Solar Energy Materials and Solar Cells 171 (2017) 148–160

Contents lists available at ScienceDirect

Solar Energy Materials and Solar Cells

journal homepage: www.elsevier.com/locate/solmat

Preparation and characterization of hybrid nanocomposite embedded organic methyl ester as phase change material

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ARTICLE INFO

Keywords:
Hybrid nanocomposite
Nucleation
Phase change material
Thermal conductivity
Thermal energy storage
Thermal properties

ABSTRACT

In recent years, the utilization of organic phase change materials (PCM) is increasingly attractive especially for the storage of thermal energy in building fabric materials absorbing solar energy. From this perspective, the thermal properties of the new copper-titania hybrid nanocomposite embedded organic methyl ester phase change material (HNPCM) were explored experimentally, for different proportions of the hybrid nanocomposite (HyNC). The test results suggest that, the incorporation of the HyNC (from 0.05% to 0.5% by wt.) into the pure PCM has attributed for achieving chemical stability, congruent phase transition temperature (35.32 °C), good latent heat potential (109.14 kJ/kg) with reduction in the supercooling degree. The crystalline fins-like structured HyNC particles have effectively improved the thermal conductivity of the HNPCMs from 2.9% to 65.2% without sacrificing thermal stability up to 218 °C, for the aforementioned HyNC proportions. Furthermore, the HNPCMs exhibited appreciable heat storage and release characteristics in terms of reduced time consumption during freezing and melting by 77.87% and 70.89%, respectively. The improved thermal properties exhibited by the HNPCMs enabled them be considered as a viable candidate to be incorporated into the exterior fabric elements of the building absorbing the solar energy thereby; the temperature swings in indoor spaces can be regulated suitably.

1. Introduction

The growing demand for developing efficient materials to fulfil the immediate energy storage requirements in a variety of cooling and heating applications in buildings has been gaining momentum in recent years. The space heating and cooling needs in buildings can be effectively catered through the utilization, absorption and storage of the high grade solar energy.

While focusing onto the space heating and cool storage applications in buildings, in majority, the latent heat storage (PCM) techniques have been largely preferred over the sensible and the thermochemical heat storage methods. There are a number of reasons behind, but on an overall, from the viewpoint of energy storage density, the PCMs have been into the real-time utilization in buildings, either in active or passive means of thermal storage [1]. From this perspective, the research works emphasizing on incorporation of the phase change materials (PCMs) into the building fabric elements are being increasingly attractive, in the last two decades [2].

The PCMs are known for their ability to store and release the thermal energy by virtue of changing their phase (from solid to liquid and vice versa) at nearly isothermal conditions with respect to their fusion temperatures. PCMs incorporated into the construction and building fabric components would allow for achieving reduced temperature swings in indoor spaces and increase the energy efficiency of the building by several folds.

In the class of PCMs including salt hydrates, carboxylic acid (fatty acid)-based materials and the eutectics, the attributes of a wide range of the organic and organic ester PCMs incorporated with thermal enhancing materials have been investigated thoroughly and given due importance in recent years.

There are numerous studies being performed in the past couple of decades, especially from the application point of view of the organic PCMs towards reducing energy consumption in buildings [2]. To emphasize, by incorporating the PCMs into building integrated components, the heating and cooling loads can be reduced, significantly.

In addition, the selection of the PCMs, with respect to their fusion temperatures being in-line with the indoor air temperature swings, could be one of the major challenging aspects for optimizing the TES attributes of the PCMs. The developmental studies on the encapsulation and shape stabilized PCM techniques would possibly help in addressing

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http://dx.doi.org/10.1016/j.solmat.2017.06.031
Received 6 January 2017; Received in revised form 20 May 2017; Accepted 18 June 2017
Available online 01 July 2017

the challenges pertaining to the operational aspects of the PCMs for different types of solar heating applications in buildings.

The technical revelations accentuating the design, integration, operational aspects and evaluation of a wide range of the PCMs dedicated for passive and active solar heating and cooling applications in buildings have been gleaned from numerous research studies and presented comprehensively [3].

The development of passive solar wall components comprising of the organic PCMs have also been reported [4–6]. The developed solar wall PCM composites exhibited good thermal storage performance in terms of improved thermal properties and thermal management for regulating the comfort conditions in indoor environments. The broad overview described the state-of-the-art TES technologies including organic PCMs as efficient methodologies for both meeting out the energy demand and enhancing energy efficiency in buildings as well, in a more sustainable way by using the solar thermal energy.

In the spectrum of incorporating the PCMs into the building materials, the PCMs would either be macroencapsulated (blended together with the building materials precisely), or microencapsulated (usually the PCM as core material and the inorganic polymeric material as shell) or nanoencapsulated (normally the PCM as core material and the inorganic polymeric material as shell at nanoscale level) [6–8].

The encapsulation methods carried out at different macroscale or microscale or nanoscale levels have revealed their significance in terms of the functional aspects and heat storage potential of the PCMs. The encapsulated PCMs would undergo the desired phase transition processes only within the shell material, which actually protects the PCM from getting leaked out [9,10].

