



Use of calcium silicate-coated paraffin/expanded perlite materials to improve the thermal performance of cement mortar

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HIGHLIGHTS

- A new calcium silicate-coated expanded perlite based form-stable PCM is prepared.
- Thermal stability of CPEP is enhanced by cladding of calcium silicate.
- Secondary encapsulation contributes to improve ITZ structures.
- The physical interaction between paraffin mixture and EP is revealed by FT-IR.
- TESC's possess acceptable compressive strength and outstanding thermal performance.

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ABSTRACT

Here we demonstrate thermal energy storage cement mortar (TESC) developed by integrating cement mortar with a calcium silicate-coated paraffin/expanded perlite based form-stable phase change material (CPEP). The thermophysical properties, microstructure and spectroscopic characterization of the CPEP and the thermal and mechanical properties of the TESC were studied. MIP and SEM results show that paraffin mixture PCM is adsorbed uniformly into the pores of expanded perlite treated by hydrochloric acid. The thermal-cycling test results indicate the good thermal stability of CPEP by the cladding of three-layer calcium silicate. Differential scanning calorimetry (DSC) results reveal that the CPEP has phase change temperature and latent heat of 24.58 °C and 88.57 J/g, respectively, and the lower phase change temperature of CPEP compared to paraffin mixture is supported by FT-IR. Moreover, the CPEP was incorporated in cement mortar at 10%, 15%, 20% and 25%, by weight of sand. It is found that the compressive strength of TESC decreases with increasing CPEP content. However, they can still be regarded as building maintenance structure materials to use. Further, the ITZ structures between CPEP and cement substrate are focused on by SEM. In addition, the thermal energy storage performance of TESC's is revealed by the results of specific heat capacity test and heating test.

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1. Introduction

Cement-based materials have been widely used as building materials and are considered to be suitable for incorporation of phase change materials (PCMs) to achieve the dual purpose of energy conservation and building thermal comfort [1–4]. Usually, there are two methods proposed to incorporate PCMs into cement-based materials. Direct incorporation methods were previ-

ously adopted because of the advantages of easy fabrication and low cost, but the PCMs incorporated into cement-based materials prone to leak after several heating-cooling cycles [5]; the PCMs can also be impregnated into cement-based materials by using form-stable PCMs [6–8]. Taking the cost reduction and simplifying processes into consideration, form-stable PCMs are prepared by blending PCMs with inorganic porous materials, such as diatomite [9], vermiculite [10], attapulgite [11] and bentonite [12], expanded graphite [13], etc.

The expanded perlite (EP) is a kind of inorganic porous building materials with a certain strength. It can prevent the leakage of PCMs during phase change process when impregnated into EP

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[14–16]. Of the most commonly used inorganic porous building materials, the EP has been widely used for incorporation of PCMs. In the current studies, the different kinds of EP based form-stable PCMs were developed for prevention of PCM leakage in cementitious composites, and the thermal energy storage cement-based composites were prepared by integrating the EP based form-stable PCMs into cement matrix [17–19]. However, as a type of inorganic porous material, the encapsulation ability of PCMs still needs to be improved [20]. Otherwise, the leakage of PCMs is still a serious issue since PCMs cannot be completely encapsulated by inorganic porous materials including expanded perlite. And solid-liquid PCMs are more vulnerable to leak from the inorganic porous materials during heating-cooling processes [21]. In order to improve the encapsulation ratio of PCMs, a method of EP treated by hydrochloric acid was proposed to increase the inner surface area, porosity and surface activity in this work. Moreover, to further restrain the leakage of PCMs from form-stable PCM composites, the method of secondary encapsulation has been proposed recently. Li et al. [22] prevented the leakage of diatomite/paraffin phase change material by using hydrophobic nanosilica as surface coating material. Memon et al. [23–25] prepared the PCM composites by incorporating paraffin into different types of porous support materials and used epoxy coatings to package the PCM composite for leakage prevention. However, the incorporation of hydrophobic nanosilica and epoxy coatings has an unfavorable effect on the compressive strength of cement-based composites due to their hydrophobic characteristic of surface. Thus, it is urgent to explore a coating material for secondary encapsulation which has the advantages including easy fabrication, higher strength and good compatibility with cement substrate. On the basis of above considerations, a novel coating material, calcium silicate, is proposed as a shell structure to clad the EP incorporated with PCMs. Remarkably, the good compatibility between calcium silicate shell and cement substrate contributes to the good combination of the form-stable PCMs and cement substrate, thus reducing interfacial defects.

In this study, the expanded perlite based form-stable PCM (PEP) was prepared by adsorbing binary paraffin mixture PCMs into the pores of EP treated by hydrochloric acid. Meanwhile, an encapsulation procedure of three-layer calcium silicate coated shell was proposed to prepare the calcium silicate-coated expanded perlite based form-stable PCMs (CPEP). And the stability and the thermophysical properties of CPEP were investigated. Then, CPEP was incorporated into cement mortar by mechanical blending, for the preparation of the thermal energy storage cement mortar (TESC). The microstructure, spectroscopic and thermophysical properties of CPEP were characterized, and the mechanical properties, thermal conductivity and specific heat capacity of the TESC samples containing different contents of CPEP were also investigated. Moreover, the thermal energy storage performances of the TESC boards with different contents of CPEP were evaluated as well.

