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To cite this article: AK Pandey et al 2023 IOP Conf. Ser.: Earth Environ. Sci. 1261 012015

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Investigation of Thermal Performance and Chemical Stability of Metal Infused Salt Hydrate Phase Change Material for **Thermal Energy Storage**

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Abstract. Thermal energy storage (TES) is a technique that is considered a very desirable technology with a high potential to overcome the gap between demand and supply. Phase change materials (PCMs) are considered to be a highly favourable thermal energy storage materials. Besides that, PCM has certain drawbacks, such as lower thermal conductivity and higher light transmission; owing to this, the heat transfer rate and energy storage density are less. The performance of the PCMs can be improved by adding highly conductive nanoparticles. In this study, various weight percentages (0.1% - 5.0%) of Copper (II) oxide nanoparticles are dispersed with salt hydrate phase change materials with melting temperature 50 °C and investigated the thermal and chemical properties using Thermogravimetric analyzer, thermal conductivity analyzer, and Fourier transform infrared spectrum. Results shows that the prepared nanocomposites have chemically and thermally stable up to 467 °C. The thermal conductivity was increased by 62.64%, at 3.0 wt% Copper (II) oxide with salt hydrate PCM. The developed nanocomposites have better thermophysical properties than pure salt hydrate, which may be applied for TES applications like solar water heating, photovoltaic thermal systems, and electronic cooling systems.

1. Introduction

Owing to the current ecological and energy scarcity, there is a change in government policies and industrial policies towards sustainable, renewable energy sources. Solar energy is the highly believable, environmentally friendly, clean, and affordable energy source of all other renewable energy [1]. The earth is received by solar energy (1.8*1011 MW), and the received energy is a hundred times higher than the power being consumed from all other resources [2]. Solar energy comes from the sun in the kind of light and heat. Photovoltaics (PV), systems directly converted solar energy in to electrical energy

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and thermal energy, the electrical power can stored in the battery, but the heat energy cannot be stored and is wasted to the atmosphere [3]. In the thermodynamic view, all heat engines generate work output and reject some heat energy. This rejected heat energy is not employed for any other useful applications. Investigators have found the waste energy used for useful applications like solar water heaters, thermal power plants, desalination, thermal management in batteries, etc. [4]. For efficient use of thermal energy in useful applications, there is a necessity to develop a thermal energy storage (TES) system.

Phase change materials (PCMs) are latent heat (LH) storage substance able of accumulating and freeing of large amount of thermal energy. However, PCMs' lower thermal conductivity limits their utility as thermal energy storage [5]. Due to this, PCMs are a less preferable material for storing solar thermal energy. Researchers [6], suggested that doping of high conductive nanomaterials such as metals, fins, carbon nanotubes (CNTs), and metal oxides into the PCM increases the thermal conductivity [7]-[8]. Dispersing higher conductive nanoparticles (NPs) into the PCM increases the thermal conductivity and alters other thermal performance. The incorporation of highly conductive NPs into the PCM is referred to as nano-enhanced phase change materials (NePCM). The change in thermophysical properties of the NePCM be liable on size, structure, concentration, and morphology [9]. Moreover, thermal conductivity mainly depending on the shape of particles, like sphere, rectangular, and cylinder, due to more surface area cylindrical particles preferred for preparing nanocomposites [10].

Recently, graphene-dispersed erythritol PCM has explored by Mayilvelnathan et al. [11]. The findings discovered that the thermal conductivity improved by 53.01% and LH decreased by 6.1% by adding 1.0 wt% graphene; also, the addition of graphene materials reduces the supercooling. Moreover, very less changes in LH capacity and melting point, after 100 thermal cycles. The thermal and physical characteristic of stearic acid was modified by dispersing TiO₂ NPs performed by [12]. Recently, a researcher reported that accumulation of binary TiO₂/graphene incorporated organic PCMs thermal conductivity improved by 179% [13]. In this research, the various percentage of TiO₂ dispersed in stearic acid using sedimentation balance techniques. It was noticed that the prepared nanocomposites thermal conductivity was increased by about 27% than base stearic acid. Also, observed that tremendous thermal stability and chemical stability after 250 thermal cycles. Moreover, exceptional thermal reliability and LH were noted after 200 heating and cooling cycles. The effect of multiwall carbon nanotubes (MWCNTs) and aluminum oxide (Al₂O₃) dispersed organic nanocomposites was studied by Aqib et al. [14]. Pandey et al. [15], performed thermophysical characterization of Polyaniline/A70 PCM. PCMs are highly effective in acting as TES batteries, with higher thermal conductivity; this thermal conductivity determines the charging efficiency of the PCMs. Numerous researchers work on different NPs incorporated PCMs to improve the thermophysical properties for TES performed.

