

Influence of preparation processes on the droplets size characteristics and storage stability of emulsified asphalt

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ABSTRACT

Emulsified asphalt is commonly used in highway engineering as a binder for interlayer bonding, slurry seals, micro-surfacing, and cold recycled asphalt mixtures. Its storage stability is a common technical challenge in the field. The preparation process of emulsified asphalt significantly impacts its storage stability. This paper examines the effects of emulsion pH, asphalt temperature, and shearing time on the storage stability of emulsified asphalt and the particle size of asphalt droplets. The morphological evolution of asphalt droplets was observed using fluorescence microscopy, and the principles of emulsion sedimentation were elucidated. Based on this, the optimal preparation conditions for emulsified asphalt were determined using response surface methodology. The results indicate that emulsion pH, asphalt temperature, and shearing time significantly affect the particle size of emulsified asphalt droplets and their storage stability. Emulsified asphalts with a larger number of droplets and smaller particle sizes exhibit superior storage stability. Furthermore, the correlation between the D50 particle size of asphalt droplets and the storage stability of emulsified asphalt is stronger than that of D10 and D90, making D50 a more suitable indicator for predicting and assessing the storage stability of emulsified asphalt. Finally, the response surface optimization identified the optimal preparation conditions for emulsified asphalt as an emulsion pH of 3.2, an asphalt temperature of 137.3°C, and a shearing time of 74 seconds.

Keywords: Storage Stability; Process; Particle Size; Morphological Observation; Response Surface.

1. INTRODUCTION

Emulsified asphalt is frequently utilized as a bonding agent between the various layers of newly constructed asphalt pavements and in asphalt pavement maintenance projects such as cold recycled asphalt mixtures, slurry seal coats, and micro-surfacing mixtures, marking its significance as a pivotal road material [1–3]. During prolonged storage, emulsified asphalt often undergoes segregation and stratification, leading to demulsification and the loss of its binding properties. Severely segregated emulsified asphalt not only fails to uniformly coat aggregates, preventing proper compaction and formation, but also results in uneven coloration and inconsistency in asphalt film thickness across the pavement surface [4–7]. The storage stability of emulsified asphalt, crucial during its storage and transportation, is primarily influenced by the type and amount of emulsifying agents and stabilizers used, the production process, and the associated equipment [8].

Existing research posits a strong correlation between particle size and stability, suggesting that under identical conditions, larger particle sizes correlate with poorer storage stability [9]. Emulsified asphalt is inherently thermodynamically unstable, with asphalt droplets dispersed in an emulsifier solution. Due to the density difference between asphalt and water, asphalt droplets are prone to sedimentation due to gravity during long-term storage and transportation, leading to the loss of emulsified asphalt's ability to bond at ambient temperatures [10]. Studies have discovered a high correlation between the storage stability of emulsified asphalt and the sedimentation velocity of droplets. According to Stokes' law, the sedimentation velocity of an individual droplet depends on the droplet's size and the viscosity of the emulsion; smaller asphalt droplets and higher emulsion

viscosity contribute to the stability of the asphalt emulsion [11]. However, when the material composition of the emulsified asphalt, including the dispersed and continuous phase ratios, remains constant, the viscosity of the emulsified asphalt does not change. Thus, under the same material conditions, the size of the asphalt droplets determines the storage stability [12, 13]. Therefore, optimizing the production process to reduce the size of asphalt droplets presents a viable technical approach to enhance storage stability. However, there is relatively little research on the particle size and storage stability of asphalt droplets in the current processing technology of emulsified asphalt.

In this regard, the present study initially elucidates the influence of emulsion pH, asphalt temperature, and emulsification time on the storage stability of emulsified asphalt and the particle size of asphalt droplets, aided by the observation of morphological evolution of asphalt droplets through fluorescence microscopy. Subsequently, the preparation process of emulsified asphalt is optimized using response surface methodology, with the objective of effectively enhancing the storage stability of emulsified asphalt.

2. MATERIALS AND METHODS

2.1. Raw materials and preparation process

The raw materials include Karamay 90# base asphalt (KLMY90#, with properties as shown in Table 1), cationic emulsifier, concentrated hydrochloric acid, and distilled water. To ensure the storage stability of the emulsified asphalt emulsion, calcium chloride (CaCl₂) is used as a stabilizer. The preparation process of emulsified asphalt involves initially heating 600g of Karamay 90# asphalt to 140°C for standby. Subsequently, the emulsifier is added to 400g of distilled water, and the mixture is heated to 55–60°C using a constant temperature electric heating jacket, followed by the addition of hydrochloric acid to adjust the pH value of the emulsion to 2. A colloid mill is then started, into which the emulsion is first introduced, followed by the gradual addition of the hot asphalt, with continuous stirring using a glass rod. After the complete addition of asphalt, the mixture is sheared in the colloid mill for 120s, then poured into a container, cooled to room temperature, and sealed for storage.

2.2. Test methods

(1) Storage Stability. Measure 300g of emulsified asphalt and filter it through a 1.18mm sieve into a storage stability test tube up to the 250ml mark, then let it stand for 1 day. After standing, take 50g samples from both the top and bottom of the tube and place them in an evaporation residue container. According to the “JTGE20-2011 Highway Engineering Asphalt and Asphalt Mixture Test Procedures” (T0651), determine the difference in the content of evaporated residue between the top and bottom samples, denoted as S(%), to characterize storage stability. The larger the S value, the poorer the storage stability.

(2) Particle Size Distribution Test. The particle size of emulsified asphalt droplets is measured using a Battersize 2600 laser particle size analyzer, with a testing range of 0.1µm to 750µm. Before testing, dilute the asphalt emulsion with 1-2 drops of pure water to ensure the sample’s opacity is between 10% and 20%. Select D10, D50, and D90 as evaluation indices, whose physical meanings are illustrated in Figure 1. D10, also known as the fine particle size, indicates that 10% of asphalt droplets are smaller than this value; D50, also called the

Table 1: KLMY90# technical indicators.

