International Journal of Heat and Mass Transfer 74 (2014) 210-214

Contents lists available at ScienceDirect

ELSEVIER

International Journal of Heat and Mass Transfer

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

Specific heat of nanofluids synthesized by dispersing alumina nanoparticles in alkali salt eutectic



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ARTICLE INFO

Article history: Received 31 January 2014 Received in revised form 24 February 2014 Accepted 26 February 2014 Available online 4 April 2014

Keywords: Nanofluid Nanoparticle Specific heat capacity Molten salt Thermal energy storage

ABSTRACT

In this study, we report large enhancement in specific heat capacity of a eutectic salt mixture on dispersing alumina nanoparticles at 1% mass concentration. Eutectic of lithium carbonate and potassium carbonate (62:38 by molar ratio) was dissolved in distilled water with alumina nanoparticles at 1.0% mass concentration and with a nominal diameter of \sim 10 nm. The specific heat capacity measurement was performed using a differential scanning calorimeter (DSC). An alternate model involving in situ phase transformation was proposed which was found to be in good agreement with the variations observed in the experimental data for the different types of nanomaterial samples used in this study. These salt nanofluids can lead to the development of efficient thermal energy storage systems which in turn can enable significant reduction in the cost of solar power.

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1. Introduction

In concentrating solar power (CSP) systems, solar energy is concentrated by a solar collector and is transported by a heat transfer fluid (HTF) into thermal energy storage (TES) units. The stored thermal energy is then utilized in various thermodynamic cycles (e.g. Rankine cycle or Stirling cycle) to generate mechanical or electrical power. TES enables storage of excess solar energy in the daytime for utilization in the nighttime or for balancing load levels in electrical grids during times of peak demand. In contemporary CSP systems synthetic organic oils (which are stable up to 400 °C) are widely used for HTF and TES [1]. Alkali salt eutectics are considered as attractive options for TES and HTF materials for solar thermal units operating at temperatures exceeding 500 °C. Alkali salt eutectics provide several benefits: (1) These eutectics have tunable melting points and the salts are stable up to \sim 600–700 °C (pushing the storage temperature to higher values - hence enhancing the overall thermodynamic efficiency of CSP systems). (2) These eutectics can enable reduction in the cost of solar power that is enabled by the higher thermodynamic efficiencies achieved at higher operating temperatures and the lower material costs that are achieved using these eutectic materials.

Typical alkali salts that are suitable for solar thermal power applications include alkali-nitrate, alkali-carbonate, alkali-chloride and their eutectic mixtures that enable a lower melting point. However, the low values of the specific heat capacity of these eutectics [2] (less than 2 kJ/kg °C) – can cause the size of the storage devices to be larger than those of the oil based systems. This can increase the total system costs, thus resulting in the loss of efficacy expected from the deployment of the higher temperature thermal energy storage systems. An inexpensive strategy for obviating this issue is to enhance the thermo-physical property values of these alkali salt eutectics by doping them with minute concentrations of nanoparticles – resulting in a stable homogeneous colloidal suspension of nanoparticles.

Stable colloidal suspensions of nanometer-sized particles in various liquid solvents are termed as "*nanofluids*" [3]. Enhanced thermal conductivity values of nanofluids have been reported in various studies [3–16]. Xie et al. [3] observed 29% enhanced thermal conductivity of alumina/ethylene glycol nanofluid for 5% concentration by volume. Hong et al. [4] observed 18% enhanced thermal conductivity of Fe nanofluid at only 0.55% concentration by volume. Li and Peterson [5] observed 52% enhanced thermal conductivity of CuO/water nanofluid for 6% concentration by volume. Several attempts were made at exploring the underlying

Abbreviations: CSP, concentrating solar power; HTF, heat transfer fluid; TES, thermal energy storage; TEM, transmission electron microscope; SEM, scanning electron microscope; DSC, differential scanning calorimeter.

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mechanisms responsible for the enhancements [17–19]. Recent studies proposed that nanofluids with well-dispersed spherical particles agreed well with the traditional effective medium theory [20]. Nanofluids with aggregated interacting nanoparticles can be also bounded by the modified effective medium theory of linear aggregation models (i.e., chain-like structures made by interconnected nanoparticles) [21,22].

Similarly, specific heat capacity values of nanofluids can be modified significantly by doping with minute concentration of the nanoparticles. However, the measurement of the specific heat capacity of nanofluids has not received as much attention in the literature (as compared to the numerous studies that continue to be reported for thermal conductivity measurements). Several studies reported that the specific heat of organic/aqueous nanofluids decreased [23–25]. Namburu et al. [23] reported 12% decrease of specific heat of water after loading SiO₂ nanoparticles at 10% concentration by volume. Zhou and Ni [24] reported water-based Al₂O₃ nanofluid showed 40% decrease of specific heat at 22% nanoparticle loading by volume. Vajjha and Das [25] observed water/ ethylene glycol mixture showed 20% decrease of the specific heat after loading ZnO nanoparticles at 7% volume concentration. On the other hand, recent studies showed that the specific heat of liquid salts can be enhanced by doping nanoparticles at minute concentrations [26-32]. Around 25% enhanced specific heat was observed from SiO₂ nanoparticle dispersions (1%) in a eutectic of $Li_2CO_3-K_2CO_3$ [26]. Specific heat of eutectic of chloride salts (BaCl₂-CaCl₂-NaCl-LiCl) showed 20% enhancement after loading SiO₂ nanoparticles at 1% [27]. Proposed mechanism for the enhanced specific heat capacity of salt-based nanofluids in the literature [30,31] is that salt eutectic is likely to form a chain-like nanostructure (similar to the percolating network formed by aggregated nanoparticles in conventional nanofluids, but different because the base fluid itself (liquid salts) forms the nanostructure and thus the amount of nanostructure formation is much larger than that of conventional nanofluids) and the nanostructure contributes to the enhanced specific heat capacity. This means that liguid salts are primarily responsible for the enhanced specific heat capacity of salt-based nanofluids and nanoparticles are just to induce the formation of nanostructure. If this is true, replacing nanoparticles for the same liquid salts should not affect the enhanced specific heat capacity of salt-based nanofluids.

