Research on Physics Stability of Emulsified Asphalt Modified by Nano Silica

Cong Li, Jie Li and Yubo Hu
Department of Airfield Logistics Support, Air Force Logistics College, NO.85Xige Street, Goulou District, Xuzhou, Jiangsu Province, China
Email: wosilecr7@126.com

Abstract. At present, research on nano-modified asphalt and asphalt mixture has made some achievements. This article applies the stabilization mechanism of pickering emulsion to prepare emulsified asphalt, and uses nano silica particles as emulsifier. On this basis, we further investigate the physical stability of emulsified asphalt modified by nano silica through analyzing and studying the sieve residue and storage stability, and establish a solid basis for subsequent development and further research.

1. Introduction
Pickering emulsion\cite{1} has been getting increased attention by researchers for its advantages of interface stability, renewability, low mammalian toxicity, low cos. It has been widely employed in in cosmetics, food, pharmacy, oil and waste water treatment industry, but it is seldom used in the preparation of emulsified asphalt. In road transportation industry, the research of nano-modified asphalt and asphalt mixture has made some achievements. Based on this idea, stabilization mechanism of Pickering emulsion is applied in the preparation of emulsified asphalt in this paper, and nano silica particles is used as emulsifier. It’s extremely important to research on physical stability of emulsified asphalt modified by nano silica for subsequent research on construction performance and pavement performance.

2. Test Preparations
2.1. Material Selection
2.1.1. Asphalt emulsifier
Asphalt emulsifier is the main raw material for preparing emulsified asphalt. The traditional asphalt emulsifiers are basically surfactant, which can prevent asphalt particles from coalescing by reducing the interfacial tension between asphalt and water to form a membrane of molecular aggregates on the surface of dispersed bituminous particles.
In this paper we choose surface-modified nano-SiO2 as the emulsifier, and the average particle size is 28 nm. The nano-SiO2 is prepared into a liquid sol with a solid content of 5%. When asphalt is dispersed into bituminous droplets under the action of shear, nano-SiO2 particles are adsorbed on the asphalt surface to form a solid particle protective layer\cite{3}, thus the preparation of Pickerin emulsified asphalt is accomplished.
2.1.2. Matrix asphalt
Asphalt is the main material for preparing emulsified asphalt. The performance of asphalt determines the main performance of emulsified asphalt. Asphalt mainly consists of four components: asphaltene, resin, saturate and aromatics. Its chemical composition varies greatly with type and source. The emulsification difficulty of asphalt with different types and chemical compositions is also different. The results show that the chemical composition of asphalt has great influence on the properties of emulsified asphalt.

In this paper, synthesizing emulsification of asphalt, comparison test of modified asphalt, pavement performance, AH-70 matrix asphalt was selected as the matrix asphalt for the preparation of emulsified asphalt. The measured technical indexes are shown in table 1.

| Type of test                                      | Test result | Technical requirements |
|--------------------------------------------------|-------------|------------------------|
| Penetration(25°C, 100g, 5s)(0.1mm)               | 71          | 60~80                  |
| Ductility(5cm/min, 15°C)(cm)                     | 126         | ≥ 100                  |
| Softening point(ring and ball method)(°C)        | 52          | ≥ 46                   |
| Wax content(distillation)(%)                     | 1.95        | ≤ 2                    |
| Density(15°C)(g/cm³)                             | 1.011       | records                |
| Solubility(trichloroethylene)(%)                 | 99.90       | ≥ 99.5                 |
| Thin-film heating test161°C, 5h                  |             |                        |
| Mass loss(%)                                     | -0.016      | ≤ 0.6                  |
| Penetration ratio(%)                             | 68          | ≥ 65                   |
| Ductility 15°C(cm)                               | 114         | ≥ 80                   |

2.1.3. Water
Water is the main component of emulsified asphalt, which can be used as the dispersing medium of asphalt and the solvent of emulsifier and additive. The selection and quality standards of water are nonnegligible. Deionized water provided by fudan university was used in this experiment and the influence of water quality on emulsification effect can be ignored.

2.1.4. Additive
Emulsified asphalt belongs to an unstable thermodynamic system, which is in in a relatively stable state. The delamination, flocculation and agglomeration of emulsified bituminous may occur with the extension of storage time and the influence of environmental temperature changes. The addition of additives can enhance the role of emulsifier and improve the stability of emulsified asphalt. Additives can be divided into two types: inorganic stabilizer and organic stabilizer, and the inorganic stabilizer mainly includes metal chloride, silicate and phosphate compounds. Generally, additives are mainly PH regulator and inorganic stabilizer.

2.2. Testing Equipment
The equipment’s for preparing and testing emulsified asphalt are shown in table 2.

| Device name                        | Model   | Device name   | Model    |
|------------------------------------|---------|---------------|----------|
| Automatic asphalt softening point tester | 1-R     | Vertical colloid mill | JM-L50    |
| Automatic asphalt penetrometer     | 3-Z     | Electric stove | DHG-9123A |
| Intelligent asphalt ductility meter | 6-Y     | Fine screen   | 0.18mm   |
| Electronic Balance                | BS 200 S | -             | -        |
3. Experimental Research

3.1. Synthesis Parameters
During the preparation of emulsified asphalt, the emulsifying temperature, the temperature of emulsifier aqueous solution, the PH value and the inorganic salt concentration all have influences on the quality of emulsified asphalt. In the early stage of the research[2], we have determined the specific parameters in the preparation process of emulsified asphalt modified by nano SiO2. Concrete parameter is shown in Table 3.

