



Effects of microencapsulated phase change materials on the performance of asphalt binders

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ABSTRACT

This work aims at evaluating the physical properties, storage stability, and temperature-adjustable performance of asphalt binder modified with microencapsulated phase-change materials (micro-PCMs). Micro-PCMs with melamine–formaldehyde (MF) resin shells and n-tetradecane cores are fabricated through in situ polymerization. The MF resin shell successfully encapsulates the n-tetradecane core. The prepared micro-PCMs have spherical profiles, smooth surfaces, and particle diameters of 100 μm–200 μm. In addition, the micro-PCMs exhibit high phase-change enthalpy and excellent thermal stabilities. A neat asphalt binder is modified with various amounts of micro-PCMs modifiers. The addition of micro-PCMs does not considerably affect the physical properties of the modified asphalt. However, excessive micro-PCMs content will adversely affect the storage stability of asphalts modified with micro-PCMs. The temperature change rate of the micro-PCMs modified asphalt near the phase-transition temperature of the micro-PCMs decreases with the increase in micro-PCMs content. The specific heat capacity of the micro-PCM-modified asphalt shows a large peak near the phase-transition temperature of the micro-PCMs, and the peak value of the asphalt modified with micro-PCMs increases with increasing micro-PCMs content.

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1. Introduction

Asphalt has various advantages, such as relatively low cost [1,2], self-healing ability [3], recyclability [4–6], and good adhesion to mineral aggregates [7]. Given these characteristics, asphalt is especially well suited for applications in road paving. Asphalt pavements may show several distresses, such as high-temperature rutting, low-temperature cracking, and room-temperature fatigue cracking because they are subjected to various traffic loads and weather-induced stresses. These pavement distresses are highly related to the viscoelastic behavior of the bitumen used in the pavements. At high temperatures, asphalt pavements easily undergo rutting owing to the reduced stiffness of the asphalt. Meanwhile, the increased viscosity of bitumen plays a significant role in the cracking of asphalt pavements at low temperatures. Many researchers have attempted to overcome these problems by improving the properties of asphalt and bitumen mixtures [8–12].

The addition of modifiers to bitumen is a common approach to improving pavement properties. Numerous bitumen modifiers, such as nanomaterials [13,14], synthetic polymers [15–17], and biological polymers [18], have been reported to date.

The use of phase-change materials (PCMs) as a novel bitumen modifier has attracted increasing interest over the past few years. PCMs can store/release large amounts of energy from/to its surroundings at a constant temperature by undergoing a phase change. Given this characteristic, PCMs are efficient tools for thermal energy storage [19,20]. PCMs can be added to the bitumen or asphalt mixture as a thermoregulator to improve the temperature performance of the resulting pavement. Numerous researchers have studied the properties of bitumen and asphalt mixtures modified with PCMs. Kong et al. [21] prepared asphalt/lauric acid blends through direct impregnation technology. They found that asphalt/lauric acid blends show great potential for conserving waste energy and regulating indoor temperature. Manning et al. [22] investigated the feasibility of using lightweight aggregates as a medium for PCMs incorporation in hot mix asphalt. Their results indicated that the incorporation of PCMs reduces the extreme low temperature value and cooling/heating rate of a sample and

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decreases the time the sample is frozen. Ma et al. [23] proposed a heat-exchange system for testing real-time temperature change behavior and calculating the specific heat capacities of an asphalt mixture modified with composite shape-stabilized PCMs. The proposed system can be used to study the thermoregulation mechanism and efficiency of PCMs in asphalt mixture. In addition, as a type of modified asphalt material, PCMs mixed with asphalt concrete should possess good thermal reliability and should not leak from the mixtures during melting.

Microencapsulated PCMs (micro-PCMs) are PCMs in liquid/solid form enveloped within a polymer or inorganic shell [24,25]. It is because of the existence of the shell material, micro-PCMs can prevent PCMs from leakage and control the change in the storage material volume as phase change occurs. However, only few studies investigated asphalt binders modified with micro-PCMs. In this study, to prevent the leakage of PCMs during phase change, powder like micro-PCMs are prepared through in situ polymerization with a melamine–formaldehyde (MF) resin shell and *n*-tetradecane core. Furthermore, asphalt binders modified with micro-PCMs are prepared by mixing base bitumen with various weight fractions of micro-PCMs. The effects of different weight fractions of micro-PCMs on the properties and performance of asphalt have rarely been investigated. The physical properties, storage stability, and temperature-adjustable performance of the modified bitumen binder are thoroughly investigated.