ITEMS	KLMY90#	METHODS
(25°C, 100g, 5s) Penetration (0.1mm)	85	T0604
Softening point (°C)	45.5	T0606
(5cm·min ⁻¹ , 10°C) Ductility (cm)	>100	T0605
Density (15°C)(g/cm ³)	0.98	T0603
After RTFOT		
Penetration ratio (25°C) (%)	73	T0604
(5cm·min ⁻¹ , 10°C) Ductility (cm)	37	T0605
Asphalt components		
Saturates/%	43.08	T0618
Aromatics/%	28.42	T0618
Asphaltenes/%	1.50	T0618
Resins/%	17.42	T0618

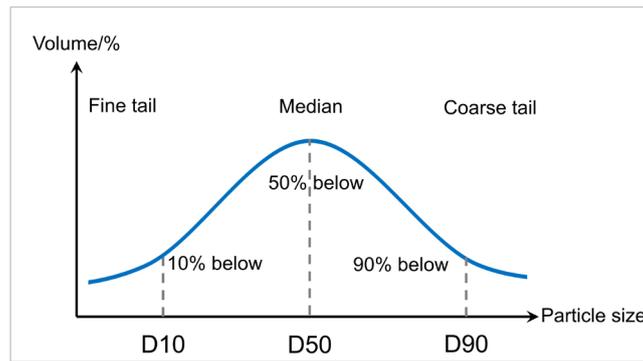


Figure 1: Physical significance of D10, D50, and D90.

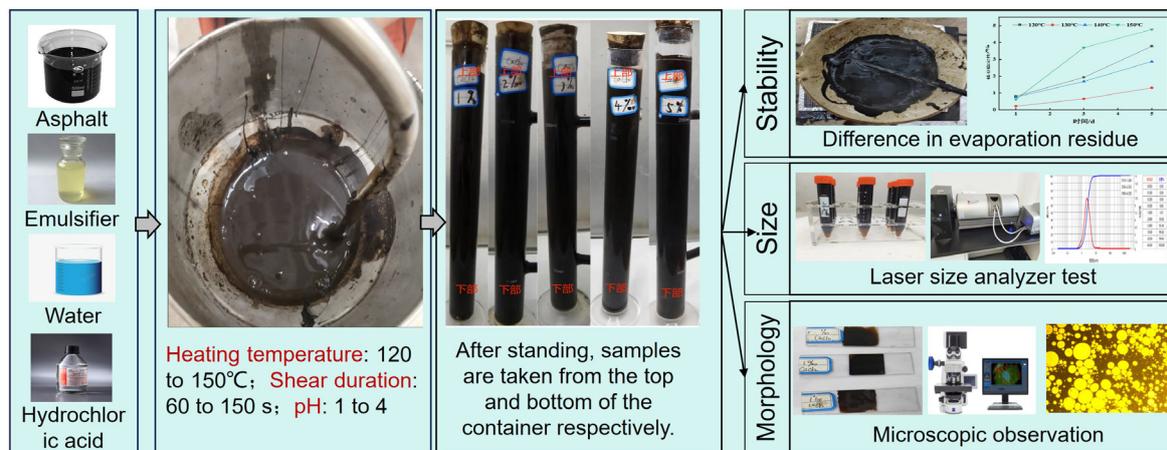


Figure 2: Experimental procedure.

median particle size, signifies that half of the asphalt droplets are smaller than this value; D90, also known as the coarse particle size, denotes that 90% of asphalt droplets are smaller than this value.

(3) Microscopic Observation. The evolution of asphalt droplets during the storage of emulsified asphalt is observed using a high-magnification optical microscope, WMF-3530, with a magnification of 4 times. Digital photographs capturing the morphology of emulsified asphalt droplets at various time points are collected. These experiments are summarized in Figure 2.

3. RESULTS AND DISCUSSION

3.1. Storage stability

As shown in Figure 3, regardless of the processing conditions, as the storage time of the emulsified asphalt extends from 1 day to 5 days, the difference in the percentage of evaporated residue between the top and bottom sections of the sample, denoted as *S*, significantly increases. This indicates a continual growth in the spatial distribution density difference of asphalt droplets within the emulsified asphalt sample. However, the rate of increase in the *S* value varies depending on the preparation process. Figure 3(a) reveals that the storage stability of emulsified asphalt deteriorates most rapidly when the pH value is 1, with the *S* value escalating from 0.39% on day 1 to 8.21% on day 5, an increase of nearly 21 times. Conversely, the slowest deterioration in storage stability occurs at pH2, where the *S* value modestly rises from 0.23% to 1.26%, indicating optimal storage stability under these conditions. Additionally, the storage stability at pH3 and 4 is similar and not ideal compared to pH1. This aligns with existing research findings that inappropriate pH values can reduce the activity of emulsifiers, leading to a decrease in the charge on asphalt particles, thereby lowering the stability of emulsified asphalt [14]. Studies have shown that emulsifier activity is diminished in a strong acid environment due to the influence of H⁺ ions, as the double layer is disrupted. When the concentration of H⁺ is moderate, the alkylamine of the

