asphalt and EWPU emulsion were weighed according to a specific ratio. Under low-speed mixing conditions, the EWPU emulsion was gradually added to the emulsified asphalt and stirred at low speed for 10 minutes. Epoxy curing agent was then slowly incorporated into the EWPU modified emulsified asphalt under low-speed stirring conditions for another 15 minutes, resulting in the formation of EWPU modified emulsified asphalt. The dosage ratio of EWPU-modified emulsified asphalt was calculated based on solid content.

2.5 Performance Test of EWPU Modified Emulsified Asphalt

Evaporation Residual Amount of EWPU Modified Emulsified Asphalt, Remaining Amount on Sieve, Storage Stability.

Evaporation residual amount of EWPU modified emulsified asphalt, Remaining amount on sieve, storage stability were tested according to the test method T0651-1993, T0652-1993 and T0655-1993 in JTG E20-2011.

Determination of the Ratio of Epoxy Resin to Curing Agent in EWPU Modified Emulsified Asphalt.

Taking the PU epoxy composite emulsion EWPU5 with epoxy resin content 5% as an example, modified emulsified asphalts with modifier addition amount of 15% and 20% were prepared. The ratio of epoxy resin and curing agent was 1:0.8, 1:0.9, 1:1, 1:1.1 and 1:1.2, respectively. After the sample was fully cured in an oven at 60 °C, the tensile strength and elongation at break of the modified emulsified asphalt were tested. The test was performed with a universal testing machine MTS-CMT5504 at room temperature, with a tensile speed of 500 mm/min.

Influence of Curing Time on Mechanical Properties of EWPU Modified Emulsified Asphalt Film.

Taking the PU epoxy composite emulsion EWPU5 modified with 5% epoxy resin content as an example, the modified emulsified asphalt with 15% and 20% modifier addition amounts was prepared, respectively. After the samples were cured in oven at 60 °C for 6 h, 12 h, 18 h, 24 h and 30 h respectively, the tensile strength and elongation at break of modified emulsified asphalt samples were tested. The test was performed with a universal testing machine MTS-CMT5504 at room temperature, with a tensile speed of 500 mm/min.

Conventional Performance Tests of EWPU Modified Emulsified Asphalt.

After heating the prepared EWPU modified emulsified asphalt material to a completely dehydrated state at different temperature, and the residues were used to prepare routine performance test samples.

Asphalt softening point, asphalt penetration (25 °C), asphalt ductility (5 °C) were measured according to the test method of T0606-2011, T0604-2011 and T0605-2011 in JTG E20-2011.

Fluorescence Microscope (FM) Observation of EWPU Modified Emulsified Asphalt.

A small piece of evaporation residue of EWPU modified emulsified asphalt was placed on a slide, then covered with a cover glass and heated. The heating temperature was not more than 120 °C, so that the evaporation residue of modified emulsified asphalt was evenly placed on the slide. Finally, the sample was observed under a fluorescence microscope (Leica, Germany).

Fourier Transform Infrared (FTIR) Spectroscopy Analysis of EWPU Modified Emulsified Asphalt.

The evaporated residue samples of emulsified asphalt obtained at different temperatures was dissolved into tetrahydrofuran. FTIR spectra were conducted using KBr pellet method by a FTIR spectrometer VERTEX 70 (Bruker Corporation).

Thermogravimetric Analysis (TGA) of EWPU Modified Emulsified Asphalt.

The TGA2 manufactured by Mettler Toledo International Trade Co. Ltd was used for thermogravimetric analysis. The thermal weight loss of the film was measured under N2 atmosphere by using about 5-10 mg of residue film with a heating rate of 10 °C /min and a temperature range of 30 to 600 °C.

Differential Scanning Calorimetry (DSC) of EWPU Modified Emulsified Asphalt.

DSC made by American TA Company was used. Aluminum pans containing 10-15 mg of sample were heated from -60 to 120 °C under nitrogen atmosphere (flow rate: 50 mL/min). The heating rate was 10 °C/min.

