



# Preparation and thermal properties of encapsulated ceramsite-supported phase change materials used in asphalt pavements

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## HIGHLIGHTS

- Two kinds of encapsulated ceramsite-supported phase change materials have been successfully prepared.
- E-PEG/CS has the better thermal reliability and chemical stability.
- The maximum peak temperature reduction of upper surface can reach 9.1 °C.

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## ABSTRACT

Two kinds of phase change materials (PCMs), poly(ethylene glycol) (PEG) and ethylene glycol distearate (EGD), were incorporated with ceramsite (CS) to obtain the composite PCMs (CPCMs) by vacuum impregnation method. The morphology of the samples was observed using scanning electron microscope (SEM) and the chemical compatibility was characterized by X-ray diffraction (XRD) and Fourier transformation infrared (FTIR). The PEG and EGD could be retained by 42.1 wt% and 34.0 wt% into pores of the CS, which was calculated according to the curves of the thermogravimetric (TG) analysis. The thermal properties and thermal reliability of the CPCMs were investigated via differential scanning calorimetry (DSC). The phase change temperature ( $T_m$ ) and heat of fusion ( $\Delta H_m$ ) of prepared CPCMs are in the range of 54–60 °C and 29–50 J/g, respectively. The  $\Delta H_m$  of the CPCMs decreased obviously after 100 melting/freezing cycling, which was improved by encapsulating the prepared CPCMs with novolac epoxy resins (NER) to produce the encapsulated CPCMs (E-CPCMs). The results of the leakage experiment and storage/release test also show that the E-CPCMs have good thermal exudation stability and thermal storage/release properties. Based on all the results of the experiment, the encapsulated PEG/CS composite (E-PEG/CS) was added into the asphalt mixture to substitute the corresponding aggregate for simulating the actual temperature-adjusting effects. The results indicate that the maximum temperature reduction of the upper surface reaches about 9.1 °C at 210 min. Therefore, the E-CPCMs have the potential to decrease the high temperatures and temperature fluctuations in the asphalt pavements.

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## 1. Introduction

Pavements are subject to the harsh environmental factors in atmospheres [1,2]. Among them, ambient temperature has a great impact on the physicochemical properties of the pavement materials [3]. The asphalt is a kind of temperature-sensitive material that exhibits elasticity at low temperatures and viscosity at high

temperatures [4]. In summer, the asphalt pavements significantly deform under the repeated vehicle loads when the temperature of the pavement surface approaches the softening point of the asphalt, resulting in rutting, shoving and some other distresses [5,6]. Simultaneously, the asphalt become hard and brittle due to the high/low-stress cycles caused by obvious diurnal temperature variations, leading to the thermal fatigue damages of the asphalt pavements [7].

Phase change materials (PCMs), also known as latent heat storage materials, release or absorb heat in almost isothermal

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conditions [8]. PCMs exhibit the great potential in terms of adjusting temperature and regulating the disequilibrium between energy supply and demand. They have been widely used in different applications such as textile, solar energy, buildings, aerospace and so on [9–11]. In recent years, many researchers have explored the application of PCMs into constructions of asphalt pavements. The extreme temperatures and temperature fluctuations in the asphalt pavements can be reduced to the alleviate the diseases caused by temperatures, extend the life of the pavements, and decrease the maintenance costs [12,13].

There are two main methods that PCMs can be incorporated into the asphalt pavements. The PCMs can be directly added into the asphalt pavements, but the chemical compositions of the asphalt change significantly. The aromatic and saturate fractions of the asphalt increase owing to the direct addition of the PCMs, resulting in the change of the colloidal structure of the asphalt [14–16]. Meanwhile, the possibility of deformation of the asphalt pavements increase because the PCMs added directly are prone to migrating when they are in a liquid state. The PCMs can also be compounded with microporous materials such as diatomite, expanded perlite and expanded vermiculite to produce composite PCMs (CPCMs) [13,17–19]. The CPCMs are blended into the asphalt pavements to replace some of the original fine aggregate to adjust the temperature of the pavements. Although this method improves the thermal stability of the PCMs, there is some mass loss of the PCMs after thermal cycles [20]. Moreover, the volume fraction of the fine aggregates in the pavement materials is small so that the volume fraction of the PCMs is limited.

In this study, the CPCMs are made by compounding PCMs with the CS which provide an excellent framework structure for the PCMs because of the advantages of excellent thermal properties, chemical stability and low cost [21,22]. The porous features of the CS is ideal for the impregnation and enhancement of the chemical and thermal stability of the PCMs [23]. What's more, the CPCMs are also encapsulated with novolac epoxy resins (NER) and the mass loss of the PCMs can be largely reduced. The microstructure, chemical compatibility, thermal stability, thermal properties, thermal reliability, exudation stability and the thermal storage/release properties were systematically studied.

## 2. Experimental

### 2.1. Materials

The ceramsite (CS) with particle size between 4.75 mm and 9.5 mm was obtained from Hubei Province of China. The crushing strength of the CS was 4 MPa and its density was 600 kg/m<sup>3</sup>. Poly(ethylene glycol) (PEG) (molecular weight of 6000) and ethylene glycol distearate (EGD) were of analytical grade. They were supplied by Shanghai Mackin Biochemical Co., Ltd. (Shanghai, China). NER whose epoxy equivalent weight ranged from 170 g to 200 g were used as encapsulating materials in this study. The softening point of the NER was between 20 °C and 30 °C. Curing agent 593 (CR593) was utilized as curing agent to accelerate the curing of the NER. The asphalt having 70/100 penetration grade was purchased from China Petroleum & Chemical Corporation and the aggregates were produced by crushed basalt mineral.

