

Study on Influencing Factors and Improvement Measures for Storage Stability of Tack-Coat Emulsified Asphalt

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Abstract: To address the issues of delamination, demulsification, and uneven spraying of tack-coat emulsified asphalt during storage and construction heating, this study employed a single-factor control method to systematically investigate the effects of emulsifier type and dosage, asphalt solids content, emulsification and soap-solution temperatures, stabilizer type and dosage, and soap-solution pH on storage stability. Stability was evaluated according to T0655 in JTG E20-2011, with cyclic heating used to simulate repeated construction heating. Long-term stability was assessed by solids content difference (SS) after 1 d, 5 d, 7 d of static storage and post-cyclic heating. Results show that Type A emulsifier performed best at an optimal dosage of 1%; solids content of 62% yielded maximum stability; optimal asphalt and soap-solution temperatures were 145~160 °C and 60~70 °C, respectively; HEMC exhibited the most significant stabilizing effect at 0.3% dosage; and soap-solution pH of 2.5~3.5 ensured interfacial film integrity and optimal storage stability.

Keywords: adhesive layer emulsified asphalt; storage stability; asphalt solid content; Soap pH

1. Introduction

Adhesive emulsified asphalt plays an important role in preventing interlayer slip and improving structural integrity by improving the interfacial bonding of pavement structural layers. However, during storage and transportation, adhesive emulsified asphalt often experiences stability issues such as settling, delamination, and premature demulsification, which affect the uniformity of spraying and the quality of film formation [1-3]. Previous studies have shown that increasing the amount of emulsifier and reducing the pH of soap solution can improve the storage stability to a certain extent. However, the current research is still mainly focused on the emulsifier system and acidity control, and the research on the specific asphalt solid content requirements of tack coat emulsified asphalt, the action mechanism of additives and the stability of lotion under repeated heating conditions is relatively insufficient [4-5]. In addition, complex working conditions such as repeated heating and long-term parking are commonly present on construction sites, and it is difficult to fully reflect their long-term stability solely through conventional static storage tests. Therefore, it is necessary to evaluate the long-term stability of the adhesive emulsified asphalt by combining cyclic heating.

Based on this, this study focused on the key factors affecting the storage stability of tack coat emulsified asphalt, systematically investigated the action rules of emulsifier type and dosage, asphalt solid content, asphalt and soap temperature, soap pH, and auxiliary type and dosage, and combined with T0655 storage stability test in JTG E20-2011, carried out cyclic heating to simulate complex construction conditions, and evaluated the long-term stability of lotion from both static and heating conditions. By comparing the significance of various factors and optimal conditions, a stabilization formula and process suitable for adhesive layer engineering are proposed, providing technical reference for its promotion and application.

2. Experimental materials and methods

2.1 Materials

AH-70 road petroleum asphalt, provided by Sinopec Jingmen Branch, with performance indicators shown in Table 1. A, B, C, and D types of asphalt emulsifiers are provided by Pingyuan Yugang Road Engineering Co., Ltd., Shanghai Longfu Road Maintenance Engineering Co., Ltd., Meide Weishi Weike Co., Ltd., and Shanghai Shijian Industrial Co., Ltd. respectively; The performance indicators of emulsifiers are shown in Table 2. HEC, CMC, and HEMC additives are all provided by Shandong Heda Group Co., Ltd; Its performance indicators are shown in Table 3; Acidic regulator hydrochloric acid, provided by McLean Biochemical Technology Co., Ltd; The experimental water is deionized water.