2. Materials and methods

2.1. Materials

The 80/100 penetration grade (90# asphalt) paving binder was selected as the base binder. Base bitumen tests were performed in accordance with the Chinese standards JTG E20-2011 [26]. The performance results are presented in Table 1.

Micro-PCMs were prepared through in situ polymerization method. *N*-tetradecane (purity 99 wt%) was used as the core material. Melamine (purity 99 wt%) and 37% formaldehyde were used as shell-forming monomers. Tween 40 (A.R.) was used as a surfactant. Acetic acid (A.R.) and triethylamine (A.R.) were used to control pH during polymerization. All chemical materials were used without further purification.

2.2. Preparation of micro-PCMs

Micro-PCMs were prepared through in situ polymerization method in accordance with the following steps: A total of 4.0 g of melamine and 10.0 ml of formaldehyde were mixed with 14.0 ml of distilled water. The pH of the mixture was adjusted to 9.0 with triethylamine. The mixture was stirred at 60 °C for 60 min to form the prepolymer solution. A total of 6.0 g of *n*-octadecane, 5.0 g of Tween 40, and 100 ml of distilled water were mechanically

emulsified at 50 °C at the stirring rate of 5000 rpm for 45 min to form a homogeneous stable emulsion. Acetic acid was used to adjust the pH of the emulsion to 4. Then, the stable emulsion was transferred to a 250 ml three-necked round-bottomed flask. The prepolymer solution was added dropwise into the emulsion while the emulsion was stirred at a rate of 400 rpm. After all of the prepolymer was added to the emulsion, the emulsion was continuously stirred at 60 °C for 100 min. Thereafter, the resultant microcapsules were filtered and washed with ethanol. The wet cake was dried in a vacuum oven at 60 °C for 24 h to remove water. The process of producing micro-PCMs is shown in Fig. 1. The dried micro-PCMs are shown in Fig. 2.

2.3. Preparation of modified bitumens

Bitumen samples modified with various weight fractions of micro-PCMs were prepared as follows: First, the base bitumen and prepared micro-PCMs were heated to 150 °C in an oven and pre-served for 30 min. Then, the base bitumen was poured into a 2000 ml spherical flask. Micro-PCMs were then slowly added to the base bitumen at a low mixing speed. After pre-mixing, the blends were heated again to 160 °C and stirred for approximately 45 min at 4000 r/min to ensure the homogeneity of the blends. Bitumen samples modified with 1 wt% (P100), 3 wt% (P300), and 5 wt% (P500) micro-PCMs were thus fabricated.

2.4. Test methods

The Fourier transform infrared (FT-IR) spectra of *n*-tetradecane, MF resin, and micro-PCMs were recorded using KBr disk on a FT-IR spectroscope from 400 cm⁻¹–4000 cm⁻¹. Micro-PCMs and modified asphalt with 5 wt% micro-PCMs content were sputter coated with gold, and surface morphologies were observed through scanning electron microscopy (SEM). The phase-change properties of the micro-PCMs were characterized through differential scanning calorimetry (DSC) at a linear heating or cooling rate of ±10 °C min⁻¹ over a temperature range of –25 °C to 25 °C in a protective nitrogen atmosphere. The thermal stabilities of the micro-PCMs were examined through thermal gravimetric analysis (TGA) in a nitrogen atmosphere from 30 °C to 600 °C at a heating rate of 10 °C min⁻¹.

The physical properties, including penetration at 25 °C, softening point, ductility at 10 °C, and storage stability of the modified bitumens were tested in accordance with JTG E20-2011.

The temperature-adjustable performance test was performed with a double insulation barrel system, and temperature was recorded in real time using a data acquisition system (Fig. 3). The testing steps were as follows: (1) Bitumen samples were loaded into a cylindrical metal mold (φ101 mm × 87 mm, Fig. 4). A thermoresistance temperature sensor was checked and embedded in the middle of the mold. Then the upper part of the mold was sealed

Table 1
Technical properties of the base bitumen (90 penetration grade).

Properties	Results	Technical requirements [27]	Specification
Penetration (at 25 °C; 0.1 mm)	85.9	80–100	T0604-2011
Softening point (°C)	52.8	≥45	T0606-2011
Ductility (at 10 °C; cm)	82	≥45	T0605-2011
Density (at 25 °C; g/cm ³)	1.07	Measured records	T0603-2011
Dynamic viscosity (at 60 °C; Pa·s)	183	≥160	T0620-2011
After RTFOT (163 °C, 85 min)			
Mass loss (%)	0.19	≤±0.8	T0610-2011
Residual penetration ratio (at 25 °C; %)	67	≥57	T0604-2011
Residual ductility (at 10 °C; cm)	43	≥8	T0605-2011

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