Enhanced thermal properties of SiO₂ nanocomposite for solar thermal energy storage applications

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ABSTRACT

In this study the thermo-physical properties of a solid nano-composite material was measured and compared with the undoped solid material. SiO₂ nanoparticles were dispersed in eutectic of lithium carbonate and potassium carbonate (62:38 molar ratio) to obtain a high temperature nanocomposite. Specific heat measurement was performed using a differential scanning calorimeter (DSC). A Laser Flash Apparatus (LFA) was used to measure thermal diffusivity. The thermal conductivity was computed based on the specific heat and the thermal diffusivity. On mixing (doping) with the nanoparticles at 1% concentration by weight the specific heat was enhanced by 5~15% and the thermal diffusivity was enhanced by 25~28%, respectively. The corresponding effective thermal conductivity of the nanocomposite was calculated to be enhanced by 35~45% compared with that of the undoped (pure) material. On performing Scanning electron microscopy (SEM) of the nanocomposite a percolation network (interconnected substructure) of a dense material was observed. Such substructures were not observed in the undoped (pure) material. It is assumed that this percolation network of higher density phase that is observed to form around the nanoparticles is responsible for the enhancement of the thermal properties of the nanocomposite.

KEYWORDS

Nanocomposite, thermal conductivity, specific heat, silica nanoparticle, molten salt, thermal energy storage

ABBREVIATIONS

CSP Concentrating Solar Power

TES Thermal Energy Storage

TEM Transmission Electron Microscope

SEM Scanning Electron Microscope

DSC Differential Scanning Calorimeter

LFA Laser Flash Apparatus

1. INTRODUCTION

The genesis of this investigation lies in the quest for designing novel materials that are compatible with the emerging needs for Concentrating Solar Power (CSP) systems. Solar energy can be harvested directly into electricity using photovoltaic. An indirect approach relies on concentrating the thermal energy in the solar spectrum to achieve a high temperature as well as storing the thermal energy obtained at a high temperature for subsequent work extraction using thermodynamic cycles (e.g., Rankine cycle or Sterling cycle) for the conversion to electricity. Contemporary commercial Thermal Energy Storage (TES) devices typically use mineral oil and are therefore limited to a maximum operating temperature of 400 °C. This enables continuous power generation during off-peak hours (such as night time) as well in presence of cloud cover. To improve the thermodynamic efficiencies of solar thermal power plants it is necessary to increase the operating temperature to 500-600 °C or higher. However, only few thermal storage materials are compatible for operating at such high temperatures. To reduce operational costs the materials required for the next generation of TES devices need to have high heat capacity, appropriate thermal conductivity, and stability at high temperatures (i.e., over 400 °C). Molten salts, such as eutectics of alkali nitrate, alkali carbonate, or alkali chloride, has melting points ranging from 200 °C to 500 °C and are stable up to 700 °C [1, 2]. Improving TES performance using these molten salts haven been reported by many research groups [3, 4]. However, thermal conductivity and specific heat of these salt mixtures are lower than desired in TES applications, especially when the salt mixtures are in the solid phase and thus charging / discharging energy mainly relies on heat conduction (less than ~ 1 W/m°C and ~ 2 J/g°C, respectively; [5, 6]).

Materials in solid phase that are mixed with nanoparticles are termed as “Nanocomposites”. Addition of nanoparticles (such as Carbon Nanotubes, or “CNT”) at minute concentrations was reported to significantly enhance the thermal conductivity of nanocomposites (over that of the undoped pure polymer “matrix”). Hence this investigation was motivated by prior reports in the literature and the aim of this study was to improve the thermo-physical property values of the salt eutectics by addition of minute quantities of nanoparticles.

