



Enhancement on thermal properties of paraffin/calcium carbonate phase change microcapsules with carbon network



Tingyu Wang, Shuangfeng Wang*, Lixia Geng, Yutang Fang

Key Laboratory of Enhanced Heat Transfer & Energy Conservation, Ministry of Education, South China University of Technology, Guangzhou 510640, Guangdong, China

HIGHLIGHTS

- A sort of phase change composites with double-layer network was developed.
- The composites thermal conductivity and thermal stability were enhanced.
- The heat transfer areas were increased due to spindle microcapsules morphologies.
- A distinct carbon network structure was detected with 20 wt% expanded graphite.

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ABSTRACT

For latent heat storage with phase change materials (PCM), heat transfer rate and energy storage efficiency are often limited by the low PCM thermal conductivity. Therefore, this paper develops a sort of new phase change composites (PCC) with double-layer network to enhance the thermal conductivity and thermal stability. Different mass fractions of expanded graphite (EG) as heat transfer promoter were added in the spindle microencapsulated phase change materials (MicroPCM). The relationship between the PCC thermal conductivity and carbon network structure was investigated. The thermal conductivity was measured by transient plane source method. The carbon network structure of PCC was detected by energy dispersive spectroscopy. Temperature-regulated property was captured by infrared imager. As a result, distinct carbon network structure in PCC was observed with 20% mass fraction of EG, the corresponding thermal conductivity was increased up to 7.5 times of the pristine paraffin. Negligible change in thermal properties of the PCC was confirmed after 100 times thermal cycling and 7 days serving durability tests. The enhancement on thermal properties of the PCC is a promising route to achieve high energy storage efficiency targets of numerous thermal applications.

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1. Introduction

Thermal energy storage (TES) technologies have played a critical role in sustainable energy infrastructure. Application of phase change materials (PCM) to improve the efficiency of energy storage is under active investigations [1–3]. PCM can absorb or release thermal energy during phase transition at a constant temperature range [4]. It has shown potential to be applied in many fields, such as battery thermal management [5–6], industrial waste heat recovery [7], building energy conservation [8] and solar energy utilization [9]. However, traditional PCM generally suffer from leakage during the solid-liquid phase transition and low thermal conduc-

tivity (λ) that to some extent limited in practical applications [10,11].

Microencapsulation technique has attracted more and more attentions in recent years. Since it can avoid the leakage of PCM from their location and also increase the heat transfer areas [12,13]. Various shapes and structures of the microencapsulated phase change materials (MicroPCM) are generated by different preparation methods with different properties of the shell materials. It is well known that CaCO_3 has three crystalline polymorphs, i.e., calcite, aragonite, and vaterite, which correspond to different morphologies and fit for different application fields [14]. The morphologies of MicroPCMs with CaCO_3 shells depend on the surfactant concentrations and synthetic temperature, it showed spherical morphology when synthesized under 35 °C in our previous work [15]. The thermal properties of spherical MicroPCM are isotropic which result in better stability and dispersibility, while

* Corresponding author.

E-mail address: sfwang@scut.edu.cn (S. Wang).

the spindle ones possess larger heat transfer areas. Hence, they are suitable for different utilization fields according to their own advantages.

Moreover, MicroPCM can achieve a stable microstructure by wrapping pristine PCM inside organic/inorganic shells [16]. For the past decades, MicroPCM with polymer shells have been widely studied, including melamine formaldehyde resin [17], poly (methyl methacrylate) (PMMA) [18], polystyrene [19], calcium alginate [20]. They have good structural stability but low thermal conductivity [21]. In order to overcome this problem, some attempts have recently been taken to encapsulate PCM with appropriate inorganic shells, such as aluminum hydroxide (Al(OH)₃) [22], silicon dioxide (SiO₂) [23], germanium dioxide (GeO₂) [24], titanium dioxide (TiO₂) [25] and calcium carbonate (CaCO₃) [26]. In consequence, these MicroPCM with inorganic shells were observed significant improvement in thermal conductivity and thermal stability than that of the pristine PCM. Nevertheless, thermal conductivities of MicroPCM with inorganic shells are still inadequate for meeting many energy storage efficiency targets.