Table 1. Performance Parameters of AH-70 Matrix Asphalt

Metric	Indicator Requirements	Test result	Experimental method
Penetration depth (25°C, 100 g, 5 s) / (0.1 mm)	60~80	62.3	T0604
Ductility (5 cm/min, 15°C)/cm	≥100	≥100	T0605
softening point /(°C)	≥46	51.2	T0606

Table 2. Performance Parameters of Emulsifiers

Emulsifier Name	Ion Type	Demulsification speed	Surface	Active Ingredient
A	Cation	Fast-reacting, fast-setting type	White paste-like solid	alkyl quaternary ammonium salts
B	Cation	Fast-reacting, fast-setting type	Brown paste-like solid	Fatty amine quaternary ammonium salt
C	Cation	Fast-reacting, fast-setting type	Dark brown liquid	Lignosulfonate
D	Cation	Fast-reacting, fast-setting type	Dark brown viscous liquid	Complex of Quaternary Ammonium Salt and Calcium Chloride

Table 3. Performance Parameters of Various Additives

Adjuvant Name	Viscosity/ (mPa·s)	pH price (1% aqueous solution)	Moisture content / (%)	Surface	Active Ingredient
HEC	4500	6.0	≤6.0%	Light yellow powder	hydroxyethyl cellulose
CMC	4000	6.5	≤8.0%	White powder	carboxymethylcellulose sodium
HEMC	60000	5.5	≤6.0%	White particle	Hydroxyethyl methylcellulose

2.2 Experimental instruments

SYD-2801E Needle Penetration Tester, SYD-0655 Constant Temperature Oil Bath Chamber (Shanghai Changji Geological Instrument Co., Ltd.); YT-4507 softening point tester (Shanghai Yutong Instrument Factory); LZN-1 Elongation Tester (Wuxi Jianyi Instrument Machinery Co., Ltd.); MD-1 emulsified asphalt colloid mill (Jiaying Mide Machinery Co., Ltd.). ZRY-500 Storage Stable Tube (Shuyang County Municipal Instrument Co., Ltd., Jiangsu Province).

2.3 Preparation of adhesive layer emulsified asphalt

Heat an appropriate amount of asphalt in a (150 ± 5) °C oven to a flowing state; Dissolve the emulsifier in water at 60-70 °C to make a soap solution, and adjust the pH to 0.8-3 with hydrochloric acid; Start the colloid mill and add soap solution, cut for 0.5 minutes; Slowly add asphalt (with a solid content of 60%) and shear for 2-3 minutes to obtain a viscous emulsified asphalt.

2.4 Test methods

According to the "Test Code for Asphalt and Asphalt Mixtures in Highway Engineering" JTG E20-2011, T0605-2011, T0605-2011, T0606-2011 are used to measure the penetration, elongation, and softening point of the base asphalt, respectively. According to the T0655 storage stability test method in JTG E20-2011, the cyclic heating method was used to simulate multiple heating environments during the construction process. The long-term stability was evaluated based on the difference in solid content (SS) after standing for 1 day, 5 days, 7 days, and 7 cycles of heating cooling of emulsified asphalt. The steps of the cyclic heating storage stability test are as follows: place the sealed emulsified asphalt stability test tube in a 70 °C constant temperature oil bath, heat it to 70 °C, turn off the heating, let it stand for 24 hours, and repeat the cycle 7 times. After the cycle is completed, the storage stability (SS) of emulsified asphalt is measured using the T0655 method, and the calculation formula is as follows:

$$S_s = |P_A - P_B| \quad (1)$$

In the formula: S_s - storage stability of the sample, unit:%, P_A - residual evaporation content of the sample in the upper branch after storage, unit:%, P_B - residual evaporation content of the sample in the lower branch after storage, unit:%.

3. Experimental results and discussion

3.1 Effects of emulsifier types on the storage stability of adhesive layer emulsified asphalt

This study conducted long-term storage stability tests on adhesive emulsified asphalt prepared with different types of emulsifiers for 1d, 5d, 7d, and 7 cycles of heating. Three sets of repeated tests were conducted for each formulation, and the average value was taken as the storage stability test result. The test results are shown in Figure 1.