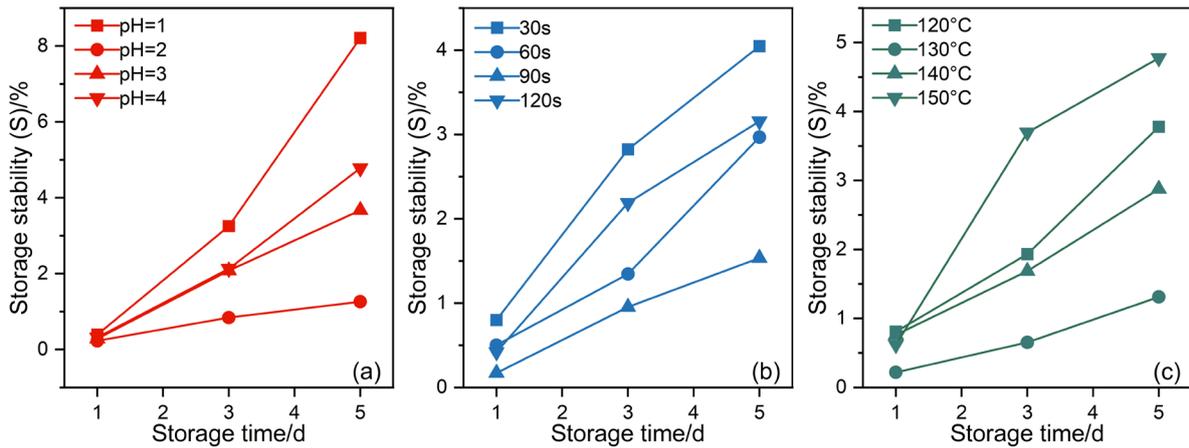


Figure 3: Impact of emulsion (a) pH value, (b) emulsification shearing time, and (c) asphalt heating temperature on the storage stability of emulsified asphalt on days 1, 3, and 5.

emulsifier forms alkylammonium ions through protonation, reacting with hydrochloric acid to form ammonium chloride, making the emulsifier more water-soluble and enhancing its emulsifying capability.

Figure 3(b) indicates that, compared to 30s and 120s, a shearing time of 90s yields optimal storage stability of emulsified asphalt at all stages. This outcome arises because insufficient shearing time does not allow for adequate shearing of the asphalt, leading to suboptimal emulsification. Conversely, as emulsification time increases, the internal temperature of the machinery can become excessively high due to the machine's own heat generation, resulting in the formation of bubbles or even boiling phenomena. The substantial evaporation of water under these conditions can lead to destabilization [15].

Figure 3(c) shows that when the asphalt heating temperature is set at 130°C, the emulsified asphalt exhibits the best storage stability, with the lowest rate of deterioration at all stages. This is because as the asphalt temperature increases, the temperature of the dispersed phase (water phase) also rises, accelerating molecular motion. This enhances the ability of the emulsifier to reduce surface tension, making it more effective at emulsifying asphalt particles. However, temperatures that are too high may exceed the critical temperature of the emulsion system. At this point, water evaporates rapidly, causing the emulsion system to destabilize [16].

3.2. The effect of preparation process on the particle size characteristics of emulsified asphalt droplets

3.2.1. Particle size characteristics

Figure 4 presents the particle size distribution curves of the upper and lower portions of emulsified asphalt samples under various pH values, processing temperatures, and emulsification times on days 1, 3, and 5. It is observable that the majority of the emulsified asphalt droplet sizes are concentrated in the 1~10 μm range, with a peak around 2 μm . Clearly, it is challenging to intuitively analyze the evolution of particle size distribution of emulsified asphalt droplets during the settling process solely based on the distribution curves. Therefore, based on the data in Figure 4, the D10, D50, and D90 of the upper and lower portions of the samples were calculated, with results shown in Figure 5.

As observed in Figure 5, regardless of the preparation process, the D10, D50, and D90 of emulsified asphalt droplets across all storage times range between 1.0~1.3 μm , 2.0~3.3 μm , and 3.3~9.7 μm , respectively. Moreover, Figure 5 demonstrates that, irrespective of the preparation process, as storage time extends from 1 day to 5 days, there is a significant increase in D10 to D90 for the majority of the upper and lower sample portions of emulsified asphalt. This indicates that the longer the emulsified asphalt is stored, the larger the asphalt droplet size becomes. This phenomenon is clearly observable across all preparation conditions. Additionally, examining Figure 4 reveals that the impact of the preparation process on the particle size of asphalt droplets is limited; when the preparation process is altered, the variation in droplet size remains within a relatively narrow range. For example, when the asphalt heating temperature is increased from 120°C to 150°C, a change in droplet size can be observed, but it still remains within a close range. Therefore, the influence of asphalt droplet size on the storage stability of emulsified asphalt should be understood from the perspective of the difference in particle size between the upper and lower portions of the samples, as shown in Figure 5.

