Graphene-Based Phase Change Composite Nano-Materials for Thermal Storage Applications

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ABSTRACT

This article reports results referring to graphene based nano-platelets functionalization to improve the thermal properties of organic based Phase Changing Materials (PCM). Over the last years the use of PCMs have being appointed as a suitable way for developing thermal energy storage solutions based on the materials frozen/melting latent heat. Nevertheless the low thermal conductivity of these materials, in particular of the organic based PCMs, is a drawback and represents a limitation to the application of these solutions on real-life energy systems. The aim of the present work is to improve the thermal conductivity of the PCMs while preserving the specific and latent heat value. Several types of graphene nano-particles (GNP) in various concentrations were employed to enhance the thermal conductivity of wax-based PCMs. A modified enthalpy-based water bath method was used to evaluate the thermal properties of the pure PCMs and the PCMs enhanced by the graphene nano-platelets. Furthermore detailed CFD simulations of the heat transfer process on a simple coil heat exchanger will be performed to assess the influence of the enhancement of the thermal properties of the PCM over the solidification/melting process for several operating conditions. Preliminary results show that both dimension and shape of the GNP are important factors for the thermal properties, which were found to be significantly improved. It was found that the addition of only 1% weight of GNP with high aspect ratio is sufficient for doubling the thermal conductivity of several common PCMs. The present results provide an efficient and easy way for improving the thermal properties of PCMs, suitable for thermal storage applications.

INTRODUCTION

Heat stored in the solid-liquid phase transition in phase-change materials is one of the most preferred forms for energy storage, due to the large latent heat and negligible temperature and volumetric changes associated with the phase transition. Paraffin wax in particular has attracted attention because of its low cost, stability and chemical inertness (Xia et al., 2010). However, paraffin wax, in common with many phase change materials, has low thermal conductivity, which inhibits quick heat transfer and thus limits its use as an energy storage material.

In order to overcome the thermal conductivity problem, common in many phase change materials, a number of different nanoparticle suspensions have been explored such as carbon-based nanostructures (nanofibers, nanoplatelets, graphite nanoparticles, graphene flakes and carbon nanotubes), metallic (Ag, Al, Cu, Fe) and metal oxides (Al₂O₃, CuO, Fe₃O₄, TiO₂) nanoparticles. It was found that in general, carbon based nanostructures and carbon nanotubes exhibit by far greater enhancement of thermal conductivity in comparison to metallic/metal oxide nanoparticles (for comprehensive, recent reviews see Khodadadi et al. 2013, Kibria et al. 2015).