### 2.2. Preparation of CPCMs and E-CPCMs

40 g of CS was placed into a conical flask and then 60 g of different PCMs (PEG and EGD) was added to cover the CS. Afterwards, the conical flask containing samples was put in a water bath with the temperature of the water at 90 °C for about 45 min. The PCMs were melted completely and the vacuum pump was started. When bubbles no longer appeared around the CS, it was believed that the pores in the CS were substantially filled with the liquid PCMs. Subsequently, the vacuum pump was turned off and the resulting samples were placed on the 4.75 mm sieve to remove the extra liquid PCMs. After cooling, the preparation of CPCMs (PEG/CS and EGD/CS) was completed. 40 g of NER and 10 g of CR593 were mixed up in a beaker and set aside for 5 min. The mixture was then stirred slowly for 1 min and 50 g CPCMs was add in it. After stirring continuously for 2 min, the resultant products

were taken out. The encapsulated CPCMs (E-CPCMs) (E-PEG/CS and E-EGD/CS) were produced after curing of the encapsulating materials for 24 h. The Schematic view for the preparation of CPCMs and E-CPCMs is show in the Fig. 1.

### 2.3. Characterization

The surface morphology and filling situations of the samples were observed by SEM (model KYKY-EM8000F). The Fourier transformation infrared (FTIR) spectra of the CS, PCMs and CPCMs were measured with a FTIR spectroscopy (model Nicolet NEXUS 670), and when the wavenumber ranged from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. The X-ray diffraction (XRD) patterns were collected by a Rigaku D/max 2550 diffractometer with Cu K $\alpha$  radiation ( $\lambda = 0.154$  nm, 40 kV and 200 mA) and a scanning range from 5° to 80° at a scan speed of 0.02°/min. The thermal properties including  $T_m$  and  $\Delta H_m$  were evaluated by the differential scanning calorimetry (DSC) (TA instruments, Q10). The scan was conducted at a heating rate of 5 °C/min from 30 °C to 90 °C under nitrogen atmosphere flow. The thermal stability was measured by a thermal analyzer (model HCT-1) under nitrogen atmosphere. The heating rate was 10 °C/min and temperature ranged from ambient temperature to 900 °C.

### 2.4. Thermal reliability performance evaluation

The thermal reliability of the samples was determined by the accelerated thermal cycling test which included 100 melting/crystallizing processes. The melting process and crystallizing process were conducted at 90 °C and 20 °C, respectively. The samples after thermal cycling test were characterized by DSC and FTIR to evaluate the changes in chemical stability and thermal properties.

### 2.5. Leakage test

The exudation stability of the samples was tested by the self-designed leakage test. Circles with a diameter of 7 cm were drawn on the center of the filter papers with the colored markers. The same amount of samples was put on the circular area and then covered by inverting petri dishes over them. Hereafter, the samples were placed in 80 °C oven for 1 h. The exudation stability can be evaluated by observing the trace of the PCMs on the filters.

### 2.6. Thermal storage/release performance evaluation

A beaker was filled with 120 g of samples and a round foam board was covered on the samples. A digital thermometer was used to record the center temperature of the samples. Two water bath pots were prepared for the experiment and the water temperature were 30 °C and 80 °C, respectively. The thermal storage/release test consisted of three parts. Firstly, the beaker with samples was put in the 30 °C water bath until the temperature reached equilibrium. Then the beaker was migrated to the 80 °C water bath until the temperature reached equilibrium. Finally, the beaker was put in the 30 °C water bath again until the temperature decreased below 30 °C. The temperature was collected every 10 s to monitor the temperature changes.

### 2.7. Temperature-adjusting test of the asphalt mixture with E-PEG/CS

The temperature-adjusting test of the asphalt mixture with E-PEG/CS was conducted in a self-designed experimental apparatus which was shown in the Fig. 2 [17]. The apparatus was equipped with self-designed heat preservation boxes, xenon lamp, and temperature controlling and recording systems. The heat preservation boxes was 300 mm  $\times$  300 mm  $\times$  250 mm in dimension. The bottoms of the heat preservation boxes were filled with 20 mm thick soil and the specimens were located on the top of the soil. The surroundings of the specimens were stuffed with insulation materials. The upper boundary was exposed to the enclosed air and was directly below the xenon lamp. The bottom boundary was located on the top of the earth soil. The specimen A<sup>#</sup> and specimen B<sup>#</sup> in this experiment were 150 mm  $\times$  150 mm  $\times$  50 mm in dimension and they were prepared in accordance with Marshall design method. The detailed descriptions of the aggregate are listed in the Table 1. One quarter of the aggregate (4.75 mm–9.5 mm) was substituted with the same quality of E-PEG/CS in the specimen B<sup>#</sup>. The temperature measurement system includes temperature sensors and a temperature recorder. The temperature sensors are contact sensors and can be used to test the temperature changes of upper and bottom surface over time. As shown in the top view of the heat preservation box (Fig. 2a), four temperature-measuring points were evenly distributed on the upper boundary. The distribution of the temperature-measuring points on the bottom surface were the same as the upper boundary. A sensor was located at every temperature-measuring point. Therefore, eight sensors were located on the upper and bottom surfaces. The other four sensors were used to measure the temperature of the enclosed air. The temperature sensors are platinum (PT100) thermistor sensors supplied by Guangzhou Spike Environmental Instruments Co., Ltd. The temperatures were all reported from an average of four sensors. The temperature recorder has 24 channels, which can accurately capture and record the temperature at the same time. The temperature values can be saved for up to one month. Firstly, the specimen was put in a constant temperature environment

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