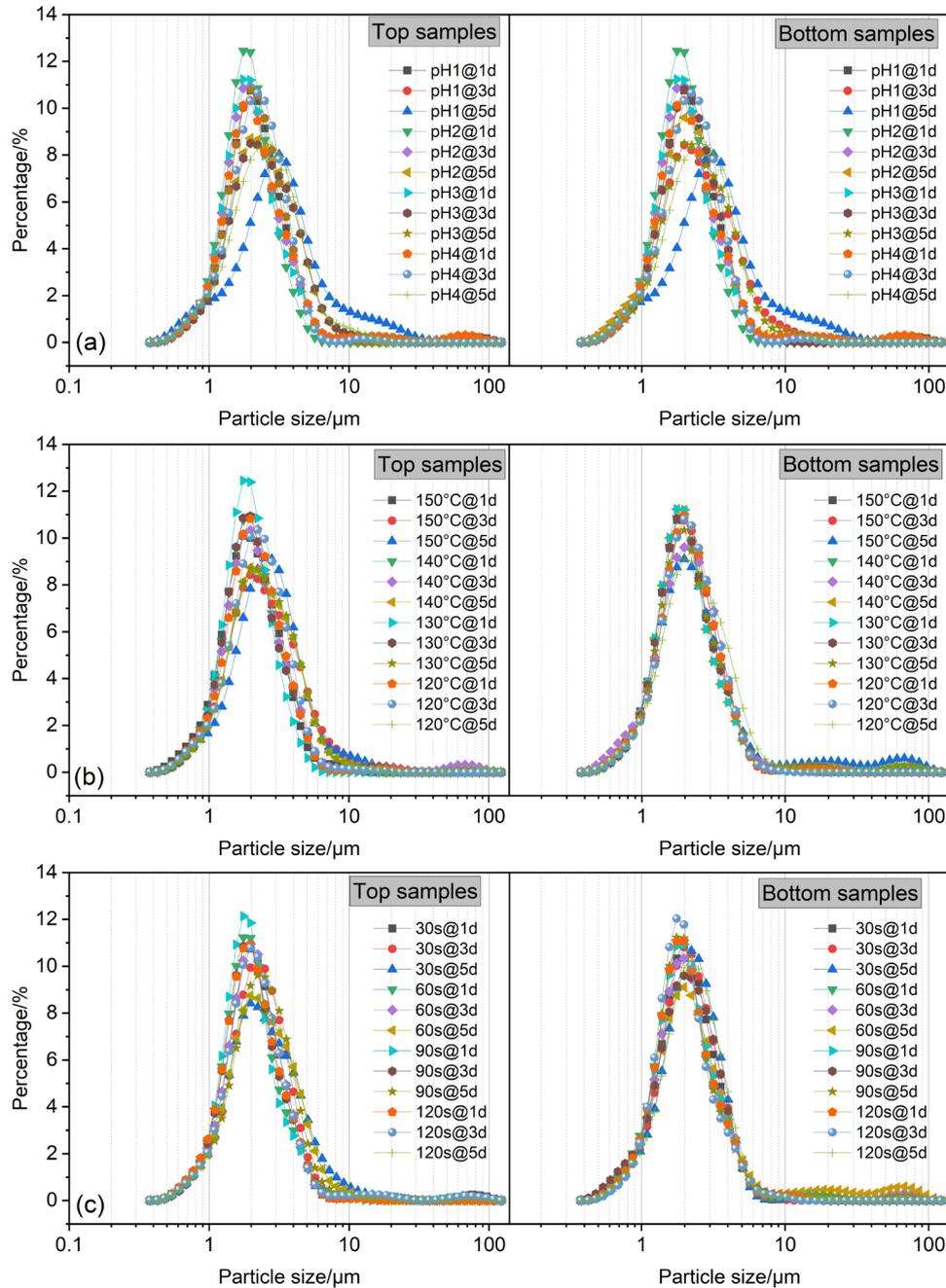


Figure 4: Particle size distribution curves of the upper and lower portions of emulsified asphalt samples under various (a) pH values, (b) processing temperatures, and (c) emulsification times on days 1, 3, and 5.

Figure 6 illustrates the difference in particle size (difference between the upper and lower sample particle sizes) of emulsified asphalt under various preparation processes. A positive particle size difference indicates that the particle size of the upper sample is larger than that of the lower one, and vice versa. Moreover, a larger absolute value of the particle size difference indicates more severe segregation and stratification of the emulsified asphalt. Overall, as storage time increases, the D10, D50, and D90 particle size differences for all emulsified asphalt samples show an increasing trend, indicating that the storage stability of emulsified asphalt deteriorates over time. Notably, at 1 day, the particle size differences in Figure 6 are mostly positive, with occasional negative values, indicating lower particle sizes are greater than the upper ones. By 5 days, the majority of particle size differences are positive, indicating upper particle sizes exceed those of the lower ones. This suggests that with extended storage time, asphalt droplets tend to aggregate upwards rather than settle downwards, colliding and adhering to other droplets to form larger droplets. This is a unique characteristic of Karamay asphalt [16].

