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Promoting the dispersion of LDHs powder in bitumen with pre-dispersion and microwave heating

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HIGHLIGHTS

- Two techniques were proposed to promote the dispersion of LDHs in bitumen based on the theory of Stokes' law.
- The size distribution of LDHs in bitumen was examined.
- Pre-dispersion alone could slightly improve the dispersion of LDHs in bitumen.
- The combination of pre-dispersion and microwave heating significantly improved the dispersion of LDHs in bitumen matrix.
- The type and content of the cosolvent were optimized.

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ABSTRACT

Layered Double Hydroxides (LDHs) are a family of intercalated functional materials with a layered structure and regarded as an effective modifier to improve the UV ageing resistance of bitumen. However, the preparation of LDHs modified bitumen is very complicated and time-consuming. Based on the theory of Stokes' law and the equation of the terminal velocity, pre-dispersion and microwave heating were proposed in this research to promote the dispersion of LDHs powder in bitumen matrix. LDHs modified pre-dispersed bitumen (LDHs-PDB) particles were first prepared and then were dispersed in bitumen matrix with the assistance of microwave heating. The cosolvent was optimized and the dispersion of LDHs in the modified bitumen was characterized by the laser particle size analyzer. It is found that the utilization of pre-dispersion alone could slightly improve the dispersion of LDHs in bitumen. The combination of pre-dispersion and microwave heating significantly improved the dispersion of LDHs in bitumen matrix. Although the distribution is still not uniform, it is believed that a reasonable design of the size distribution of LDHs-PDB particles may result in a uniform vertical distribution of particles in bitumen matrix.

1. Introduction

Modification of bitumen is a common practice to improve its physical properties and performance [1]. Nowadays, various modification methods of bitumen have been developed by researchers. Polymer modification, including polyethylene (PE), polypropylene (PP), ethylene–vinyl acetate (EVA), ethylene–butyl acrylate (EBA), styrene–butadiene–styrene (SBS) and so on, has been one of the most popular approaches [2]. These polymer modified bitumen with better properties and performance are widely used in many areas [3]. With the development of transportation and the increase of the volume of traffic, more and more functional requirements of bitumen were demanded. In this condition, polymer modification may not always reach the requirement of the pavement. As a consequence, some inorganic materials were considered in bitumen modification, such as TiO_2 and graphite [4,5]. For instance, the idea of anti-ultraviolet ageing bitumen was proposed with the in-depth understanding of the mechanism of ultraviolet (UV) aging on bitumen. Some researchers have already conducted a series of experiments in this field, and found that a kind of hydrotalcite (HT)-like materials, i.e., Layered Double Hydroxides (LDHs) was a good new type of bitumen UV anti-aging agent [6–10].

LDHs are a family of intercalated functional materials with a layered structure [8]. The general chemical composition of LDHs is $[M^{2+}_{1-x}M^{3+}_{x}(OH)_2]^{x+}(A^{n-}_{x/n})\cdot mH_2O$. M^{2+} and M^{3+} are respectively divalent and trivalent cations; x is the content variation of metallic elements; A^{n-} represents the interlayer anion; and m represents the amount of water located in the interlayer galleries [11]. It is founded that LDHs can improve the UV aging resistance of bitumen, and the

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asphalt mixtures with more LDHs can present a better low-temperature performance and fatigue resistance [9,10]. Thus, LDHs could be an alternative modifier used in the bitumen to improve the lifetime of asphalt pavements [7]. However, the preparation of LDHs modified bitumen is very complicated and time-consuming. Most of LDHs powder centralizes in the upper layer of bitumen while mixing, resulting in a very bad dispersion. Therefore, some methods should be taken to improve the dispersion of LDHs in bitumen. In this paper, two effective methods were designed and optimized to promote the dispersion of LDHs powder in bitumen.

As the LDHs modified bitumen consists of two distinct phases, the dispersion process of LDHs in bitumen can be theorized based on Stokes' law [1]. According to this theory, this process in modified bitumen is governed by the terminal velocity of the dispersed phase in Newtonian liquid. Obviously, the terminal velocity in the system will positively correlated with the displacement velocity of the particles. Several researchers have already used this theory to explain the mechanism of separation in bitumen modification [12–14]. As is known, the terminal velocity is the velocity of the displacement of the particles when the gravity force on the particles equals the drag force on the particles in a Newtonian liquid and can be quantified by Eq. (1):

$$V_{t\infty} = \frac{2a^2\Delta\rho g}{9\eta} \quad (1)$$

where

a = radius of dispersed particle,
$\Delta\rho$ = difference of density between particle and Newtonian liquid medium,
g = gravitational acceleration, and
η = viscosity of liquid medium.