However, from the above literature found that salt hydrate with Copper (II) oxide (CuO) composite PCM is hardly ever found in recent studies. This will motivate us to expansively investigate the performance of nano CuO being used as the NPs in salt hydrate PCM for enhancing the thermophysical properties. This research becomes novel to explore the opportunity for finding the potential use of nano dispersed PCM (CuO/salt hydrate) in solar applications. The foremost objective is to experimentally analyze the chemical permanence, thermal conductivity and thermal stability of PCM by adding CuO NPs at six different weight concentrations (0.1, 0.5, 1.0, 2.0, 3.0, and 5.0 wt%). The chemical stability, thermal conductivity and thermal stability was evaluated by Fourier Transform Infrared (FTIR) spectroscopy, thermal conductivity analyzer and Thermogravimetric Analyzer (TGA). Also, the obtained experimental results of NePCM were compared with pure salt hydrate PCM. The present research is ordered as follows; section 2 demonstrates the materials and techniques used to prepare and characterization of the NePCM, the thermal, chemical stability and thermal conductivity were characterized and described in section 3, and section 4 deliberated the conclusion.

2. Materials and methods

The salt hydrate S50 PlusICE is used in the current study as base PCM. The in-organic PCM was obtained from the Phase change materials products Ltd., UK. The melting temperature and LH of PCM

are 50 °C and 100 kJ/kg, respectively. The Copper (II) oxide nanoparticles (CuO) 30-50 nm size was supplied by Alfa Aesar.

2.1. Preparation of NePCM

The inorganic PCM-added CuO nanocomposite was prepared using a two-step approach. In this study, we varied the concentration of CuO NPs by changing the percentages used (0.1, 0.5, 1.0, 2.0, 3.0, and 5.0 wt%). Salt hydrate was taken in a beaker and kept on hot plate, heated to a temperature above melting while being stirred. The necessary wt% of CuO added with the liquified PCM. The salt hydrate/ CuO mixture was sonicated (FS-1200N) for about 60 min. Throughout the process, the hot plate is kept above the melting point of the PCM to get well dispersion stability and prevent agglomeration of nanoparticles, after ultrasonication allows to solidify the NePCM.

2.2. Characterization of NePCM

Thermogravimetric analyser (TGA) was used to analyze the prepared samples decomposition temperature or thermal stability. TGA was performed using TGA-4000, Perkin Elmer, and samples were taken in a 180µl crucible and exposed to 10°C/min from atmospheric temperature to 650°C under N₂ atm. FTIR spectrum was used to perform the chemical structure of developed PCM nanocomposite, the FTIR spectrum 2, Elmer Perkin apparatus was used with wave number from 400 cm⁻¹ to 4000 cm⁻¹. The thermal conductivity of NePCM was examined by TEMPOS, at room temperature with error of ±5%.

3. Results and discussion

3.1 Thermal stability analysis

The formulated nanocomposites decomposition temperature was examined by TGA (TGA-4000, Perkin Emer). Figure 2 illustrates the decomposition temperature of pure PCM and its composites with various weight fractions (0.1-5wt%). The inorganic salt is decomposed into 2 stages; in the first stage, most of the water particles are decomposed around 295 °C, and in second stage, the magnesium nitrate salts are decomposed, and remaining is some oxides and residues. The results shows, the pure salt hydrate PCM starts the weight loss at 66 °C and the loss is around 27% at 291 °C, which is associated with loss of water particles. The rest of the PCM are decomposed at 455°C, the leftover 9.54% which is residues and some oxides, the same was mentioned by Wu et al. [16]. Also, after heating up to 650 °C, the leftover percentage is 8.65%.



Figure 1: TGA results of salt hydrate PCM and CuO dispersed PCM composites

International Postgraduate Conference for Energy Research 2022 (IPCER-2022)		IOP Publishing
IOP Conf. Series: Earth and Environmental Science	1261 (2023) 012015	doi:10.1088/1755-1315/1261/1/012015

In nanocomposite, the decomposition initiated at 72, 67, 71, 72, 73, 73 °C and ended at 464, 465, 459, 467, 476, 466 °C for S50C-0.1, S50C-0.5, S50C-1, S50-2, S50C-3, and S50C-5 respectively. In the initial stage, the water particles are decomposed at 298, 283, 281 283, 282, 270 °C, and the weight loss is 20, 22, 20, 21, 19, and 18%, respectively. The weight left after the decomposition of magnesium nitrate is 12.5, 9.58, 11.7, 12.9, 13.6, and 18%. Also, the final weight after 650 °C is 11.19, 8.65, 10.46, 11.2, 12.12, and 16.41% for S50C-0.1, S50C-0.5, S50C-1, S50C-2, S50C-3, and S50C-5 respectively. From the results observed that with the dispersion of CuO into the S50 PCM, the starting and ending of decomposition are considerably increased. Due to higher decomposition temperature, the prepared nanocomposites have excellent thermal stability because of the increase in onset and final decomposition temperatures. The reason for the improved stability is the formation of a thermal barrier in the nanocomposites. The thermal barrier formation is due to the dispersion of CuO into the PCM, the thermal decomposition temperature for all composites is higher than salt hydrate PCM. Due to higher thermal stability, nanocomposites are an excellent material for TES applications.