3 Results and Discussions

3.1 Performances of EWPU Modified Emulsified Asphalts

According to the test method of JTG E20-2011, the performance of modified emulsified asphalt with different EWPU content was tested. The results are shown in Table 1.

Table 1. Performance test of EWPU modified emulsified asphalt.

EWPU content (%)		0	5	10	15	20	25	30
Evaporation residue content (%)		59.3	57.3	55.6	54.2	52.9	51.8	50.8
Remaining amount on sieve (%)		0.05	0.05	0.06	0.07	0.07	0.08	0.08
Storage stability (%)	1d	0.42	0.49	0.57	0.71	0.78	0.88	1.23
	5d	2.30	2.75	3.43	4.16	4.79	5.53	7.12

According to the technical specifications for highway asphalt pavement construction outlined in JTG F40-2004, the minimum requirement for the evaporation residue of emulsified asphalt used for spraying is 50%, and the residue on the sieve should not exceed 0.1% for road use. The stability of emulsified asphalt stored at room temperature for 1 day and 5 days should not exceed 1% and 5%, respectively. As shown in Table 2, the content of evaporated residue in modified emulsified asphalt meets the spraying requirements, and the residue on the sieve meets the road use standards. However, the increase in the amount of EWPU modifier leads to a gradual decrease in the storage stability of the modified emulsified asphalt. When the EWPU dosage is ≤ 20%, the storage stability for 1 day and 5 days meets the requirements, indicating that the EWPU and asphalt have good compatibility within this composition range. When the dosage of EWPU is 25%, the storage stability after 1 day meets the specifications, but it cannot meet the specifications after 5 days. When the dosage of EWPU reaches 30%, the storage stability for both 1day and 5 days cannot meet the requirements.

3.2 Determination of the Ratio of Epoxy Resin to Curing Agent in EWPU Modified Emulsified Asphalt

Taking the PU epoxy composite emulsion EWPU5 (with 5% epoxy resin content) as an example, the EWPU5 modified emulsified asphalts with 15% and 20% modifiers were prepared. The ratio of epoxy resin to curing agent was 1:0.8, 1:0.9, 1:1, 1:1.1 and 1:1.2 respectively. After curing in an oven at 60 °C, the tensile strength and elongation at break of the modified emulsified asphalts with different ratios of epoxy resin and curing agent were tested (Figure 1).

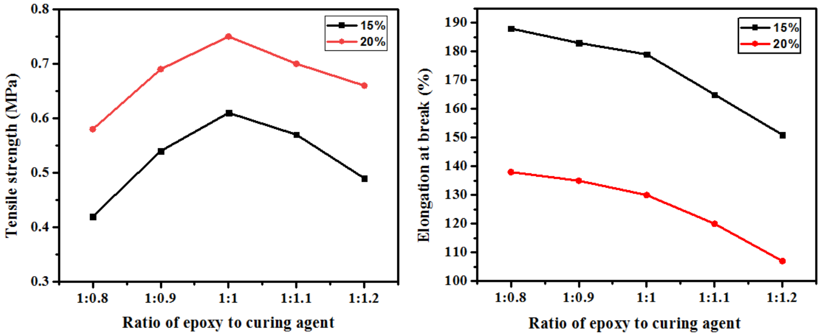

Fig. 1. Tensile strength and elongation at break of EWPU5 (the content of 15% and 20%) modified emulsified asphalts with different ratio of epoxy resin to curing agent.

The results demonstrate that the tensile strength of the modified emulsified asphalt increases first and then decreases with the increase of the proportion of the curing agent, while the elongation at break gradually diminishes. This phenomenon can be attributed to incomplete crosslinking when a small amount of curing agent is used, resulting in lower tensile strength. However, when the ratio of epoxy resin and curing agent reaches 1:1, both tensile strength and elongation at break reach their maximum values. Subsequently, an excessive amount of curing agent leads to decrease in molecular weight between crosslinking points in the cured product and hinders the formation of an ideal crosslinking network. Therefore, this reduction has a negative impact on the tensile strength and elongation at break of modified emulsified asphalt, while also causing waste of curing agents. Considering the overall application performance and economic factors, it is recommended to use the optimal ratio of epoxy resin to curing agent of 1:1 for the EWPU emulsified asphalt system.