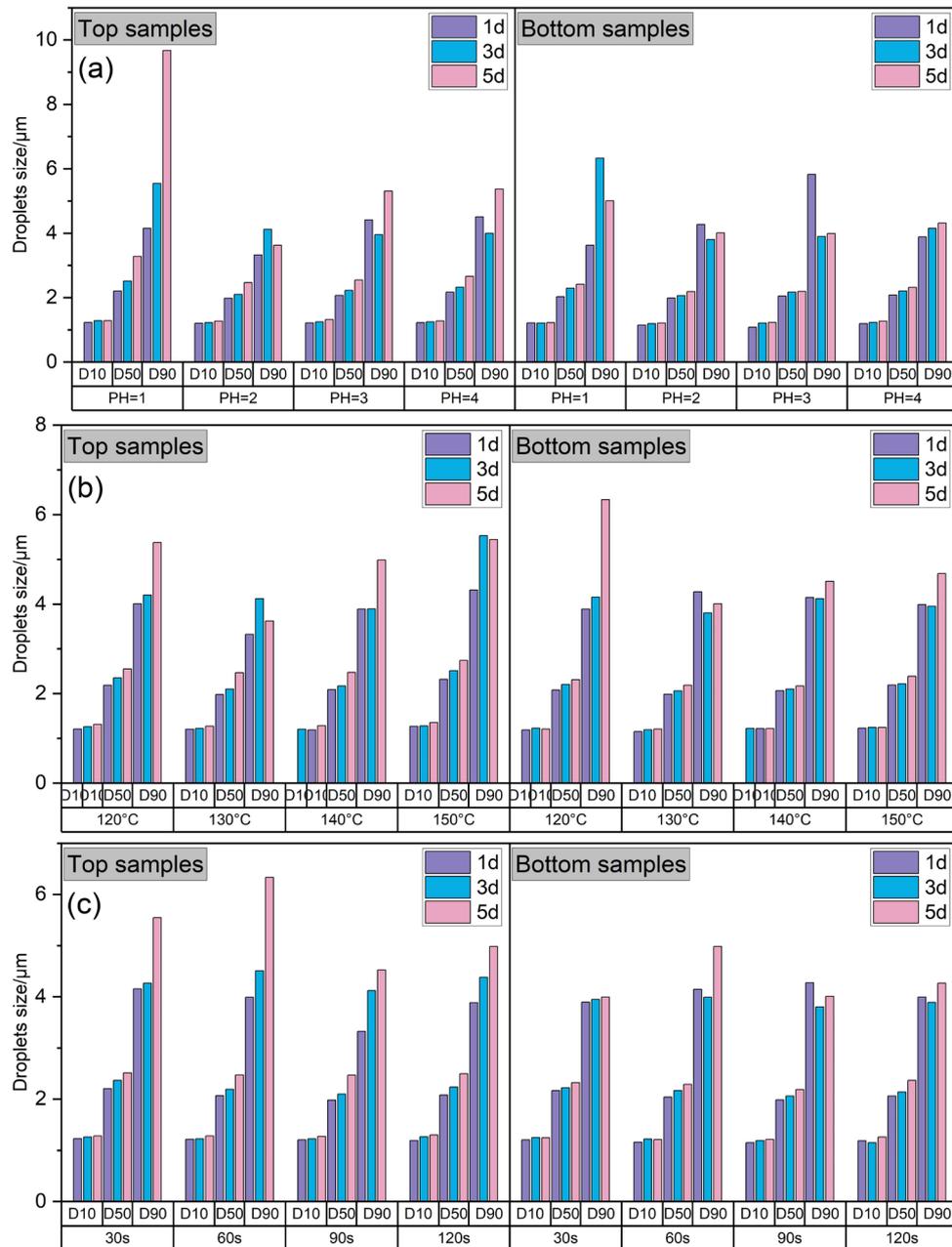


Figure 5: D10, D50, and D90 of asphalt droplets in emulsified asphalt under various (a) pH values, (b) processing temperatures, and (c) emulsification times over a storage time of 1 to 5 days.

Observing Figure 6(a), when the emulsion pH value is 1, the difference in D50 and D90 particle sizes is the greatest, especially at 3 days, indicating the most severe segregation and stratification of emulsified asphalt. When the pH value is 2, 3, or 4, the differences in asphalt droplet sizes are closer, with the smallest difference at pH2, indicating optimal emulsion stability at this pH. Furthermore, Figure 6(b) shows that when the asphalt heating temperature is between 120°C and 140°C, the differences in particle size are relatively similar across all storage times, while at 150°C, the particle size difference significantly increases. In Figure 6(c), except for 120s, when the shearing time is between 30s and 90s, the differences in asphalt droplet sizes are similar, with the smallest difference observed in emulsified asphalt prepared with a shearing time of 90s.

3.2.2. Relationship between asphalt droplet size and storage stability

Figure 7 demonstrates that the storage stability of emulsified asphalt deteriorates as the size of the asphalt droplets increases, showing a linear relationship. This phenomenon can be observed in the relationship between