3.2. Chemical stability analysis

FT-IR spectra is used to measure the chemical structure of prepared nanocomposites. The functional group of pure salt hydrate and the composite is displayed in 3(a). The salt hydrate characteristic peaks at 3243 cm⁻¹ for O-H stretching, 1644 cm⁻¹ for N=O bending, a mixture of N-O stretching and bending of N=O at 1333 cm⁻¹, and a peak for NO³⁻[17]. It was noticed that the formulated nano PCM composite had found similar functional groups and have no additional or new peaks detected compared to pure PCM. It was demonstrated by the results that the nanoparticles and the salt hydrate PCM mixed physically and there was no chemical reaction between the salt hydrate and the CuO NPs, showing the stability of the formulated composite. Additionally, a peak at 3243 cm⁻¹ for O-H stretching proved the occurrence of unsaturated or aromatic compounds.

3.3. Thermal conductivity analysis

Thermal conductivity of salt hydrate and different weight concentration of CuO are illustrated in Figure 3(b). The pure salt hydrate thermal conductivity was noted at 0.455 W/mK. The thermal conductivity is enhanced 13.34, 30.44, 42.86, 49.5, 62.64 and 51.65% for S50C-0.1, S50C-0.5, S50C-1, S50C-2, S50C-3, and S50C-5 respectively than pure salt hydrate. Thermal conductivity of prepared nanocomposites increased nonlinearly as the increase the concentration to 5 wt% from 0.1 wt%. The conductivity of the nanocomposite increased up to a weight concentration of 3 wt% CuO, and then it decreased down. A similar trend was observed with paraffin wax with CuO [5]. However, the maximum increment of thermal conductivity is 0.74 W/mK at 3 wt% and 62.64% enhancement than pure PCM. There are two mechanisms play to increase or decrease the thermal conductivity of nanocomposites, namely, electron and phonon thermal transport.



Figure 2: (a) FTIR graph of NePCM and (b) Thermal conductivity of NePCM.

To enhance the electron transport, the substance should have linked to a good thermal network originating from the nanomaterials without obstructing the phonon transport. To increase the phonon transport, the phonons themselves need to have the larger free mean path. A greater free mean path may be hampered by phonon impurities scattering, phonon -phonon, and phonon boundary interface. Any one above interactions will have noteworthy effect on heat conductivity. Interestingly, the decrement of conductivity attributes to the agglomeration of particles when using higher weight percentage particles. The accumulation of nanomaterials leads to a non-uniform composite structure and decrease the heat conductivity. Also, the agglomeration of nanomaterial breakdown the thermal network between the nanoparticle and PCM; due to that, the conductivity decreases.

4. Conclusion

In this present research, the thermal, chemical, and thermal conductivity properties of developed nanocomposites were analyzed and had the following conclusions. The prepared salt hydrate/CuO nanocomposite PCM effectively enhances the thermophysical properties. The TGA results shows that the developed nanocomposites thermal decomposition temperature was raised from 454 °C to 467 °C, due to the excellent physical bonding interaction between salt hydrate PCM and CuO nanoparticles, which shows good thermal stability of all prepared composites. The FTIR results showed that incorporating CuO NPs into PCM was physically mixed, and no other peak or functional groups noted, which shows no chemical reaction between salt hydrate and CuO particles. The heat conductivity of pure salt hydrate PCM was measured at 0.455 W/mK. At the same time, the prepared composite's thermal conductivity varies from 0.516 to 0.74 W/mK for CuO concentrations of 0.1 to 5 wt%, respectively. The optimal enhancement in thermal conductivity by 62.64% at 3 wt% CuO, owing to this higher thermal conductivity, the prepared composite is recommended that their realistic application in TES systems. The inorganic PCM embedded CuO offered a higher thermal conductivity and a superior thermal and chemical stability than pure PCM; therefore, the developed composites are considered a potential candidate for TES applications. However, large-scale TES performance on salt hydrate/CuO nanocomposite PCM needs further consideration. It is recommended that to reduce CuO aggregation and split into nano CuO single atoms that the ultrasonication procedure be carried out for a longer duration. Due to uniform dispersion and suspension of the single atom of nano CuO within PCM, resulting in increased homogeneity and improved heat transfer. Further research will involve increasing the heat transfer characteristic of the inorganic PCM by integrating numerous nanocomposite materials. In addition, future research will investigate the reliability analysis by using an accelerated thermal cycler

5. Author Contribution

A. K. Pandey: Conceptualization, Methodology, Funding acquisition, Investigation, Writing–review & editing, Resources, Supervision. **Reji Kumar R:** Methodology, Formal analysis, Investigation, Data curation, Visualization, Writing–original draft. **M Samykano:** Writing–review & editing, Supervision **D Buddhi:** Investigation, Writing–review & editing, Supervision. **VV Tyagi:** Writing–review & editing, Resources.

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Acknowledgements

Authors acknowledges the financial assistance of Sunway University through Sunway University's International Research Network Grant Scheme 2.0 (IRNGS 2.0) 2022 (STR-IRNGS-SET-RCNMET-01-2022) for carrying out this research.