3.3 Effect of Curing Time on Mechanical Properties of EWPU Modified Emulsified Asphalt

Taking the polyurethane epoxy composite emulsion EWPU5 modified (with 5% epoxy resin content) as an example, the modified emulsified asphalts with 15% and 20% modifiers were prepared. After the samples were cured at 60 °C for 6h, 12h, 18h, 24h and 30h respectively, the tensile strength and elongation at break of the EWPU modified emulsified asphalts were tested.

It is evident from Figures 2 that the tensile strength of modified asphalt increases while the elongation at break decreases with an increase in curing time. This can be attributed to continuous evaporation of water and gradual enhancement of degree of curing, leading to formation of an interactive network structure. Curing reaction reaches completion within 24 h, and the modified asphalt containing 20% EWPU modifier exhibits higher tensile strength and lower elongation at break compared to that containing 15% EWPU modifier.

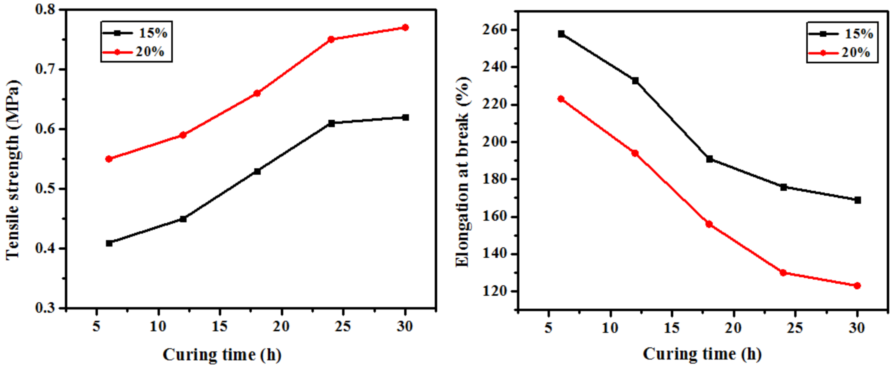

Fig. 2. Influence of curing time on tensile strength and elongation at break of the EWPU5 (the content of 15% and 20%) modified emulsified asphalt.