In terms of this equation, the terminal velocity of the particles is directly related to the square of the radius of the particles and inversely related to the viscosity of the medium. So, two ways can be considered to promote the dispersion of LDHs in bitumen. One was increasing the radius of dispersed particle and the other was decreasing the viscosity of liquid medium by heating the bitumen.

In order to increase the radius of the particles, LDHs powder was pre-dispersed with bitumen matrix at a very high LDHs-bitumen ratio firstly, and then the produced LDHs modified pre-dispersed bitumen (LDHs-PDB) was turned into little pieces of particles. In this situation, the radius of dispersed particles was obviously increased. Meanwhile, the displacement of LDHs powder would be much like a process of dilution rather than dispersion, which will absolutely accelerate the dispersion speed of LDHs powder.

On the other hand, it is very clear that a higher temperature leads to a lower viscosity of liquid medium. So, the rapid heating method was also believed as a good way to promote the dispersion of LDHs powder. In this study, the microwave heating was applied due to its high heating efficiency and speed.

Table 1
The properties of LJ-70# base bitumen.

Tests	Results	Specification
Penetration (25 °C, 100 g, 5 s) [0.1 mm]	65.7	60–80
Softening point (ring and ball) [°C]	50.2	44–54
Ductility (5 cm/min, 15 °C) [cm]	>150	⩾100
Density (g/cm^3)	1.01	–

Table 2
The properties of LDHs powder.

Tests	Results
Appearance	White powder
LDHs (%)	99.5
Bulk density (g/cm^3)	0.45
Humidity content (%)	⩽3
Apparent density (g/cm^3)	0.30
Bulk density (g/cm^3)	0.43
True density (g/cm^3)	2.22

2. Materials

In this paper, the bitumen use was LJ-70# base bitumen, provided by Hubei Guochuang Hi-tech Material Co. Ltd. of China. LDHs powder was provided by Ruifa Chemical Company Limited of China. The composition of LDHs is $Mg_{1-x}Al_x(OH)_2(CO_3)_{x/2}\cdot mH_2O$, Where x is the content variation of metallic elements, $0.2 \leqslant x \leqslant 0.33$; m is the amount of crystal water, $0 \leqslant m \leqslant 2$.

Three kinds of cosolvent powders were also used to improve the microwave heating rate of LDHs-PDB particles. This work presented in this paper is the subject of a pending patent, so those three cosolvent powders could only be denoted as A, B and C. The properties of these materials are shown in Tables 1–3.

As can be seen in Tables 1 and 2, the apparent and bulk density of LDHs (0.30 g/cm^3, 0.43 g/cm^3) is much lower than pure bitumen (1.01 g/cm^3), but its true density (2.22 g/cm^3) is twice of pure bitumen. In this situation, the density of LDHs-PDB particles will be larger than pure bitumen while the density of LDHs powder is still lower. According to Eq. (1), it is believed that the terminal velocity will obviously improved by the application of pre-dispersion method.

3. Experiments

3.1. Preparation of LDHs modified bitumen

LDHs modified bitumen were involved in this paper. The LDHs powder was added into 70# base bitumen at different weight ratios, respectively were 3 wt.%, 4 wt.%, 5 wt.% and 35 wt.%. The process of addition could be divided into several steps while the base bitumen was being stirred. The mixing temperature was kept at 145 °C and the stirring speed was around 100 r/s. After the LDHs powder was added into the base bitumen. The mixing process was continued for another one hour. Then the LDHs modified bitumen was prepared.

3.2. Preparation of LDHs-PDB particles

According to the theory mentioned above, LDHs powder was added into 70# base bitumen with a weight ratio of 1:1 at 145 °C firstly. The process of addition could be divided into several steps while the base bitumen was being stirred. After all LDHs powders were added, the mixing process was continued for another one hour. The temperature was controlled at 145 °C during mixing and the stirring speed was around 100 r/s. After that, the

Table 3
The properties of cosolvent.

Tests	A	B	C
Reagent-grade	AR	AR	AR
Appearance	White powder	White powder	White powder
Solubility	Insoluble in dichloromethane and ethanol		
Relative dielectric constant	4.5	9.7	11.8