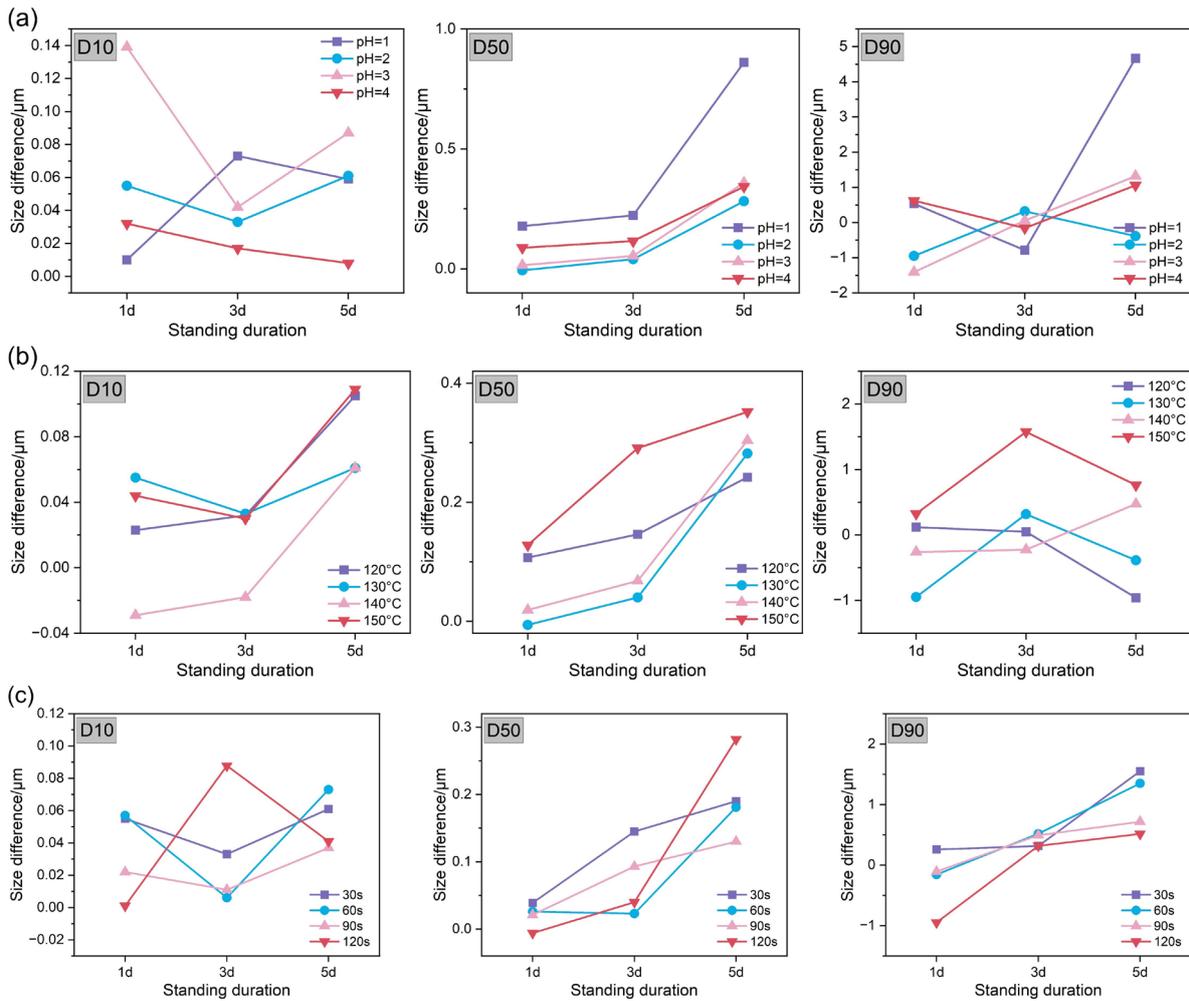


Figure 6: Difference in particle size between the upper and lower portions of emulsified asphalt droplets over a storage time of 1 to 5 days, prepared by different (a) pH values, (b) processing temperatures, and (c) emulsification times.

D10, D50, D90, and storage stability. Furthermore, Figure 7 indicates that the correlation between particle size and storage stability of emulsified asphalt is more pronounced in the upper sample than in the lower one. For example, the linear correlation coefficients (R^2) of D10 with storage stability are 0.54 for the upper sample and 0.31 for the lower sample; for D50, the numbers are 0.85 for the upper and 0.75 for the lower; for D90, R^2 is 0.76 (upper) and 0.11 (lower). Therefore, utilizing the particle size of the upper asphalt sample to predict or assess the storage stability of emulsified asphalt is more accurate than using the lower sample. Lastly, Figure 7 indicates that the correlation between the D50 of asphalt droplets and the storage stability of emulsified asphalt is stronger than that for D10 and D90, with a linear correlation coefficient R^2 of 0.85, higher than 0.54 for D10 and 0.76 for D90.

According to the JTG-F40-2004 “Technical Specifications for Highway Asphalt Pavement Construction” in China, the S values of emulsified asphalt should not exceed 1% and 5% after 1 day and 5 days, respectively. Based on the relationship between the D50 of the upper asphalt droplets and S, the particle size of the asphalt droplets should not exceed $2.14\mu\text{m}$ and $2.79\mu\text{m}$ after 1 day and 5 days, respectively.

3.2.3. Microscopic observation

Figure 8 shows that regardless of the preparation process, the distribution density and particle size of asphalt droplets in the upper and lower portions of emulsified asphalt are relatively similar at 1 day. As the storage time extends to 5 days, the particle size of the upper asphalt droplets becomes larger, with observations of multiple asphalt droplets aggregating and flocculating, forming a flaky distribution; the particle size of the lower asphalt droplets increases compared to day 1, but their size and distribution density are less than those of the upper

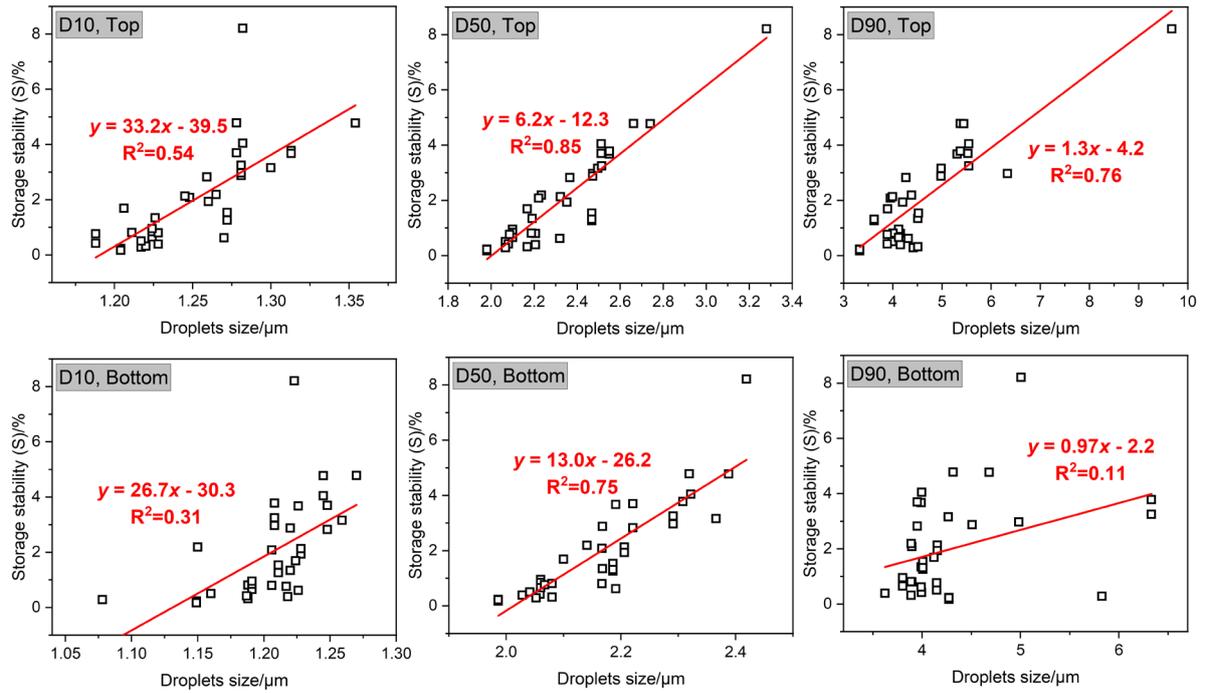


Figure 7: Relationship between particle size of emulsified asphalt droplets and storage stability.