In this study a different set of nanofluids were synthesized by dispersing alumina nanoparticles in a eutectic of carbonate salts, where the eutectic consisted of a mixture of lithium carbonate and potassium carbonate. This carbonate salt eutectic was chosen to compare with the literature [26,30,31], to confirm the proposed model in these studies, and, in addition, due to its thermal stability at high temperatures (over 600 °C) [33]. In these experimental measurements – the mass concentration of the alumina nanoparticles in the alkali salt eutectics was fixed at 1.0% (to enable a consistent comparison with earlier studies) [26–31]. Experimental measurement of the specific heat capacity values as a function of temperature were performed using a differential scanning calorimeter (Q20, TA Instruments, Inc). Thermal equilibrium model (also known as the "mixture rule") was used to predict the enhancement of the specific heat of the nanofluids:

$$c_{p,t} = \frac{\rho_{np}\phi_{np}c_{p,np} + \rho_s\phi_s c_{p,s}}{\rho_{np}\phi_{np} + \rho_s\phi_s} \tag{1}$$

where c_p is specific heat, ϕ is the volume, and ρ is the mass of the constituents of the mixture. Subscripts *t*, *np* and *s* denote the appropriate thermo-physical property values for the resultant nanofluid, nanoparticle, and liquid salt (the base of nanofluid), respectively. The predictions from the Model (1) were compared with the experimental measurements.

2. Experiments

Lithium carbonate (Li_2CO_3) and potassium carbonate (K_2CO_3) were procured from Sigma Aldrich Co. and alumina nanoparticles were procured from Meliorum Tech. The average size of the alumina nanoparticles was confirmed by the manufacturer to be \sim 10 nm. The protocol for synthesis of the nanofluids is described next. (Pure eutectic of lithium carbonate and potassium carbonate was synthesized in the same protocol) Initially, 2.0 mg of alumina nanoparticles and 198.0 mg of eutectic were precisely measured by a microbalance (CPA26P, Satorius AG) and the amorphous powders were dispensed in a glass bottle (of 25 ml volume). The eutectic consisted of lithium carbonate and potassium carbonate in 62:38 M ratios. The bottle was then filled with 20 ml of distilled water and ultra-sonicated for 200 min (Branson 3510, Branson Ultrasonics Co.). The aqueous solution with dispersed nanoparticles was poured into a glass petri-dish (10 cm diameter). The petri-dish was placed on a hot plate (C-MAG HP7, IKA) at 100 °C and was housed in a glove box with filtered air circulation to enable rapid evaporation of the water from the salt solution. This enabled uniform dispersion of the nanoparticles in the amorphous eutectic powder after the water was evaporated completely. Complete evaporation of water was accomplished in approximately 30 min and special care was taken to prevent bubble nucleation or pool boiling of the solution (pool boiling was found to cause degradation of specific heat due to agglomeration of the nanoparticles). For the specific heat capacity measurements, two samples were independently synthesized and individually tested four times to ensure the repeatability of the measurements.

It is very important to follow a consistent testing, measurement and characterization protocol for the nanofluids. Standard protocol for DSC testing (ASTM-E1269) was therefore employed to measure the specific heat of the samples. Standard Tzero hermetic pan and lid (TA instruments, Inc.) were used to prevent weight loss of the samples during repeated thermo-cycling. Before dispensing into the hermetic pans, the salt samples were heated at 300 °C for 2 h to remove any chemically bonded (hydrated) water molecules. An automated thermo-cycling program was implemented in the DSC instrument (Model: Q20, TA Instruments) for performing the specific heat capacity measurements of the samples and is described next. At the beginning of each thermo-cycle, the temperature of the DSC cell was held at 150 °C for 8 min to ensure a stable temperature base-line. The temperature was then ramped to 560 °C at a fixed rate of 20 °C/min. The temperature was then held constant at 560 °C for 8 min to ensure a stable temperature base-line. The sample was then cooled down to 150 °C using successively, flow of nitrogen. For each sample, this thermo-cycling program was repeated many times to verify repeatability of the measurements and to ensure the temperature stability of the nanoparticles in the molten salt eutectic.

3. Results and discussion

The average specific heat capacity values for each thermo-cycle obtained from the DSC measurements for the samples are shown in the Table 1 (these results are also plotted in Figs. 1 and 2 where the specific heat capacity is plotted as a function of temperature by averaging the results from each thermo-cycle). To verify the accuracy of the measurement protocol, the specific heat capacity of the pure eutectic was measured and compared with the literature data. The measurement uncertainty in these experiments is estimated to be 5.3%. The average value of the specific heat capacity of the eutectic was measured to be 1.65 kJ/kg °C with a standard deviation of 0.09 kJ/kg °C (5.3%), which is in good agreement with the value reported in the literature [2] (1.60 kJ/kg °C).

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