2. Materials and experimental methods

2.1. Raw materials

Paraffin mixture is composed of 52# paraffin and liquid paraffin. Their properties are shown in Table 1. The chemical components of EP are shown in Table 2. The cylindrical compress strength of EP was determined to be 33 kPa according to the Chinese National Standard GB/T17431.2-2010. NaOH (AR), hydrochloric acid

Table 1
Properties of paraffin.

	Melting temperature	Latent heat
52# paraffin	54.1 °C	140.5 J/g
Liquid paraffin	–135 °C	—
Paraffin mixture (mass ratio of 1:4)	28.47 °C	112.42 J/g

(10 wt%) and CaCl₂ (AR) were supplied by Harbin Xinchun Chemical Factory. The properties of potassium silicate solution are shown in Table 3. The P.O 42.5 ordinary Portland cement used in this work was purchased from Yatai Co. Ltd (Tianepai Cement, China, Harbin) with all properties meeting the requirement of GB175-1999. Tables 4 and 5 show the physical properties and gradation profile of medium sand, respectively; Grade I fly ash (FA) used in this study was produced in the Harbin No. 3 Generating Station and its chemical components are shown in Table 2.

2.2. Preparation and characterization of CPEP

2.2.1. Acid treated activation of EP

The pores of EP are always blocked up by impurities. In order to increase the inner surface area, porosity and surface activity, the microporous structure of EP is necessary to be activated for better adsorption properties. Two treatments of EP were taken before paraffin mixture adsorption: (1) only dehydration treatment, and (2) acid treatment after dehydrated. The processes of acid treatments are mentioned as follows: EP was first immersed in 10% hydrochloric acid solution for 12 h. Then NaOH was used to neutralize the EP until pH ~7 after acid treated, and the EP was dehydrated after washed. Thereafter, the EP was placed into a high temperature furnace to calcine at 500 °C for 2 h. Thus the flammable impurities and residual moisture were removed from the surface of the EP, and the pore structure of the EP was activated. The paraffin mixture adsorption capacity of the treated and untreated EP was evaluated to verify the activation effect of acid treatment. In particular, the treated and untreated EP with the same mass were weighed after adsorbing the paraffin mixture at 80 °C for 12 h, and the mass change would be recorded and compared.

2.2.2. Preparation of CPEP

A small amount of treated expanded perlite was immersed in the excessive binary paraffin mixture PCMs at 80 °C to ensure that the paraffin mixture can be well impregnated for 10 h. After that, the PEP was prepared successfully. In order to prevent the leakage of paraffin mixture, an encapsulation procedure of three-layer calcium silicate coated shell was proposed (Fig. 1). PEP was first placed into the potassium silicate solution for 24 h, and a layer of potassium silicate solution would be attached to the surface of the PEP. Thereafter, the PEP was impregnated in 2 mol/l CaCl₂ for 24 h and a layer of calcium silicate coated shell on the surface of the PEP was produced due to the reaction between potassium silicate and CaCl₂. After that, The PEP was taken into the potassium silicate solution for 24 h again to form the second calcium silicate layer on its surface, and then impregnated into 2 mol/l CaCl₂ for 24 h to form the third layer. At last, the prepared CPEP was put into a rapid curing box for 3d maintenance.

2.2.3. Testing methods for characterizing CPEP

The phase change temperature and latent heat of CPEP were determined by differential scanning calorimetry (DSC, LINSEIS PT10). The micromorphology of CPEP was observed using an environmental scanning electron microscope (FEI Co., Quanta 200). Fourier transform infrared (FT-IR) spectra of CPEP were tested on Spectrum One (PerkinElmer, USA). Raman spectra of CPEP were recorded with a Renishaw in Via Raman microscope (Renishaw Corporation, Britain). By a thermal conductivity measuring apparatus (XI'AN XIATECH, TC3000), the thermal conductivity of sample was measured on the tablet with size of 40 mm × 40 mm × 10 mm. The thermal-cycling tests containing 150 thermal cycles were carried out to monitor the mass change of the CPEP and PEP. The form stable PCMs of 50 g were put into the oven with the set temperature of 35 °C for 1 h, and then taken into the refrigerator with the set temperature of –15 °C for 1 h.

2.3. Fabrication and properties of TESC

2.3.1. Mix proportion

Mix proportion of control cement mortar (CM) is given in Table 6. Four TESC samples were prepared by adding the CPEP into the cement mortar followed by thoroughly mixing. The CPEP was used as sand admixture, and the replacement levels are 0%, 10%, 15%, 20% and 25%, by weight of sand. The CMs with varying replacement level of the CPEP added are named as TESC-10, TESC-15, TESC-20 and TESC-25, respectively.

2.3.2. Compressive strength test

The CM and the TESC samples were cast into several cubes using the mold with the dimensions of 70.7 mm × 70.7 mm × 70.7 mm and demoulded after 48 h. All the cubes were cured for 28 days in a standard curing room where temperature and relative humidity (RH) are 20 ± 2 °C and 98 ± 1%, respectively. The 28-day compressive strengths of the CM and the TESC samples were measured according to GB/T 17671-1999.

2.3.3. Microstructure analysis

Typical microstructures of TESC samples were observed by using SEM. Samples used were taken from TESC with 20 wt% CPEP (TESC-20) at 28 days.

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