Prior investigations on nanocomposite materials have primarily been focused on enhancing the thermo-physical, electrical and optical properties of polymers. These studies were also primarily focused on the addition of Carbon Nanotubes (CNT) to the polymer matrices. Wang et al. reported that the thermal conductivity of polymers was enhanced by 30% when mixed with CNT (at 1% concentration by weight) [7]. Thermal conductivity values were enhanced by 100% when an epoxy was mixed with CNT at 1.5% concentration by weight [8]. Haggemueller et al. [9] mixed high density polyethylene (HDPE) with single walled carbon nanotube (SWCNT) at 20% by volume and observed 700% enhancement in the thermal conductivity (over that of pure HDPE). The anomalous enhancement of thermal conductivity of nanocomposites can be explained by the formation of percolation network consisting of an embedded network of interconnected nanotubes which provide a preferential path for heat transfer [9]. However, it is not known whether nanocomposites realized by mixing spherical nanoparticles would show similar enhancement of thermal conductivity – since the formation percolation networks demands an embedded structure of high aspect ratio nanoparticles.

Similarly, the specific heat of the nanocomposites is also expected to be enhanced when nanoparticles are added into a mixture of molten salts. In point of fact, nanoparticles have been shown to possess higher specific heat values than the bulk property values of the same material due to their extremely high surface area to volume ratio [10-12]. When nanoparticles are added into a mixture of molten salts, they induce the mixture to be separated locally near the nanoparticles due to the difference in electrostatic interaction between each salt compound and nearby surface atoms of nanoparticles. Separated salt compound starts to crystallize on the nearby nanoparticles, to grow away from the nanoparticles and eventually to form interconnected nanostructure (percolation network) [13]. These nanostructures have very high surface to volume ratio like nanoparticles and have higher specific heat than the bulk salt mixture. Several researches have been reported for the enhanced specific heat of molten salt / nanoparticle mixture [14-23].

In this study, nanocomposites were synthesized by mixing eutectic of Li_2CO_3 and K_2CO_3 (62:38 molar ratio) with SiO_2 nanoparticles (at 1% concentration by weight). This eutectic was chosen for applicability to future generations of TES devices for CSP applications. The specific heat and the thermal diffusivity of the nanocomposite were measured in the solid phase using a differential scanning calorimeter (DSC) and a laser flash apparatus (LFA), respectively. Scanning Electron Microscopy (SEM) was used to characterize the microstructures in the nanocomposite and the pure alkali metal salt eutectic, for investigating the potential mechanisms responsible for the anomalous enhancement observed during the measurements of the thermo-physical property values.

2. EXPERIMENTS

The nanocomposites were synthesized by a simple two step liquid solution method. SiO_2 nanoparticle (~10 nm (manufacturer's specification; ~23 nm in diameter was observed experimentally)) was procured

from Meliorum Technologies, Inc. The salts - lithium carbonate and potassium carbonate were procured from Sigma Aldrich, Co. The procedure of synthesis of the nanocomposite is as follows: Initially 200 mg mixture of the nanoparticles and the eutectic was prepared in the dry state. The weight concentration of the SiO₂ nanoparticles was 1 % in the dry mix. Li₂CO₃ and K₂CO₃ were mixed in 62:38 molar ratio. The solid mixture was then dissolved in 20 ml of distilled water in a clean glass beaker and sonicated by ultrasonicator (Branson 3510, Branson Ultrasonics Corporation) for ~100 minutes to ensure uniform dispersion of the nanoparticles. The glass beaker containing the solution was then placed on a hot plate (C-MAG HP7, IKA) set at 200 °C to evaporate the water from the solution. The typical time required to evaporate the water solution completely was ~ 3 hours. The created nanocomposites were thermally cycled between 150 °C and 560 °C several times to ensure the formation of nanostructure as reported in the literature [18, 19]. The specific heat of the solid phase of the pure eutectic and of the nanocomposite was measured using a differential scanning calorimeter (Q20, TA instruments, Inc) using a standard DSC testing method (ASTM E1269). Standard Tzero hermetic sample holders are used to avoid potential sample loss and the measurement was repeated 4-9 times in each experiment (i.e., the temperature of the sample was cycled between 4~9 times to ensure repeatability of the measurement). The moisture effect of chemical reaction was confirmed by analyzing the DSC heat flux signal. The experimentally obtained specific heat was compared with theoretical thermodynamic mixing model (also known as the thermal equilibrium model [24]):

$$c_{p,t} = \frac{\rho_p V_p c_{p,p} + \rho_f V_f c_{p,f}}{\rho_p V_p + \rho_f V_f} \quad (1)$$

where $c_{p,t}$, $c_{p,p}$, and $c_{p,f}$ are the specific heat of the nanocomposite, the nanoparticle, and the pure eutectic, respectively; while ρ_p is the density of the nanoparticle, ρ_f is the density of the pure eutectic, V_p is the volume of the nanoparticle, and V_f is the volume of the pure eutectic.