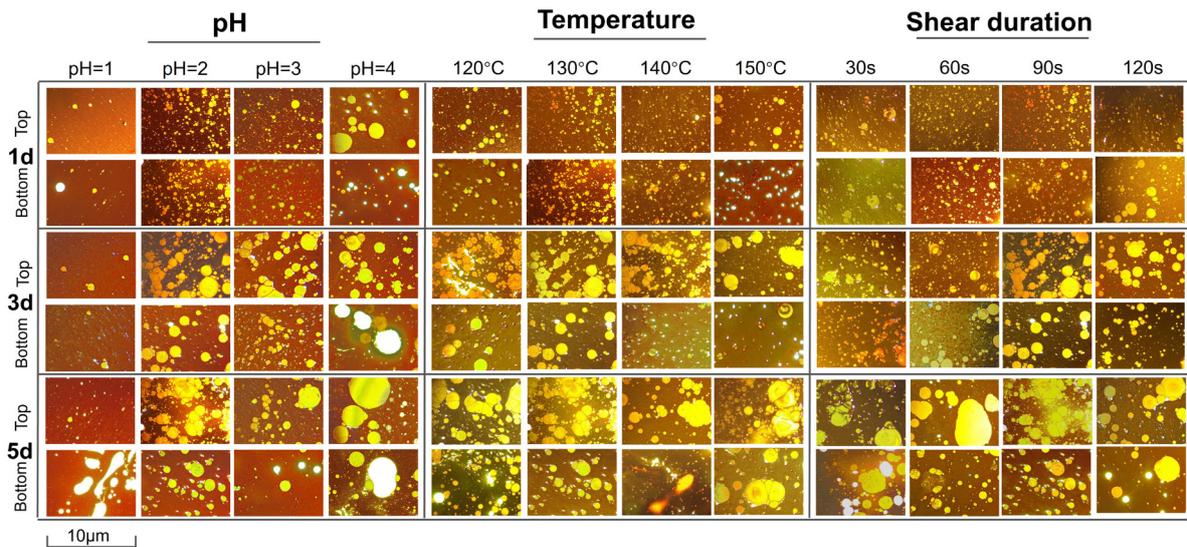


Figure 8: Microscopic images of emulsified asphalt droplets.

asphalt droplets. Therefore, the results of microscopic observation once again confirm that for emulsified asphalt based on Karamay asphalt, the deterioration of storage stability is characterized by the aggregation of emulsified asphalt in the upper space and occurrence of demulsification [17–20]. Furthermore, from the microscopic images, it is observed that at pH=2, asphalt temperature of 130°C, and shearing time of 90s, the difference in distribution density and particle size of asphalt droplets between the upper and lower portions is smaller compared to other processes, confirming better storage stability under this process.

Figure 9 reveals that low-density asphalt droplets, represented by Karamay asphalt and characterized by a high content of light components, exhibit upward flotation during segregation rather than the traditional downward sedimentation. This behavior diverges from our past understanding of emulsified asphalt demulsification and cracking processes [21]. This pattern can be intuitively understood through a subsequent figure. During the storage of the emulsion, asphalt droplets initially exhibit a tendency to float upwards. Following this, the

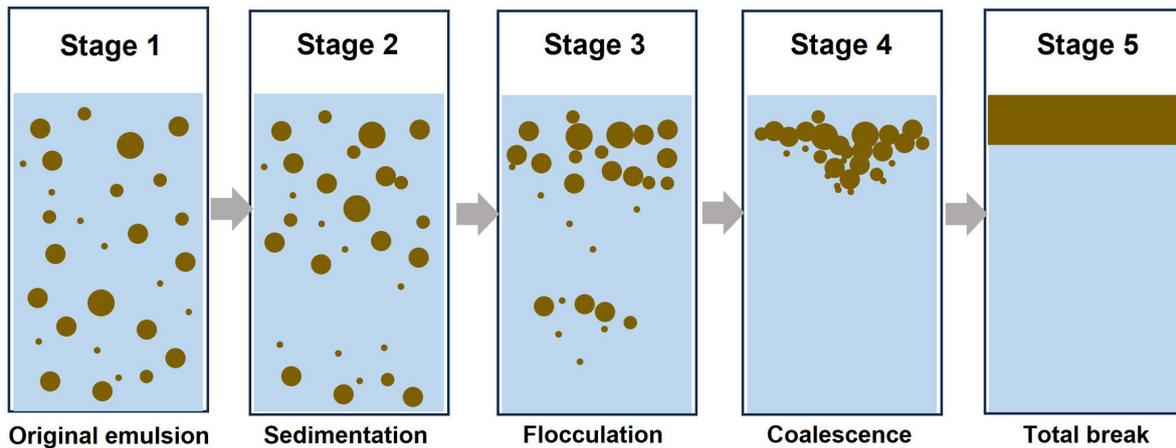


Figure 9: The demulsification process of emulsified asphalt droplets.

Table 2: Factors and levels for the response surface methodology.

LEVELS	X_1	X_2	X_3
	SHEAR DURATION/S	EMULSION pH	ASPHALT TEMPERATURE/°C
-1	60	2	120
0	75	3	130
1	90	4	140

droplets begin to contact each other at the top of the emulsion, leading to flocculation. However, they remain partially protected by the emulsifier's film, thus retaining their shape. This explains why, during the particle size analysis and microscopic observation at 1 day, 3 days, and 5 days, the particle sizes of the upper asphalt droplets are consistently larger than those of the lower droplets. Subsequently, irreversible aggregation occurs, with asphalt droplets extensively interconnecting. After the first coalescence, these phenomena accelerate, leading to the rupture and separation of the emulsion phase, culminating in complete demulsification. Based on our experimental results, the time required for coalescence and total break is speculated to exceed 5 days, as the emulsified asphalt observed within the 5-day period was still in stage 3.

