



# Preparation of expanded graphite/polyethylene glycol composite phase change material for thermoregulation of asphalt binder

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## HIGHLIGHTS

- An expanded graphite/polyethylene glycol (EG/PEG) composite PCM is prepared.
- The maximum mass ratio of EG and PEG in EG/PEG composites is 1:7.
- EG/PEG composites have favorable compatibility with asphalt binder.
- EG/PEG composites enhance the thermal conductivity of asphalt binder.
- EG/PEG composites regulate the temperature of asphalt binder effectively.

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## ABSTRACT

Phase change materials (PCMs) have been widely applied as heat storage and thermal regulation units in many areas. However, the utilization of PCMs in asphalt pavements for thermal regulation is still limited because of the low thermal conductivity and melted phase leakage of the common used solid-liquid PCMs. The aim of this paper was to prepare a shape-stabilized expanded graphite (EG)/polyethylene glycol (PEG) composite phase change material (EG/PEG) with high thermal conductivity, and to verify its feasibility of regulating temperatures of asphalt binder. EG and PEG were selected as supporting material and working substance respectively. The EG/PEG composites and their modified asphalt binders were fabricated respectively by vacuum adsorption and physical blending process. X-ray diffraction (XRD) and differential scanning calorimeter (DSC) analyses indicated that the crystallization ability and phase change behavior of PEG was hindered by the increase of EG in the composites. The nitrogen adsorption analyses revealed that the EG matrix had an adsorptive limitation to PEG. The aforementioned results indicated that the maximum mass ratio of EG and PEG in the composites without any leakage during the melting period was 1:7. This conclusion was also proved by scanning electron microscopy (SEM) and Fourier transform infrared (FT-IR) spectrum. The thermogravimetric analyses (TGA) verified the viability of EG/PEG composites during the hot mixing process of asphalt mixture. The thermal analysis experiments indicated that the thermal conductivity and compatible stability of asphalt binder were improved by adding EG/PEG composites, and the thermal regulation effects of EG/PEG composites on the asphalt binder was verified by a thermocouples proved.

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## 1. Introduction

Asphalt pavements have been widely applied for the construction of highway due to the merits of flat surface, low noise and high wear resistance. However, as a temperature-sensitive material, asphalt mixture is susceptible to rutting distress and thermal

cracking when the environmental temperatures are extremely high or low, and consequently the lifespan of asphalt pavements is shortened [1]. In order to prevent rutting of asphalt pavements, researchers have proposed numerous methods such as designing new pavements gradation [2], adopting polymer modified asphalt binders [3] and improving surface reflectance [4] and moisture [5]. In order to minimize distresses related to thermal cracking, fiber modified asphalt binders [6], surface-treated aggregates [7], emulsion asphalt slurry sealing [8] and asphalt overlay [1] have become the common selected treatments. Notwithstanding the effectiveness of the aforementioned techniques, shortcomings still

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exist on solving thermal distress completely in the practical utilization of pavements, and an innovative and effective approach to regulate asphalt pavements temperature is essential and urgent.

Phase change material (PCM) is a substance which can absorb or release heat when the material changes from solid to liquid and vice versa. When the phase change process happens, the material remains at a constant temperature or a narrow temperature range, and thus the PCMs can be employed as latent heat storage and thermal regulation units [9]. PCMs have been widely applied to the areas of textiles [10], buildings [11], food packaging [12], medical therapies [13], etc. The applications of PCM in asphalt pavements are also receiving more attention gradually. Xavier [14] incorporated n-tetradecane-type paraffin waxes with the solidification temperature of 2 °C or 5 °C into the surface courses of road pavements to reduce the number of freezing situations. Chen [15] studied the temperature-control mechanism and selection criteria of PCM used in asphalt pavements, and a kind of shape-stabled PCM was also fabricated. Ma [16,17] investigated a composite shape-stabilized PCM by using ethyl cellulose membrane and silica matrix, in order for the PCM survive in the construction and service process of hot-mix asphalt (HMA). Si [18] prepared three types of composite PCMs based on the consideration of temperature distress forms of asphalt pavements in order to solve temperature damages. Bryan [19] verified the feasibility of using lightweight aggregate (LWA) as a medium to incorporate PCM in HMA, with the purpose of reducing temperature fluctuations of asphalt pavements. Mohammad [20] sealed the phase change LWA with waterproofing membrane, in order to prevent liquid PCM leakage during the hot mixing process of asphalt mixtures. Michal [21] designed a new asphalt mixture by replacing one of the mineral aggregate fractions with PCM soaked LWA, the thermal stabilization and permanent deformation resistance of the modified asphalt pavements were evaluated.