The nanocomposite samples and pure eutectic samples were shipped to Dynalene Inc. for measurement of the thermal diffusivity of the samples using a Laser Flash Analysis (LFA 447 Nanoflash, Netzsch Instruments N.A. LLC.). LFA is a device to measure thermal diffusivity using a laser to heat one side of sample and measuring the temperature rise on the other side with time. The instrument used in this study was limited to a maximum temperature of 300 °C and thus three fixed temperatures are chosen for the study (150 °C, 225 °C, and 300 °C). The thermal conductivity was then computed based on the density and the specific heat of the samples. The experimentally measured thermal conductivity values were then compared with conventional theoretical models, such as the Hamilton-Crosser model (Equation (2); [25]) and the Maxwell-Garnett model (Equation (3); [26]). The Hamilton-Crosser model is formulated as:

$$k_t = k_f \left[\frac{k_p + (n-1)k_f - (n-1)V_p(k_f - k_p)}{k_p + (n-1)k_f + V_p(k_f - k_p)} \right] \quad (2)$$

where k_t , k_f , and k_p are the thermal conductivity of nanocomposite, the pure eutectic, and the nanoparticle, respectively; while n is empirical shape factor. For spherical nanoparticles, $n = 3$ (which is a valid approximation for silica nanoparticles used in this studies). The Maxwell-Garnett model is formulated as:

$$k_t = k_f \left[\frac{(1 + 4 \frac{R_b k_f}{d}) + 2V_p (1 - 2 \frac{R_b k_f}{d})}{(1 + 4 \frac{R_b k_f}{d}) - V_p (1 - 2 \frac{R_b k_f}{d})} \right] \quad (3)$$

where R_b is interfacial thermal resistance (Kapitza resistance [27]) between the nanoparticle and the surrounding eutectic molecules; while d is the diameter of the nanoparticle. The interfacial thermal resistance between SiO_2 nanoparticles and eutectic of Li_2CO_3 and K_2CO_3 can be computed numerically (e.g., by molecular dynamics simulation) and was reported to be $6.8 \times 10^{-9} \text{ m}^2\text{K/W}$ [28]). The measurement uncertainty for the specific heat data is estimated to be less than 3% (relative). The measurement uncertainty for the thermal diffusivity values is estimated to be less than 5% (relative). Hence, the uncertainty in the thermal conductivity data is estimated to be less than 6%.

3. RESULTS AND DISCUSSION

The specific heat of the pure eutectic and the SiO_2 nanocomposite (at 1% concentration by weight), are shown in Figure 1 and the data from the measurements are listed in Table 1. The specific heat of the SiO_2 nanocomposite was enhanced by 5~15% over the temperature range of the measurements. To verify the accuracy of the specific heat measurements for the nanocomposites, the specific heat of the pure eutectic was measured in this study and subsequently compared with literature data (Araki et al., 1988). As shown in Table 1 the difference in specific heat values between literature data and measurements performed in this study are within 1~6%. A major discrepancy is observed on comparing the measured values for the specific heat for the nanocomposite with the values calculated using the thermodynamic mixture rules proposed in the literature (e.g., Equation 1). Figure 1 shows that the predictions from Equation 1 do not deviate significantly from the pure eutectic values, whereas the experimental data for pure eutectic is enhanced by a significant margin (higher than the measurement uncertainty for specific heat). The discrepancy may result from the formation of interconnected nanostructure by salt compounds when doped with nanoparticles. According to the recent study on the specific heat of molten salt nanofluids [13, 19], nanoparticles tend to separate salt mixture due to their difference in electrostatic interaction between the nanoparticles and each salt compound of the mixture. Separated salt compound then crystallizes on the surface of nanoparticles and forms a nanometer-sized

structure. The structure grows further away from the nanoparticles and eventually forms interconnected nanostructure, whose specific heat is higher than the bulk salt mixture [13, 19].