3.4 Conventional Performance of EWPU Modified Emulsified Asphalt

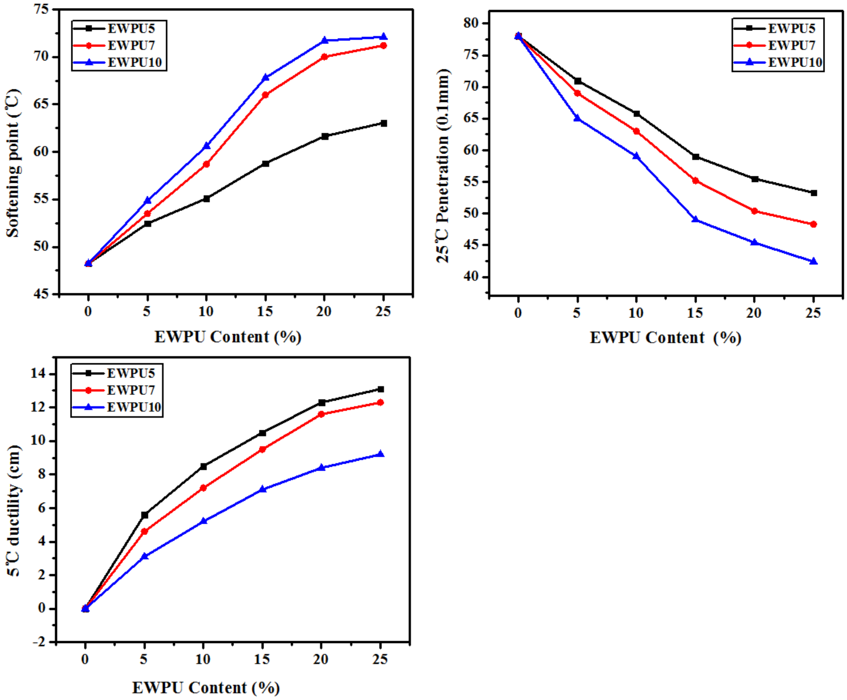

Fig. 3. Effects of types of EWPU modifiers and EWPU dosage on the Conventional properties of modified emulsified asphalts

EWPU modified emulsified asphalt was prepared by using EWPU5, EWPU7 and EWPU10 with epoxy resin content of 5%, 7% and 10% as modifiers. The influence of EWPU content on the conventional performance (softening point, penetration and ductility) of modified emulsified asphalt was measured, and the results are shown in Figure 3.

It can be seen from Figure 3a that with the increase of EWPU content, the softening point of modified emulsified asphalt gradually increases. When the content of EWPU is increased from 5% to 20%, the softening points increase significantly. When the EWPU content reaches 20% or more, the softening point rises slowly. The softening point of EWPU modified emulsified asphalt with different epoxy resin contents is significantly different. With the increase of epoxy resin content in EWPU, the softening point of emulsified asphalt shows an upward trend. The softening point of EWPU4 and EWPU5 modified asphalt can reach above 70 °C, indicating that the EWPU modifier has a significant impact on the high-temperature performance of emulsified asphalt. The addition of epoxy resin has a significant improvement on the high-temperature performance of emulsified asphalt.

From Figure 3b, it can be seen that as the EWPU content or the content of epoxy resin in EWPU increases, the penetration of modified emulsified asphalt gradually decreases, indicating that the addition of EWPU makes the asphalt binder thicker and harder.

The ductility of EWPU modified emulsified asphalt gradually increases with the increase of EWPU content, as shown in Figure 3c. It is worth noting that the increase slows down when the EWPU content reaches 20%. On the contrary, the increase in epoxy resin content in EWPU leads to a decrease in the ductility of emulsified asphalt. When the EWPU content is 20%, there is a significant difference of 57% between EWPU-5 and EWPU3. These findings highlight the substantial impact of EWPU modifier on the low temperature performance of emulsified asphalt, with epoxy addition resulting in increased brittleness and hardness within the binder. Optimal modification effects are observed at a EWPU5 and EWPU7 content range of ~20%.

3.5 FM Observation of EWPU Modified Emulsified Asphalt

Modified emulsified asphalt with different EWPU7 contents was prepared, and the distribution and structure of EWPU polymer modifiers in the emulsified asphalt were observed using a fluorescence microscope at a magnification of 100 times. The green fluorescence observed in the micrograph indicates the presence of EWPU modifier, while the black background represents asphalt.

It can be seen from Figure 4 that when the amount of modifier is 5% and 10%, the distribution of EWPU7 is sparse. When the amount of EWPU7 increases to 15% and 20%, the modification effect is obvious and the distribution of modifier is still relatively uniform. When the amount of modifier was further increased to 25% and 30%, aggregation and uneven distribution of EWPU7 were observed. Therefore, the optimal dosage of modifier is 15% to 20%.