The heat transfer rate governing the power capacity, service life and energy storage efficiency is dominated by thermal conductivity [27]. For a certain quality of PCM, higher heat transfer rate result in more total thermal energy that could be stored/released within a unit time. In this way, energy storage efficiency can be enhanced by improving thermal conductivity. Hence, in order to accelerate heat transfer and improve energy storage efficiency, the necessity to further explore PCM with high thermal conductivity and high thermal stability is evident. A literature survey indicates that high thermal conductivity fillers or inserts are sought to achieve high energy storage efficiency [28]. Liu et al. [29] demonstrated that the heat storing/releasing rate is effectively enhanced by adding expanded graphite (EG) to the MicroPCM with melamine resin shell. Li et al. [30] focused on improving thermal conductivity by using the MicroPCM with urea formaldehyde resin shell that merged into modified carbon nanotubes. Yang et al. [31] strengthened the heat transfer through employing the MicroPCM with polymethyl methacrylate shell that supplemented with modified silicon nitride powders. Wang et al. [32] significantly enhanced thermal conductivity by 22 times with 20 wt% EG loaded via utilizing the MicroPCM with melamine formaldehyde shell. Wang et al. [33] enhanced thermal conductivity by incorporating reduced graphene oxide in the MicroPCM with silica shell. Thereinto, carbon materials are the best fillers for enhancing thermal conductivity since high thermal conductivity can be achieved with low density. Till now, researchers mainly focus on the methods, varieties and optimal mass fractions of adding high thermal conductivity fillers into pristine PCM or MicroPCM with organic shells. Each study has presented remarkable increase in the thermal conductivity, which is due to the inherent high thermal conductivity of carbon fillers. Nevertheless, it is more important to form an effective filler network to obtain high thermal conductivity with less filler content. However, few studies have deeply investigated the principles and methods of forming the filler network, especially based on the MicroPCM with inorganic shells.

In this paper, we reported an experimental investigation to show that the MicroPCM with calcium carbonate shell and loading with 20 wt% EG could significantly increase thermal conductivity by up to 7.5 times compared to the pristine paraffin, heat storing/releasing rates were accelerated effectively as well. The phase change enthalpy and thermal conductivities of the MicroPCM/EG composites changed less than 3% over 100 solid-liquid phase transition cycles and 7 days serving durability tests, which indicating the phase change composites (PCC) possess good thermal stability during thermal cycling. With these prominent thermal properties, the PCC designed by this work will be a potential candidate for the thermal storage applications.

2. Experimental

2.1. Materials

Paraffin (RT 42) was used as PCM provided by ZDJN PCMS Co., Ltd., China. Calcium chloride (CaCl₂) and sodium carbonate (Na₂CO₃) were acted as monomers of the MicroPCM shell materials. Sodium dodecyl benzene sulfonate (SDBS) was treated as surfactant. The above reagents were all of analytical grade and obtained from Tianjin Kemiou Chemical Reagent Co., Ltd., China. Deionized water was homemade.

EG was served as the high thermal conductivity fillers and prepared from expandable graphite powder by the microwave method. It was supplied by Qingdao Graphite Co., Ltd., China.

2.2. Preparation

Fig. 1(a) shows the procedures to synthesize the MicroPCM via self-assembly precipitation method. The preparation process was carried out in a 500 ml three-neck flask equipped with a thermostatic water bath (60 °C) and a mechanical stirring (300 rpm). Firstly, paraffin (10 g) was melted into liquid state and mixed with the surfactant solution that containing SDBS (1.046 g) in deionized water (60 ml). The solution was stirred for 20 min and paraffin droplets gradually aggregate to form spindle shapes. Next, an aqueous solution of CaCl₂ (5.55 g) in deionized water (70 ml) was added into the above mixture dropwise and followed with agitation for 4 h. At this stage, a stable oil-in-water emulsion was formed. Afterwards, an aqueous solution of Na₂CO₃ (5.3 g) in deionized water (70 ml) was added dropwise in the flask and stirring for 6 h. Finally, the precipitation reaction was completed and the resultant microcapsules were filtered, washed and dried at 50 °C for 12 h.

Fig. 1(b) illustrates the PCC containing 10 wt%, 20 wt% and 30 wt% EG (denoted as S1, S2 and S3 respectively) were prepared by conventional dispersion method. The as prepared MicroPCM and EG according to the proportion were mixed in the whirlpool mixer and agitated for 30 min. Then, the mixture were poured into a cylindrical stainless steel mould (Φ 20 mm). The cylindrical compressed MicroPCM/EG composites were formed by dry pressing of MicroPCM and EG powders. Ultimately, the PCC products were cut and polished to form sheets.

2.3. Characterization

2.3.1. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra was obtained using a Bruker Tensor-27 instrument on a KBr disk at room temperature. The specimens were mixed with KBr sheets and the wave numbers ranging from 4000 to 500 cm⁻¹.

2.3.2. Energy dispersive microscopy (EDS)

The carbon network structure of the PCC was observed by Oxford Inca Energy-350 energy dispersive X-ray spectrometers under the view of a SEM. The PCC specimens were polished with #2000 emery paper and sprayed coating with a thin layer of gold-palladium alloy.

2.3.3. Scanning electron microscopy (SEM)

The morphology of the MicroPCM and PCC were measured by JEOL JSM-7400F scanning electron microscope with 20 kV acceleration voltage.

2.3.4. Transmission electron microscopy (TEM)

The microstructures of the MicroPCM was tested by JEOL JEM-2100 transmission electron microscope. The samples were dis-

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