The existing methods of PCM incorporation in asphalt mixture are mainly consisting of the direct immersion method, microencapsulation method and aggregate impregnation method. Each of these methods has their advantages, and the limitations are also distinct: direct immersion method is easy to perform, but the leakage often occurs after multiple thermal cycles [14,15]; microencapsulation method can protect working substance from external environment, however the thermal stability and heat-transmission efficiency of polymers cannot meet the temperature regulation requirement of asphalt pavements [17,22,23]; aggregate impregnation method can provide sufficient mechanical strength for PCMs, yet the absorption capacities and temperature regulating effects are limited [19,20,24,25]. Therefore, it is necessary to find a reliable and efficient method to incorporate PCM into asphalt mixtures.

Expanded graphite (EG) is an emerging carbonaceous material with excellent thermal conductivity, absorption capacity, thermal stability and compatibility. It can serve as PCM supporters to reduce liquid leakage as well as enhance heat transfer efficiency of PCM. EG-based composite PCMs have been applied in some areas [26–28], however, the feasibilities of using such materials to regulate the temperature, as well as to enhance the thermal transfer rate of asphalt binder, are still unknown. In the present study, EG was selected as supporting material and polyethylene glycol (PEG) was used as a working substance. The EG/PEG composites with different mass fractions were fabricated by adsorbing liquid PEG into the pore structures of EG, whilst the EG/PEG modified asphalt binders were produced by physical blending. The crystallization abilities and phase change behaviors of EG/PEG were measured by X-ray diffraction (XRD) analyses and differential scanning calorimeter (DSC), respectively. Then the pore structure of EG/PEG was assessed by scanning electron microscope (SEM) and  $N_2$  adsorption instrument. The thermal stability of EG/PEG

composites was characterized by thermogravimetric analyzer (TGA), and the interaction between EG and PEG was evaluated by Fourier transform infrared (FT-IR) spectrum. To evaluate the influence of EG/PEG composites on the thermal conducting rate and compatibility with asphalt binder, thermal constants analyses and storage stability tests were conducted by using a thermal constant analyzer. Fiber grating temperature transmitters were utilized to characterize the thermoregulating effects of EG/PEG composites on hot mix asphalt.

## 2. Experiment materials

A reagent grade PEG supplied by Sinopharm Chemical Reagent Co., Ltd (Wuhan, China) was used as phase change material without any purification. It had an average molecular weight of 2000 and melting temperature of 50–55 °C. EG was supplied by Qingdao graphite Co., Ltd (Qingdao, China), the average particle size of the selected EG was 150  $\mu\text{m}$  and its expansion ratio was 270 mL/g.

One base asphalt binder, AH-70 (KOCH Bitumen Co., Ltd., Wuhan, China), which is extensively utilized in China, was selected for this research. The basic properties of raw materials were presented in Table 1.

## 3. Research methods

### 3.1. Preparation of materials

#### 3.1.1. Preparation of expanded graphite/polyethylene glycol (EG/PEG) composites

The EG matrix was desiccate in vacuum oven at 80 °C for 16 h to eliminate moisture in order to measure the weight accurately. Then the PEG was dissolved in distilled water homogeneously at a temperature of 70 °C. Subsequently, the weighed EG was dispersed in PEG aqueous solutions with a physical mixing process at a constant speed of 300 rpm for 30 min, the mass ratios of EG and PEG were 1:5, 1:6, 1:7, 1:8, 1:9, respectively. Finally, the blends of EG and PEG aqueous solutions were placed in a vacuum pump at a temperature of 80 °C for 4 h, in order for the aqueous solvents to be vaporized and the PEG to be completely absorbed into the pore structures of EG. The final products with EG and PEG mass ratios of 1:5, 1:6, 1:7, 1:8, 1:9 were named as EP<sub>1</sub>, EP<sub>2</sub>, EP<sub>3</sub>, EP<sub>4</sub>, EP<sub>5</sub>, respectively. The schematic for the preparation of EG/PEG composites was demonstrated in Fig. 1.

#### 3.1.2. Preparation of EG/PEG modified asphalt binders

Based on the volumetric specific heat and density of AH-70 asphalt binder (1.902 MJ/m<sup>3</sup>·K and 1021 kg/m<sup>3</sup>, respectively) and the phase transition latent heat of the selected PEG ( $\Delta H \approx 180$  J/g) in this paper, it was determined that no less than 16.69 g of pristine PEG was required to achieve a minimum temperature variation of 5 °C for 300 g of AH-70 asphalt binders. Therefore, the required EP<sub>1</sub>, EP<sub>2</sub>, EP<sub>3</sub>, EP<sub>4</sub>, EP<sub>5</sub> in this research were 19.47 g, 19.07 g, 18.78 g, 18.54 g and 18.38 g respectively to achieve the same thermoregulation effects. The modified asphalt binders were prepared by means of mixing AH-70 asphalt binder with EG matrix, pristine PEG and different EG/PEG composites respectively. The blending process was conducted by a propeller mixer at a constant speed of 600 rpm for 15 min, and the experimental temperature was 130 °C. The AH-70 asphalt binder was named as A<sub>0</sub>, and the EG, PEG and different EG/PEG modified asphalt binders were referred to as A<sub>1</sub>–A<sub>7</sub>, respectively. The compositions of the modified asphalt binders were presented in Table 2.

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