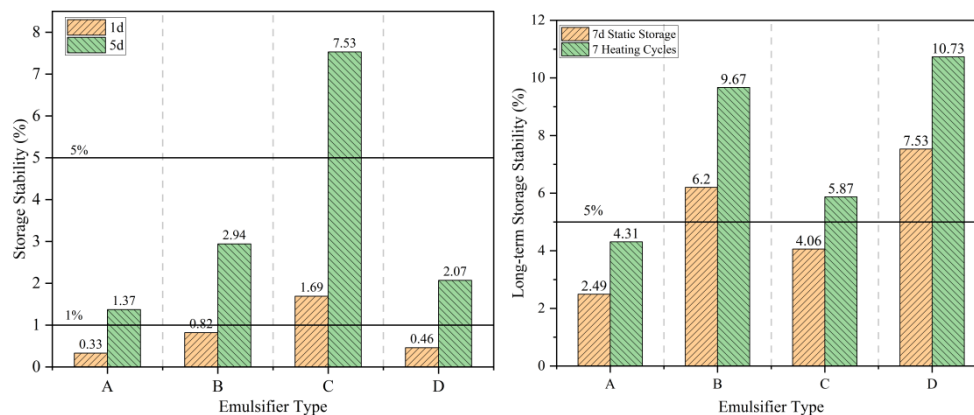


Figure 1. Relationship diagram between emulsifier type and storage stability

From Figure 1, it can be seen that under the same process, the storage stability of the adhesive emulsified asphalt prepared by four cationic fast cracking and fast setting emulsifiers at 1d and 5d is $A > D > B > C$ type. Specifically, only C-type emulsifiers do not meet the requirements for 1d storage stability; At 5 days, the difference in C-type solid content reached 7.53; B. The D-type is 2.94 and 2.07 respectively, and the emulsified asphalt in the adhesive layer begins to break down and layer; The stability comparison after 7 days of standing and 7 cycles of heating is $A > C > B > D$ type; The core of its stability difference is that the molecular structure of emulsifier affects the strength of oil-water interface facial mask. Type A (alkyl quaternary ammonium salt) has simple structure, regular lipophilic chain, high strength of interface facial mask, and good adhesion between layers; Type B (aliphatic amine quaternary ammonium salt) contains polyamine, large head base, slightly loose facial mask and poor continuity; D-type (quaternary ammonium salt calcium chloride composite) Ca^{2+} induces calcium soap bridging in asphalt at high temperatures, accelerating aggregation; C-type (lignosulfonate) has large molecules, multiple rigid aromatic rings, the most loose and weak membrane, and the worst interlayer bonding ability.

Compared with the other three emulsifiers, alkyl quaternary ammonium salt emulsifier (type A) has the best storage stability and long-term storage stability at 1d, 5d, and 7d. Therefore, the storage stability of alkyl quaternary ammonium salt emulsifier (type A) will be studied in the future.

3.2 Effect of emulsifier dosage on storage stability

The dosage of emulsifier directly affects the interlayer adhesion and system balance of emulsified asphalt road, and its adaptability is related to storage stability [6]. Based on Section 3.1, this section will investigate the long-term storage stability of A-type emulsifier with dosages of 0.8%, 1.2%, 1.6%, and 2.0% for the adhesive layer emulsified asphalt. The test results are shown in Figure 2.

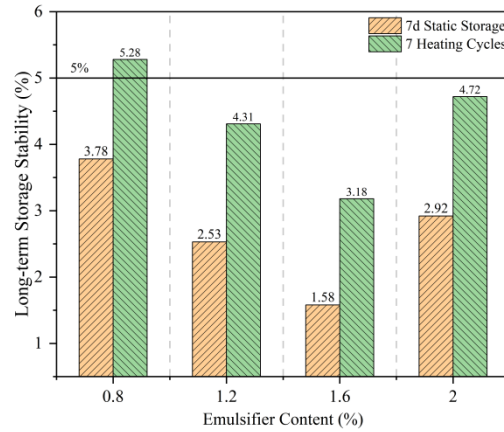


Figure 2. Relationship between emulsifier dosage and long-term storage stability

According to Figure 2, the results show that when the dosage is 0.8%, the solid content difference after 7 days of standing is 3.78%. However, after 7 cycles of heating, the layering and demulsification of emulsified asphalt are relatively strong, and the solid content difference is 5.28%, which does not meet the specification requirements; After increasing the dosage to 1.2%, the stability was significantly improved after 7 days of standing and decreased to 2.53%, and after 7 cycles of heating, it decreased to 4.31%; Continuing to increase to 1.6% and 2.0%, the stability slightly improved after 7 days of standing at 1.58% and 2.92%, and 7 cycles of heating at 3.18% and 4.72%, respectively. This is because when the dosage is low, the interface adsorption is not saturated, the surface emulsifier coverage of asphalt particles is insufficient, which is prone to coagulation and emulsion breaking, and it is difficult to spray smoothly during construction; When the dosage is $\geq 1.2\%$, the facial mask tends to be saturated, and the particle dispersion and suspension stability are enhanced, which is conducive to maintaining uniform atomization and film forming effect during spraying.