The thermal diffusivity of the pure eutectic and the SiO₂ nanocomposite (at 1 % concentration by weight) are shown in Table 1 and Figure 2. The thermal diffusivity of the pure eutectic linearly decreased with temperature, and the same trend can be observed for the SiO₂ nanocomposite as well. The enhancement of the thermal diffusivity value of the nanocomposite over that of the pure eutectic is observed to be consistently in the range of 25~28 % and the enhancement is observed to be almost independent of temperature (though the actual values for the thermal diffusivity for both materials decreases with increase in temperature).

Table 1. Density, specific heat, thermal diffusivity, and thermal conductivity of the pure eutectic and the SiO₂ nanocomposite (at 1% concentration by weight).

		150 °C	225 °C	300 °C
Nanoparticle	ρ_p [g/cm ³]	2.196 [29]	2.196 [29]	2.196 [29]
	$c_{p,p}$ [J/gK]	0.915 [29]	0.994 [29]	1.054 [29]
Pure eutectic	ρ_f [g/cm ³]	2.202	2.202	2.202
	$c_{p,f}$ [J/gK] (literature) (error, %)	0.987 (1.053 [6]) (6%)	1.176 (1.140[6]) (3%)	1.244 (1.227 [6]) (1%)
	α_f [mm ² /s]	0.209	0.192	0.180
	k_f [W/mK]	0.455	0.498	0.493
Nanocomposite	ρ_t [g/cm ³]	2.202	2.202	2.202
	$c_{p,t}$ [J/gK] (measured) (Enhancement %)	1.131 (15%)	1.263 (7%)	1.365 (10%)
	α_t [mm ² /s] (measured) (Enhancement %)	0.267 (28%)	0.243 (27%)	0.225 (25%)
	k_t [W/mK] (Enhancement %)	0.668 (47%)	0.678 (36%)	0.677 (37%)

Figure 1. Comparison of the specific heat measurements of the pure eutectic of lithium carbonate and potassium carbonate (62:38 molar ratio) with that of the nanocomposite (synthesized by addition of ~10-30 nm diameter SiO₂ nanoparticles at 1% by weight). Theoretical estimates using the thermal equilibrium model are plotted for comparison.

Figure 2. Comparison of the thermal diffusivity values of the pure eutectic of lithium carbonate and potassium carbonate (62:38 molar ratio) with that of the nanocomposite (synthesized by addition of ~10-30 nm diameter SiO₂ nanoparticles at 1% by weight).

A scanning electron microscope (FE-SEM, JEOL-7500) was used to observe the micro-structural features that are embedded in the nanocomposite. SEM images of the pure eutectic (without SiO₂ nanoparticle) were compared with that of SiO₂ nanocomposites, and are shown in Figure 3 and Figure 4, respectively. By comparing the two SEM images, it is observed that a percolation network (interconnected network) of a higher density phase (which appears as lighter color in the SEM image) is formed in the SiO₂ nanocomposite (Figure 4). The material composition of the different colored regions was confirmed to be that of the eutectic - by using Energy Dispersive X-Ray Spectroscopy (EDS) in the SEM instrument (however, the measurement uncertainty in the EDS is typically ~ 3% and therefore would not be adequate for confirming the presence of silica nanoparticles in the sample, since the silica nanoparticles were mixed at an average mass concentration of 1%). Hence, the higher density phase in the solid eutectic is expected to have a higher thermal conductivity and therefore provide preferential pathways for heat conduction within the nanocomposite. Hence this percolation network is the plausible mechanism for the enhancement of the thermal conductivity and diffusivity of the nanocomposite.

Figure 3. SEM image of the pure eutectic of lithium carbonate and potassium carbonate (62:38 molar ratio).

Figure 4. Representative SEM image of the SiO₂ nanocomposite. These percolation networks of lighter color eutectic material (indicative of higher density phase) may be responsible for the enhancement of the thermal conductivity of the nanocomposite.