3.3. Optimization of emulsified asphalt preparation process based on response surface methodology

The preparation process significantly influences the storage stability of emulsified asphalt, making the study of optimal preparation processes extremely important. Response Surface Methodology (RSM) is a widely utilized approach for process optimization, offering the ability to achieve the most accurate results with relatively few experiments. To this end, an experimental design was employed using emulsification time (X_1), emulsion pH value (X_2), and emulsification temperature (X_3) as independent variables, with the storage stability after 1 day (Y_1) serving as the response variable. The levels of the factors used in the experiment are detailed in Table 2.

Utilizing Design-Expert software to analyze the response variable Y_1 , the quadratic regression equation obtained for the 1-day storage stability in relation to various factors is as follows:

$$y_1 = 17.829 - 0.117X_1 - 1.143X_2 - 0.172X_3 + 0.0008X_1X_2 + 0.00018X_1X_3 + 0.0017X_2X_3 + 0.0006X_1^2 + 0.137X_2^2$$

The regression model was subjected to an analysis of variance (ANOVA) and significance testing. The computed results indicated that the model F-value is 71.65 with a P-value of <0.0001, which is less than 0.05, signifying that the model's differences are statistically significant. Additionally, the model's coefficient of determination, R^2 , is 0.9893, indicating a high degree of correlation within the model. Both X_2 (emulsion pH value) and X_3 (temperature) have P-values <0.05, demonstrating that for the 1-day storage stability, both the pH value of the emulsion and the temperature are significant influencing factors, with the order of influence

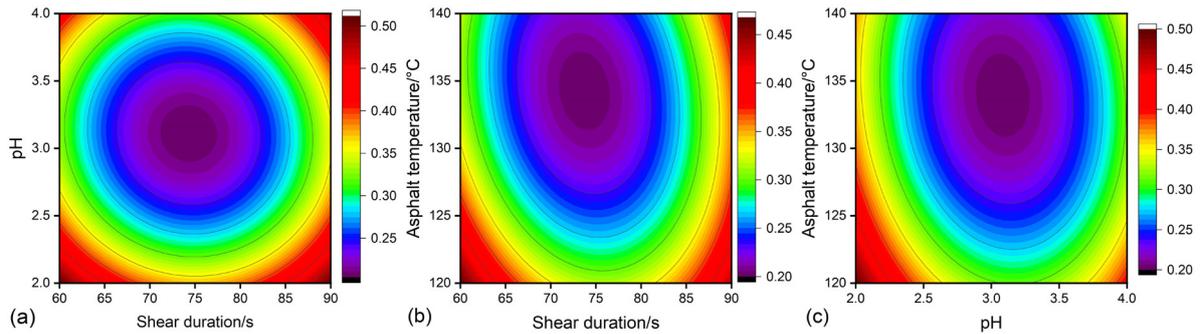


Figure 10: Results of RSM, (a) shear duration vs. pH; (b) shear duration vs. asphalt temperature; and (c) pH vs. asphalt temperature.

Table 3: Predictive value.

RSM	PREDICTIVE VALUE	TESTED VALUE			DIFFERENCE
		1	2	3	
1d storage stability	0.22	0.25	0.27	0.26	0.03

being asphalt temperature > emulsion pH value > shearing time. To clarify the interactions between factors, a three-dimensional surface plot based on the response surface model was constructed to depict the interactions between the experimental factors, with results shown in Figure 10.

Analysis via Design Expert software version 10 determined that the optimal preparation conditions for emulsified asphalt are: emulsion pH of 3.2, asphalt temperature of 137.3°C, and shearing time of 74.6 seconds. Under these conditions, three parallel experiments were conducted, with results presented in Table 3.

4. CONCLUSIONS

- (1) The storage stability of emulsified asphalt decreases as the storage time increases. The pH value, preparation temperature, and shearing time significantly affect the particle size distribution, parameters, and shape of the emulsified asphalt, which in turn influences the instability phenomena such as oil-water separation, Ostwald ripening, flocculation, and agglomeration. The highest storage stability of emulsified asphalt is observed when the pH value is between 2 and 3, the temperature is at 130°C, and the emulsification time is 90 seconds.
- (2) The particle size of the emulsified asphalt droplets follows a normal distribution, with the D10, D50, and D90 of the emulsified asphalt droplets ranging between 1.0~1.3μm, 2.0~3.3μm, and 3.3~9.7μm, respectively. As the storage time extends, the particle sizes of the asphalt droplets in both the upper and lower portions of the emulsified asphalt gradually increase, with the particle size of the upper droplets being larger than that of the lower droplets. This indicates that the base asphalt, represented by Karamay asphalt, exhibits upward aggregation in terms of layering and segregation after emulsification. This conclusion is also fully confirmed through fluorescence microscopy observation.
- (3) The storage stability of emulsified asphalt shows a linear correlation with particle sizes D10, D50, and D90. The correlation between particle size and storage stability is stronger for the upper sample than for the lower sample, and the relationship between D50 and storage stability is significantly stronger than that between D10 and D90. Therefore, D50 can be used to predict and assess the storage stability of emulsified asphalt.
- (4) The storage stability of emulsified asphalt was optimized using the Response Surface Methodology, resulting in the identification of optimal preparation conditions: emulsion pH of 3.2, asphalt temperature of 137.3°C, and shearing time of 74 seconds. The average storage stability after 1 day was 0.22% (greater than 1%), meeting the requirements for engineering applications.

5. ACKNOWLEDGMENTS

This study is jointly found by the Tianshan leading talents in scientific and technological innovation (2022TSY-CLJ0045), Natural Science Foundation of Xinjiang Uygur Autonomous Region, China (2020D01A92), Science and Technology Projects of Xinjiang Communications Investment Group Co., Ltd (ZKXFWCG-202211-010).

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