3.3 The effect of asphalt solid content on the long term storage stability of adhesive emulsified asphalt

The solid content of asphalt is a key indicator that affects the thickness and long-term storage stability of the adhesive layer of emulsified asphalt. On the basis of a solid content of 60%, this study investigated the effect of asphalt solid content on the long-term storage stability of adhesive emulsified asphalt at 56%, 58%, 60%, 62%, 64%, 66%, and 68%, and evaluated its flowability. The experimental results are shown in Figure 3.

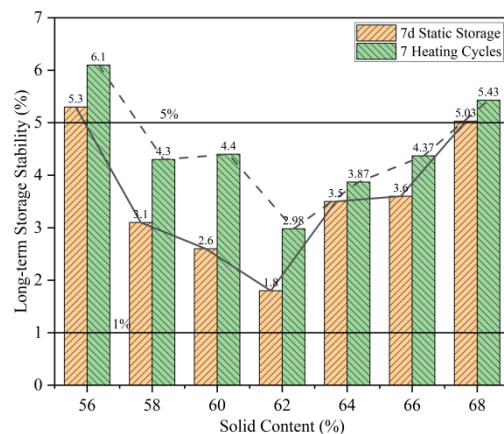


Figure 3. Relationship between solid content of emulsified asphalt and long-term storage stability

From Figure 3, it can be concluded that the long-term storage stability of adhesive emulsified asphalt

shows a trend of first decreasing and then increasing with the change of asphalt solid content difference; When the solid content of asphalt is lower than 62%, the viscosity of lotion is insufficient, the density difference between oil and water increases, and it is easy to settle and delaminate after standing for 7 days. After cyclic heating, the particle agglomeration is more significant, and the stability exceeds the specification; When it exceeds 66%, the asphalt particles become too dense, collision agglomeration intensifies, and local emulsion breaking occurs in both static and cyclic heating, resulting in decreased stability. Within the range of 58% to 66%, the optimal stability of the adhesive layer emulsified asphalt is 1.8% after 7 days of standing at 62% and 2.98% after 7 cycles of heating. This is because at this solid content, the system has the best matching degree of viscosity, flowability, and particle dispersion, which can not only suppress settling but also ensure uniform spray atomization and continuous film formation, which is conducive to improving the engineering applicability of the adhesive layer emulsified asphalt.

3.4 Methods for improving storage stability based on emulsification temperature

The temperature of asphalt and soap solution has an important influence on the emulsification process and the formation of interfacial facial mask, and is the key factor to ensure the storage stability of emulsified asphalt for tack coat and the quality of spraying film formation [7]. Regarding this, the effects of asphalt and soap temperature on the 1-day storage stability of emulsified asphalt with a 1% A-type emulsifier content and a 62% solid content were investigated to determine the optimal emulsification temperature. The results are shown in Figure 4.