TEM images were obtained to verify the size and shape of the SiO₂ nanoparticles (Figure 5). The TEM image shows that the nanoparticle size in the nanocomposite mixture is ~ 23 nm. The nanoparticle size was found to vary in the range of ~10-30 nm in this study. No significant change in the particle size distribution of the nanoparticles was observed on performing characterization studies using TEM, both before and after the thermo-cycling of the nanocomposites in the DSC instrument.

Figure 5. TEM image of the SiO₂ nanocomposite. The size of the SiO₂ nanoparticle is measured to be ~23 nm in this image.

Based on the measured values for the specific heat and the thermal diffusivity of the samples, the values for the effective thermal conductivity of the pure eutectic and the SiO₂ nanocomposite were calculated. The density of the nanocomposite was obtained by assuming a simple mixing rule based on mass conservation. The thermal conductivity data are plotted as a function of temperature in Figure 6. No temperature dependent trend was observed in the thermal conductivity values for both materials. The thermal conductivity values were found to fluctuate marginally with increase in temperature for both materials.

The enhancement in the thermal conductivity for the nanocomposite (over that of the pure eutectic) was found to be in the range of 37~47 %, at different temperatures. Significant discrepancies were observed on comparison of the experimental data with the mixing rules proposed in the literature (Equations 2 and 3). Figure 6 shows the comparison of the experimental data with the predictions from the theoretical models for the thermal conductivity of mixtures using the two different models (Equations 2 and 3). The theoretical predictions are almost identical with the data for the pure eutectic while the experimental data for the nanocomposites are significantly higher (and are significantly larger than the measurement uncertainties). The discrepancy between the measurement and the theoretical predictions may result from the formation of interconnected nanostructure by salt compounds when doped with nanoparticles.

Table 2. Comparison of experimentally measured data with theoretical estimates for the thermal conductivity of SiO₂ nanocomposite at 1% concentration by weight.

Thermal conductivity, k [W/mK]	150 °C	225 °C	300 °C
Pure Eutectic	0.455	0.498	0.493
Standard deviation	0.026923	0.016275	0.015136
Maxwell-Garnett model (Enhancement %)	0.455 (0%)	0.497 (0%)	0.492 (0%)
Hamilton-Crosser model (Enhancement %)	0.458 (0%)	0.501 (0%)	0.496 (0%)
SiO ₂ nanocomposite (Enhancement %)	0.668 (47%)	0.678 (36%)	0.677 (37%)
Standard deviation	0.179251	0.039349	0.023911

Figure 6. Comparison of the thermal conductivity values of the pure eutectic of lithium carbonate and potassium carbonate (62:38 molar ratio) with that of the nanocomposite (synthesized by addition of ~10-30 nm diameter SiO_2 nanoparticles at 1% by weight). Theoretical estimates using the Hamilton-Crosser model (Equation 2 [25]) and the Maxwell-Garnett model (Equation 3 [26]) and are plotted for comparison.

4. CONCLUSION

Eutectic of lithium carbonate and potassium carbonate (62:38 molar ratio) was explored as a material for TES in CSP applications. A nanocomposite material was synthesized by mixing the eutectic with ~10-30 nm diameter spherical silica nano-particles at 1 % concentration by weight. The synthesis involved a two step procedure involving dry mixing followed by dissolution in an aqueous solution and evaporation of the water to obtain a uniform mixture. Specific heat and thermal diffusivity of the nanocomposite were measured by a differential scanning calorimeter (DSC) and a laser flash apparatus (LFA), respectively.

The specific heat of the nanocomposite was enhanced by 5~15% compared with the pure eutectic. The thermal diffusivity was enhanced by 25~28%. The corresponding thermal conductivity data showed an enhancement of 37~47%. The enhancement of the specific heat can be explained to be due to the

formation of interconnected nanostructures (percolation network) from salt compounds induced by the addition of nanoparticles.

The enhancement of the thermal conductivity of the nanocomposite in the solid phase - cannot be explained satisfactorily by the existing mixing models in the literature. In the SEM images interconnected substructures (percolation networks) composed of a lighter color phase of higher density eutectic material was observed to be embedded in the nanocomposite. This percolation network structure is absent in the pure eutectic. This percolation network seems to be one of the plausible factors responsible for the enhancement of the thermal conductivity of the nanocomposite.

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