However, in addition to the above, the incorporation of the thermally conductive nanomaterials into the pure PCM have facilitated them to relatively perform better, while compared to the macro and microencapsulated PCMs. The high surface-to-volume ratio, interfacial effects, thermo-kinetic and heterogeneous nucleation properties of the nanomaterials are expected to augment the heat transfer and thermal storage characteristics of the PCMs.

From this standpoint, there were a good number of nanomaterials (including silver, copper, carbon-based, silicon-based) incorporated PCMs been prepared, analyzed and developed for the heat storage applications in buildings, in recent times [11–14].

In the present work, the thermal energy storage properties of a new organic ester phase change material embedded with the copper-titania (Cu-TiO_2) hybrid nanocomposite (HyNC) were investigated experimentally. The organic ester PCM tested in this study was methyl cinnamate (MC), which is the methyl ester of cinnamic acid. The methyl cinnamate is a non-hazardous white to light yellow solid with a strong aromatic or fruity balsamic odour, which has been utilized as a fragrance ingredient in many cosmetics and non-cosmetic product applications [15].

In the spectrum of variety of organic PCMs utilized in buildings in recent years, their application is largely based on wallboards, internal partition wall etc., for achieving the required temperature regulation in indoor spaces [15–17]. Thus, the nucleus of the present work was focused on exploring the thermal energy storage properties of the methyl cinnamate PCM incorporated with different mass proportions of the HyNC for catering the cooling requirements in building envelopes absorbing the solar energy.

The prime scope for the development of the new HNPCMs in this study can be realized in terms of their effectual thermal energy storage properties being exhibited. This can be helpful in suggesting them as potential candidates to be incorporated into the exterior fabric elements of the buildings absorbing solar energy, and for satisfying the temperature regulation requirements in indoor spaces.

The characterization and testing in terms of surface modifications, crystalline nature, chemical stability, phase change temperature, latent heat potential, thermal conductivity, thermal stability and TES attributes of the HyNC and the HNPCMs were investigated experimentally.

Table 1
Summary of the physico–chemical properties of the pure methyl cinnamate (MC) PCM.

Description	Methyl cinnamate (MC)
Class	Organic ester phase change material
Synonym	Methyl cinnamylate; Methyl 3–phenyl–2–propenoate; 2–Propenoic acid, 3–phenyl–; Methyl ester
Molecular formula	$C_{10}H_{10}O_2$
Physical state and appearance	Crystal/crystalline
Odour	Fruity–like
Molecular weight	162.19 g/mol
Colour	White to light yellow
Melting point	33–38 °C
Density	1.041 g/cm^3
Solubility	Insoluble

The effects of the HyNC being embedded with different proportions (0.05% to 0.5% by wt.) into the pure MC PCM to accomplish the required enhancement in thermal properties and TES performance were signified through the experimental results and the test are presented.

2. Experimental

2.1. Materials

The precursors used in this HyNC preparation were copper acetate and titania procured from Sisco Research Laboratories (SRL) and Qualigens Fine Chemicals-Fischer Scientific. Ascorbic acid used as the reducing agent was purchased from Sisco Research Laboratories (SRL). The methyl cinnamate used as the PCM was procured from Alfa Aesar. The physico–chemical properties of the pure methyl cinnamate PCM are summarized in Table 1.

Ethanol as the dispersant and deionized double distilled water (DDW) collected from Millipore distiller was utilized as solvent-cum-cleaning medium for all the reactions. The designated chemicals used in this work were of the analytical grade, and have been used without further purification.

2.2. Preparation of HyNC and HNPCM

The facile preparation of the copper–titania (Cu–TiO_2) hybrid nanocomposite consisted of the following steps: (1) Dispersion of the titania (5 g) particles in ethanol solution was carried out by ultrasonication method (using UP200S-Hielscher instrument) for 30 min,(2) To this solution, the copper acetate (0.5 g) dissolved in warm DDW water was mixed under continuous stirring at 350 rpm, (3)The aqueous solution containing adequate quantity of the ascorbic acid and sodium borohydride reducing agents was prepared, (4) To the mixture solution obtained in step (1), the aqueous solution prepared in step (2) is mixed drop wise and left for 120 min at 45 °C, 350 rpm and ambient pressure until the reaction was completed, (5) The subsequent washing and filtration of the HyNC colloids as obtained from step (3) followed by vacuum drying eventually yielded the Cu–TiO_2 HyNC in the powder form.

In the steps of preparation of the nanostructures incorporated PCM, the as–prepared copper–titania (Cu–TiO_2) HyNC was embedded into the pure MC PCM through the ultrasonication re–dispersion method. The concentration of the Cu–TiO_2 HyNC was varied from 0.05% to 0.5% by weight using the electronic weighing balance (Shimadzu Ax-200 with internal calibration, precision: 0.0001 g). The schematic representation and the pictorial views of the preparation of the HyNC particles and HNPCM samples, respectively, are depicted in Fig. 1 and Fig. 2(a-b).

2.3. Characterization and experimentation

The morphological, chemical and thermal properties of HyNC and