Table 3. Synthesis parameters of preparation of emulsified asphalt modified by nano SiO2

| Synthesis parameters | emulsifying temperature(°C) | emulsifier aqueous solution(°C) | PH | inorganic salt concentration |
|----------------------|-----------------------------|---------------------------------|----|-----------------------------|
| Numeric valu         | 140                         | 80                              | 3  | 15%                         |

3.2. Preparation Technology
Nano-SiO2 solution was stirred and immersed in hot water for 1-2 min. Hydrochloric acid was added to make the solution have a PH of 3. NaCl was added according to 15% of the diluted water to enhance the stability of the emulsion. Heat the modified nano SiO2 solution to 80 °C and keep warm. Asphalt temperature should be controlled at 140 °C. Colloid mill JM - 150 model is adopted to improve the emulsification[2]. Start the colloid mill and preheat it for 30s before emulsification, then add hot water and circulate for 30 seconds, and repeat it again. Afterwards pour the asphalt and emulsifier solution into the colloid mill in a 1:1 ratio. Clean the colloidal mill as soon as possible after the emulsified asphalt has been obtained. Preparation process flow chart is shown in Figure 1.

![Figure 1. Preparation process of emulsified asphalt modified by nano silica](image)

3.3. Experimental Methods
In the storage procedures of emulsified asphalt, there is a force inside the emulsion that reduces the interface energy and makes it change to a more stable state. That's stability of the emulsified asphalt. This paper mainly focuses on the research of physical stability. Compared with the ordinary emulsified asphalt prepared by ordinary emulsifier, the experimental study is carried out under the same conditions, and investigate the physical stability of emulsified asphalt modified by nano silica through analyzing and studying the sieve residue and storage stability.

After the preparation of two kinds of emulsified bitumen, the ordinary emulsified bitumen is labeled as S1, and the emulsified bitumen modified by nano-SiO2 is labeled as S2. Then cool S1 and S2 to room temperature and store in sealed condition stewing for 1d, 5d, 10d, 20d respectively. Carry out the experiment by referring to <Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering>, and experiment method and technical requirements are shown in Table 4. The sieve residue and storage stability of two kinds of emulsified asphalt stewing after 1d, 5d, 10d, 20d. Test method for storage stability of emulsified asphalt stewing after 10d, 20d can refer to the method for storage stability of emulsified asphalt stewing after 1d.
### Table 4. Test methods and technical requirements for emulsified asphalt

| Test code                | Technical requirements | test method |
|-------------------------|------------------------|-------------|
| Sieve residue test      | ≤0.1                   | T0652       |
| storage stability test  | 1d(%)                  | T0655       |
|                         | 5d(%)                  |             |

#### 3.4. Analysis and Research of Test Results

Data of Sieve residue test is shown in Table 5 and Figure 2, and the data of storage stability test is shown in Table 6 and Figure 3.

### Table 5. Effects of storage time on sieve residue of emulsified asphalt

| Sample number | 1d(%) | 5d(%) | 10d(%) | 20d(%) |
|---------------|-------|-------|--------|--------|
| S₂            | 0.02  | 0.03  | 0.06   | 0.07   |
| S₁            | 0.01  | 0.08  | 0.15   | 1.24   |

### Table 6. Effects of storage time on storage stability of emulsified asphalt

| Sample number | 1d storage stability(%) | 5d storage stability(%) | 10d storage stability(%) | 20d storage stability(%) |
|---------------|-------------------------|-------------------------|-------------------------|-------------------------|
| S₂            | 0.25                    | 1.9                     | 2.5                     | 3.2                     |
| S₁            | 0.45                    | 4.1                     | 8.2                     | 10.3                    |

Figure 2. The change of sieve residue of emulsified asphalt

Figure 3. The change of storage stability of emulsified asphalt
We can analyze the data and line graph as follows:
(1)Within the storage time range of the test, the increasing of sieve residue and storage stability of the two emulsified asphalt show that the physical stability of both emulsified asphalt decrease with the increase of storage time. However, the degree of decrease is different. Emulsified asphalt modified by Nano-SiO2 has better physical stability.
(2)In the first 10 days, the sieve residue of both emulsified asphalt did not change significantly, and increased slightly. After 10 days, the sieve residue of ordinary emulsified asphalt increased significantly, and the sieve residue of emulsified asphalt modified by Nano-SiO2 remained stable.
(3)The storage stability of ordinary emulsified asphalt changed dramatically compared with that of emulsified asphalt modified by Nano-SiO2. For the two emulsified asphalt, the difference of initial stability (5d) changes sharply with the increase of storage time, indicating that 5d storage stability can reflect the long-term storage stability of emulsified asphalt to some extent.

4. Conclusion
By comparing the experimental data of the two emulsified asphalt, it can be found that, with the extension of storage time, the physics stability of emulsified asphalt modified by Nano-SiO2 is better than that of ordinary emulsified asphalt. Therefore, it can be considered that emulsified asphalt modified by Nano-SiO2 has better physical stability, laying a foundation for further improvement of its stability.

5. References
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