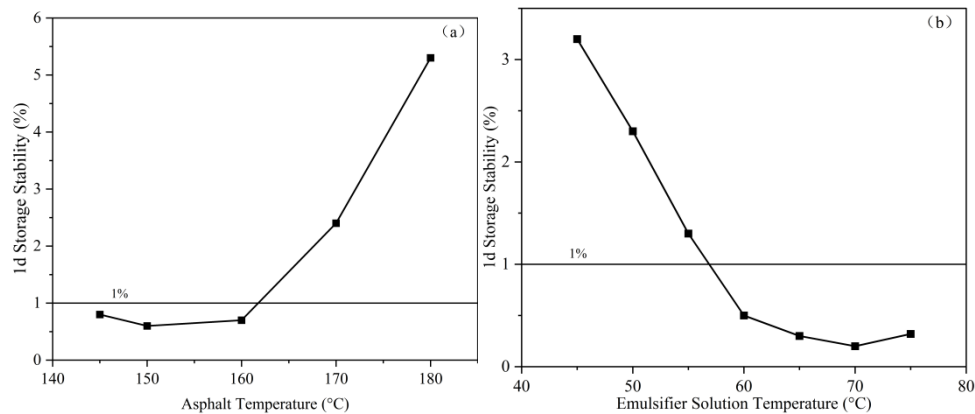


Figure 4. Effect of temperature on the 1-day storage stability of emulsified asphalt in the adhesive

From Figure 4 (a), it can be seen that increasing the temperature of asphalt can reduce the viscosity of the system and improve dispersibility. However, when the temperature is too high, phenomena such as boiling and local demulsification are prone to occur, leading to a decrease in storage stability. Therefore, the suitable emulsification temperature for asphalt is 145-160°C. Figure (b) shows that when the temperature is lower than 60°C, the emulsifier is not completely dissolved, and the interfacial facial mask is difficult to form, resulting in obvious sedimentation during storage; When the temperature is higher than 70°C, the thermal movement of the aqueous phase increases, and the interface facial mask is easy to be destroyed, which makes lotion easier to coalesce under the condition of cyclic heating. Based on the results of standing and circulating heating, the soap solution can ensure that the emulsifier is fully dissolved and maintains strong activity within the range of 60~70°C, forming a uniform and dense interfacial facial mask, which makes the tack coat emulsified asphalt have good dispersibility and anti settling ability.

3.5 Methods for improving storage stability based on additives

Additives have a significant effect on the long-term stability of emulsified asphalt by increasing the viscosity of the aqueous phase, improving the density difference between oil and water, and inhibiting particle settling [8]. This study will investigate the impact of the above three additives (with a dosage of 0.1% -0.5%) on the long-term storage stability of adhesive emulsified asphalt based on A-type emulsifier, emulsifier dosage of 1%, and solid content of 62%. The experimental results are shown in Figure 5.

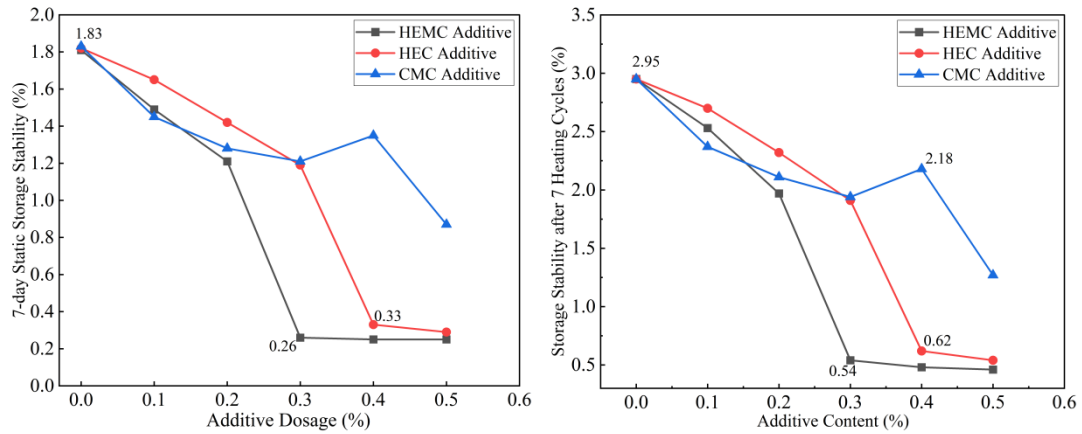


Figure 5. Trend diagram of the long-term storage stability of emulsified asphalt versus additive

