Preparation and Thermal Characterization of Cu-TiO₂/ Stearic Acid Composite Mixture as Phase Change Material for Solar Energy Storage

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Abstract—In this study, Cu-TiO₂/stearic acid composite mixture was synthesized, characterized and experimentally tested. The composite mixture was prepared by mixing different weight percentage Cu-TiO₂ hybrid nanoparticles in stearic acid. The dispersion process is facilitated with ultrasonicator. The Cu-TiO₂ hybrid nanoparticles were characterized with Scanning Electron Microscopy, Fourier Transform Infrared spectroscopy analysis was used to determine the chemical stability and composition of composite mixture. LFA 467 hyperflash flash diffusivity apparatus was used to measure the enhancement in thermal conductivity for the additions of 0.3, 0.8, wt% of Cu-TiO₂ hybrid nanoparticles. For 0.8 percent addition of Cu-TiO₂ hybrid nanoparticles, the thermal conductivity was increased by 48 percent, compared to pure stearic acid. The result shows that the addition of hybrid nanoparticles into the stearic acid increases the solidification and melting time, thus confirming the increase in thermal conductivity. The improved thermal characteristics make the composite mixture as a potential candidate for solar thermal energy applications.

Keywords: phase change materials, Thermal Properties, Thermal energy storage, Nanoparticles, Thermal conductivity.

1. INTRODUCTION

The research for energy conservation has been consistently increasing, due to the increase in gap between the energy demands. In the upcoming years, Countries like India are expected to be more dependable on renewable energy and through continuous research in energy management and adopting various energy storage techniques like TES in buildings; the energy load in buildings is expected to decrease significantly. The Thermal energy storage systems were cost effective and greener solution than other energy saving techniques [1,2]. Solar energy techniques suing phase change materials play an important role in reduction of greenhouse gases [3]. Out of several thousand options of phase change materials available, fatty acids are used widely throughout the world. The phase change materials are not widely used through the world due to their low conductivity and low heat transfer characteristics. These drawbacks are rectified by the addition of nanoparticles [4].Due to high surface-to-volume ratio, , thermophysical properties, Brownian motion, diffusion effects of the nanomaterials, embedded into the pure PCM, the heat transfer interactions within the PCM matrix layers is increased. There are many nanomaterials (including silver, copper, carbon-based) incorporated PCMs been prepared and tested for thermal energy storage applications [5]. Numerically and experimental investigation of nano dispersed phase change materials in buildings for heating and cooling applications has been conducted by Kalaisevam et al., The investigation results showed that the mixture can be an alternative to the conventional latent thermal energy storage system. Among the several PCMs, fatty acids were widely used because of the advantages of low supercooling, high heat capacity, good thermal and chemical stability. This newly prepared PCM can be a potential candidate for solar thermal energy storage applications due to its enhanced conductivity and heat transfer characteristics.

2. MATERIALS AND EXPERIMENTAL METHODS

2.1 Materials

The precursors used in this preparation were copper acetate and titania procured from Sisco Research Laboratories (SRL) and Qualigens Fine Chemicals-Fischer Scientific. Ascorbic acid purchased from Sisco Research Laboratories (SRL) was used as the reducing agent. Ethanol as the dispersant and deionized double distilled water (DDW) was utilized as solvent-cum-cleaning medium for all the reactions. The chemicals used in this work were analytical grade, and have been used without further purification.

2.2 Preparation of Hybrid nanoparticles and HyNc

The preparation of Hybrid nanomaterial and Hybrid nanocomposite were explained in the Figure.1. The coppertitania (Cu–TiO₂) hybrid nanocomposite is prepared by dispersing the titania (5 g) particles in ethanol solution. The process was carried out by using ultrasonication method (using UP200S-Hielscher instrument) for 30-45 minutes. The copper acetate (0.5 g) dissolved in warm DDW water was mixed under continuous stirring to the dissolved solution. The ascorbic acid and sodium borohydride were added as a reducing agent to get colloids. The filtration of the colloids as obtained from followed by vacuum drying eventually yielded the Cu–TiO₂ HyNC in the powder form. The nano composite powder is weighed 0.8 percentages and embedded into the stearic acid through the ultrasonication re–dispersion method.



Fig. 1: (a) Schematic representation for the preparation of the HyNC and HNPCM

2.3 Experimental

The morphological, rheological, chemical and thermal properties of Hybrid nano particles and Hybrid nanocomposite were characterized and investigated using X-ray Diffraction, Scanning electron Microscopy, Difefrential scanning calorimetry, Laser Flash Analysis. The heat transfer characteristics of HyNC and HNPCM were investigated using the experimental setup explained in Figure.2.