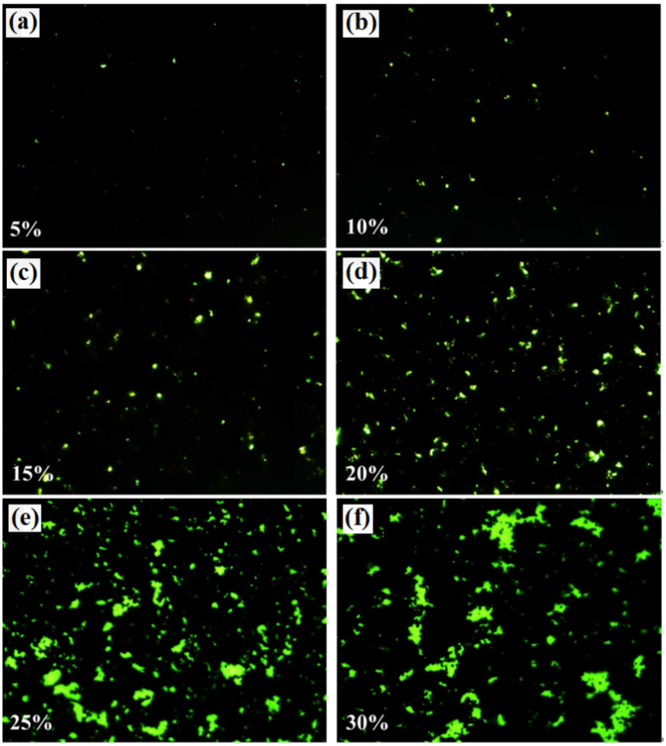

Fig. 4. FM images of EWPU7 modified emulsified asphalt. The EWPU7 content: (a) 5%, (b) 10%, (c) 15%, (d) 20%, (e) 25% and (f) 30%.

3.6 FTIR Analysis of EWPU Modified Emulsified Asphalt

To further analyze whether EWPU undergoes chemical changes during the modified emulsified asphalt process, FTIR tests were conducted on emulsified asphalt, EWPU modified emulsified asphalt and pure EWPU7 modifier (Figure 5). The absorption peak at 3340 cm-1 is the N-H stretching vibration peak of the carbamate group, the peak at 1535 cm-1 is the bending vibration absorption peak of N-H bond, and the peak at 1730 cm-1 is C=O characteristic absorption peak of the carbamate group. The above peak exist in curve b of Fig. 5, but not in curve a, indicating that the carbamate group only exists in the modified emulsified asphalt, but does not exist in the emulsified asphalt, which proves that EWPU is mixed into the emulsified asphalt. 910 cm-1 is the characteristic peak of epoxy group, and there is no corresponding peak in curve b. The characteristic peak of -OH at 3550 cm-1 is enhanced, indicating that after the addition of curing agent, all epoxy groups participate in the crosslinking reaction, forming a crosslinking structure that can improve the performance of emulsified asphalt. The above analysis indicates that EWPU has a dual effect of physical and chemical modification in the process of emulsified asphalt modification.

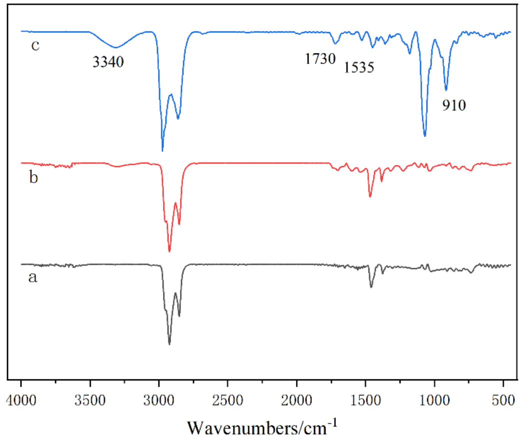

Fig. 5. FTIR spectra of emulsified asphalt (a), modified emulsified asphalt containing 20% EWPU7 (b), and pure EWPU7 modifier (c).

3.7 TGA Analysis of EWPU Modified Emulsified Asphalt.

The TG curves of emulsified asphalt and EWPU modified emulsified asphalt are presented in Figure 6, and the corresponding data are provided in Table 2. EWPU7 modification increased the temperatures of TB5, TB10 and TB 50 by 63, 60 and 10 °C, respectively. Furthermore, the residual content of the modified emulsified asphalt was found to be 2.5% higher than that of the unmodified counterpart, indicating that EWPU modification enhanced thermal stability of asphalt.