Figure 5 shows that there are significant differences in the improvement effects of different additives, with HEMC having the most prominent effect, followed by HEC, and CMC having the weakest effect. At 0.3% dosage, HEMC can significantly improve the viscosity of lotion and form a certain spatial network structure, so that the particles can be more stably dispersed in the water phase. The solid content difference under the condition of standing for 7 days and under the condition of circulating heating is reduced to about 0.26% and 0.54% respectively, which is significantly better than other additives. HEC can only achieve similar effects at a dosage of around 0.4%, while CMC did not show a stable improvement trend at all dosages. Considering construction and ease of use, the adhesive layer emulsified asphalt needs to maintain good spray flowability. Excessive viscosity will affect atomization and film formation, so the dosage of additives should not be too high. Based on the comprehensive test results, a HEMC dosage of 0.3% can achieve the best balance between storage stability and engineering construction performance.

3.6 Research on storage stability enhancement scheme based on soap PH value

The PH of soap solution affects the protonation degree of emulsifier and the double electric layer structure of asphalt particles, which is the core factor determining the strength and dispersion stability of facial mask of adhesive layer emulsified asphalt interface [9]. Only at the appropriate PH value can the stability of lotion system be maintained. Therefore, this study will adjust the pH value of the soap solution based on the previous stage and investigate the effect of different soap solution PH values on the storage stability of emulsified asphalt. The experimental results are shown in Figure 6.

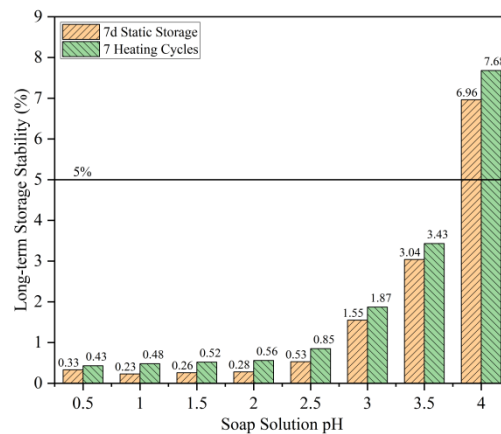


Figure 6. Effect of soap solution PH on the long-term storage stability of viscous emulsified asphalt

Figure 6 shows that as the PH increases, the storage stability significantly decreases; When $\text{PH} \leq 3.5$, the active group of the emulsifier is fully protonated, which can form a dense interfacial facial mask on the surface of asphalt particles. The repulsive force between particles is enhanced, and it can maintain high stability under static and cyclic heating conditions. When $\text{PH} \geq 4.0$, the degree of protonation decreases, emulsifiers are difficult to effectively adsorb, particles quickly aggregate, leading to phenomena such as emulsion breaking, oil separation, and severe stratification, and storage stability

cannot meet the requirements at all. For adhesive emulsified asphalt, a lower pH is not only beneficial for improving the dispersion of the system, but also for forming a uniform and continuous film after spraying, thereby improving the interlayer bonding effect. Based on comprehensive analysis, controlling the pH of soap solution within the range of 2.5-3.5 can achieve optimal storage stability and construction applicability.

4. Conclusion

1) Alkyl quaternary ammonium salt type A emulsifier can form a strong interface facial mask structure, with excellent particle dispersion, and its comprehensive storage stability is the best, and the appropriate dosage is 1%.

2) The asphalt solid content has a significant impact on the storage stability of the tack coat emulsified asphalt, of which 62% solid content can make the viscosity, fluidity and dispersion of the lotion reach a better balance, and has the optimal stability under the conditions of standing and circulating heating.

3) The suitable temperature for asphalt emulsification is 145-160 °C, and the suitable temperature for soap solution is 60-70 °C. This temperature range can ensure the effective dissolution of emulsifier, the full dispersion of asphalt particles, and maintain a continuous and dense interface facial mask structure.

4) The enhancement effect of HEMC in the additive is the most significant, which can significantly reduce the difference in solid content at a dosage of 0.3%, and its stability is significantly better than HEC and CMC.

5) The pH value of the soap solution should be controlled within the range of 2.5-3.5. A lower pH can enhance the protonation degree of the emulsifier, improve the repulsion between particles, and ensure the long-term storage stability and spray adaptability of the adhesive emulsified asphalt.

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