Fig. 2: Schematic representation of the PCM testing system.

The testing system contains a VCRS cooling unit, testing tank and thermocouples connected to data acquisition system.

3. RESULT AND DISCUSSION

The 3 dimensional FESEM images clearly showed the formation of hyrid nanoparticles. The polydispersed quasispherical HyNC particles, with copper nanoparticles adsorbed on the surface of titania nanoparticles as shoen in Fig. 3. The formation and arrangement of the HyNc was further confirmed by the XRD measurement results as presented in Fig. 4. Precisely, the sharp characteristic peaks observed in the XRD pattern infer that the highly crystalline copper nanoparticles were formed over the titania nanoparticles. The characteristic peaks obtained at the corresponding planes were observed to be in good agreement with the JCPDS standards [7].



Fig. 3a: Morphology of Hybrid nanoparticles using FESEM

As seen in Fig. 5, There were no new peaks found in the FTIR spectrum, for the corresponding addition of hybrid nanoparticles This signifies that the interaction between stearic acid, hybrid nanoparticles is physical and there is no chemical reaction between the nanoparticles and PCM making the composite chemically reliable.



Fig. 4: XRD pattern of Hybrid nanoparticles

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Fig. 5 FTIR spectroscopy of Pure and HyNc

The latent heat and phase change peak temperature of melting and solidification is given in Table.1. Addition of nanoparticles reduced the supercooling as shown in the DSC in Figure.6. There were changes observed in the latent heat of HYNC, Phase transition temperature corresponding to the addition of nanoparticles. These changes were detected due to the Physio-chemical reaction between the nanoparticles and the PCM. The reduction in solidification and melting latent heat did not cause any severe problem in the charging and discharging process. For solar energy storage systems, latent in the range of 100-125 is preferred.



The heating and cooling is completed in a single step as there is a complete charge and discharge of the Phase change material. From the previous literatures, the addition of nanoparticles is limited to 0.8 weight percentage. Further addition resulted in agglomeration which affected the increase in thermal conductivity and also the effectiveness of the HYNC thus limiting the concentration of HYNC to 0.8 mass% in the experimental investigations. The viscous dissipation effects, reduction in melting and freezing latent heat and other cascading effects resulted in limiting the addition of nanoparticles to an optimum level of 0.8 mass% . The thermal conductivity of pure PCM and HyNc were measured using LFA 467 hyperflash flash diffusivity thermal analyzer. As the thermal conductivity varies with the temperature, measurement was done on the same temperature for all the samples. The samples were tested at 30°C with Graphite spray was coated on both the sides of sample holder for complete absorption of all the energy coming from the heat source. The thermal conductivity of pure stearic acid at 25°C was found to be 0.201 W/mK and increased to 0.298 The heat transfer characteristics of pure stearic acid and HyNc were tested in the experimental setup and depicted in Fig. 7a and 7b.





Fig. 7. (a) Heat transfer characteristic during melting cycle. (b) Heat transfer characteristics during solidification cycle.

 Table 1: Heat storage and release characteristics of pure and HvNc

Nano	1latent heat		` peak temperature /°C	
particle Mass/ %	Cooling J/g	Heating J/g	Cooling	Heating
0	166.2	166.5	51.43	58.29
0.8	161.4	162.1	54.91	53.89

The total time taken for complete melting varied from 1809, 2040 seconds for pure and 0.8 wt% HyNc. The time taken for complete solidification varied from 2375, 2488, seconds for pure and 0.8 wt% HyNc. The experimental results indicated that there was a reduction in melting time and solidification time. This result affirmed the increase in thermal conductivity of HYNC. Increase in thermal conductivity resulted in heat transfer enhancement as the HYNC charged and discharged at a faster rate than the pure PCM. The reduction time for melting and solidification process increased with increase in mass% of Cu-Tio₂ nanoparticles up to 0.8%, but further more addition of nanoparticles was expected to cause considerable reduction in latent heat, which would affect the performance of the HYNC.

4. CONCLUSION

In the present work, a newly prepared stearic $acid/Cu-Tio_2$ HyNc was tested for its heat storage capabilities and other thermal properties. The experimental results of HYNC have led to the following conclusions: The synthesized hybrid

nanoparticles enhanced the heat transfer characteristics of the PCM. The FTIR results have proved that there is only physical reaction between the nanoparticles and the base PCM while no chemical reaction exists in between them. This increases the chemical stability and the life of the HYNC. The thermal conductivity of the HyNc was increased by 48%. The melting and solidification time of the HyNc was reduced considerably when compared to pure stearic acid. This result shows the effect of nanoparticles in the phase change materials establishing that it is a vital player in the thermal energy storage systems.

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