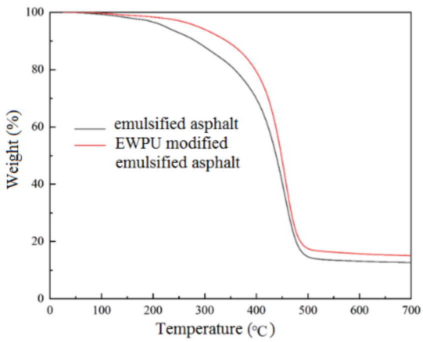

Fig. 6. TG curves of emulsified asphalt and EWPU modified emulsified asphalt (EWPU7 content of 20%).

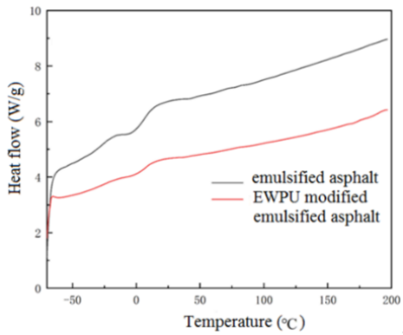

Fig. 7. DSC spectra of emulsified asphalt before and after EWPU modification

Table 2. TG analysis results of emulsified asphalt and EWPU modified emulsified asphalt.

Sample	Emulsified asphalt	EWPU modified emulsified asphalt
Tb5 (°C)	224	287
Tb10 (°C)	282	342
Tb50 (°C)	438	448
Residue content (%)	12.1	14.6

Note: Tb5, Tb10 and Tb50 are the corresponding temperatures when the weight loss of asphalt is 5%, 10% and 50%, respectively.

3.8 DSC Analysis of EWPU Modified Emulsified Asphalt.

The DSC spectra of emulsified asphalt and modified emulsified asphalt (EWPU7 content of 20%) are shown in Figure 7. The glass transition temperature (Tg) of emulsified asphalt is -26.3 °C, while the Tg of EWPU modified emulsified asphalt is -28.7 °C, indicating that addition of EWPU improves the low-temperature performance of emulsified asphalt. Compared with EWPU modified emulsified asphalt, unmodified emulsified asphalt has a smaller and flatter absorption peak, indicating that the performance of EWPU modified emulsified asphalt exhibits stronger stability.

4 Conclusion

(1) The EWPU modified emulsified asphalt was prepared successfully. The results for the evaporation residue, sieve residue, and storage stability of the EWPU modified asphalt indicate that all performance indexes meet the standard requirements when the content of EWPU modifier is ≤ 20%.

(2) The curing effect of epoxy resin was evaluated through tensile testing. The optimal ratio of epoxy resin to curing agent is 1:1, and the curing time at 60 °C is 24 h.

(3) EWPU significantly improves the conventional performance of emulsified asphalt. The increase in EWPU content leads to a gradual increase in softening point and ductility, while penetration shows a gradually decreasing trend, indicating that EWPU simultaneously improves the high-temperature and low-temperature performance of emulsified asphalt.

Moreover, different modifiers (EWPU5, EWPU7 and EWPU10) exhibit varying effects on the modification of emulsified asphalt. The higher the content of epoxy resin in the EWPU modifier, the higher the softening point, and the lower the penetration and ductility of the modified emulsified asphalt. This indicates that the addition of epoxy resin further improves high-temperature stability, but reduces low-temperature crack resistance. Overall, when the EWPU7 content is 20%, the comprehensive performance of modified emulsified asphalt is the best.

(4) The content of EWPU modifiers has a significant impact on the distribution of EWPU in emulsified asphalt. EWPU has a dual effect of physical and chemical modification in the process of emulsified asphalt modification. In addition, EWPU modification improves the thermal stability of emulsified asphalt.

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