In the last decade or so extensive research has been focused on the study of the thermal properties of nanofluids, defined as fluids with nanoparticle suspensions. Nanofluids with suspensions of metallic or nonmetallic solid nanoparticles with sizes between 1 and 100nm from materials with higher conductivity than the base fluid exhibited higher thermal conductivity in
comparison with the equivalent macro-particle suspensions (Bashirnezhad et al., 2015, Keblinski et al., 2008, Özerinc et al., 2010, Mushed et al. 2005). These enhancement effects can be large in relatively low particle volume or mass fractions making nanofluids attractive from a cost effect point of view as well. Typically, the thermal conductivity of the composite material was found to depend on the thermal conductivities of the constituent materials, the concentration of the diluted phase, as well as size and shape of the dispersed particles. In particular, a non-linear dependence on concentration was observed in the limit of low concentration. Thermal conduction was found to improve with decreasing particle size, although non-linearly and non-consistently (Yu et al., 2008), presumably because the smaller particles would have a larger surface to volume area, thus enabling better heat conduction. A number of theoretical studies have suggested that the reduced particle size induces mechanisms in the suspension that could account for the increased thermal conductivity such as, the Brownian motion of the nanoparticles, liquid layering at the liquid/particle interface and the effect of nanoparticle clustering (Keblinski et al. 2002). In particular, the effect of clustering was found to be important. Effective medium theories that take into account the clustering effects give better agreement with experimental values of thermal conductivity (Prasher et al. 2006, Gharagozloo et al. 2008, Bonnet et al. 2007). The effective volume of a cluster is considered much larger than the volume of the particles due to the lower packing fraction (ratio of the volume of the solid particles in the cluster to the total volume of the cluster) of the cluster (Mushed et al. 2008). Heat transfer is assumed to be faster within such clusters and thus the volume fraction of the highly conductive phase, now taken to be the cluster rather than the solid particles, is also larger. Experiments have shown that indeed nanoparticles may not stay dispersed for long, forming as well as clusters, linear chain configurations, and fractal shaped aggregates (Kim et al. 2007, Mushed et al. 2005, Zhu et al. 2006). In addition, longer aspect ratio particles such as nanofibers, carbon nanotubes, or nanowires were found to enhance thermal conductivity even further (Timofeeva et al., 2009, Deng et al. 2007, Cherkasova et al. 2008, Xie et al. 2008). A number of studies show that when spherical and cylindrical particle shapes are compared, the cylinders show an increased thermal conductivity over the spheres for the same volume fraction and particle volume. It was hypothesized that the longer aspect ratio particles can form a mesh that spans throughout the fluid and thus conducts heat throughout the fluid (Yu et al. 2008, Timofeeva et al. 2009). Evidence of percolation in carbon nanostructures/polymer composites have been reported by Bonnet et al. 2007, Kim et al. 2013 and Shehzad et al. 2014.

With respect to change phase materials, quite a few studies have been reported in the literature involving a number of different higher conductive additives and those pertaining to paraffin wax are summarized below. Graphite nano-fibers with diameters of 2-1000 nm and lengths up to 100 μm with a thermal conductivity of around 880 W/mK, mixed with paraffin (melting temperature of 56 ℃ and thermal conductivity of 0.25 W/mK) were reported to increase the thermal conductivity up to 180% at an 8% wt fraction. A simultaneous decrease in the latent heat of fusion from 271.6 J/g from pure paraffin to 242.7 J/g for the 8% wt NP enhanced paraffin was also observed (Warzoha et al., 2012). Carbon nanostructures have even higher thermal conductivities in certain directions as high as 3000 W/mK and are therefore very attractive as additives for phase change storage materials. Zeng et al. (2008) have used multi-walled nanotubes with an outer diameter of 10-30nm and length of 5-15μm with 1-tetradecanol (TD) as the PCM (with melting temperature of 35 ℃), with mass fractions up to 5%. They observed a linear enhancement of the thermal conductivity of up to 30% with negligible change in the heat of fusion. Li (2013) studied the thermal conductivity of nano-graphite/paraffin composites with graphite particles with diameters of 35nm. They found greatly increased thermal conductivities of 0.3650 W/mK for mass fraction of 1% up to a value of 0.9362 W/mK for 10% mass fraction. At the same time the latent heat was reduced from 209.33 J/g for pure paraffin to 202.6 J/g and to 181.81 J/g for 1% and 10% mass fraction of graphite nanoparticles respectively.

Research in the Drzal group has shown that exfoliated graphite nanoplatelets with diameter of 15μm, thickness of less than 10nm and BET surface area of around 30m²/g, when mixed with paraffin (n-docosane, with melting temperature 53-57℃) with mass fraction up to 7% xGnP-15, increase the thermal conductivity linearly to a value of 0.8 W/mK. In addition, they reported negligible changes in the latent heat fusion with the addition of up to 7% mass fraction of graphite nanoplatelets. The xGnPs were found to maintain the layered structure similar to natural graphite flake (Kin and Drzal 2009).

In this work we have chosen to work with exfoliated expanded graphite nanoplatelets of differing sizes, aspect ratios and surface area similar in concept to those used by the Darzl group. The nanoplatelets have an in-plane and out-of-plane
thermal conductivity of 3000 and 6W/mK respectively. They were chosen because of their superior thermal conductivity, comparable to that of carbon nanotubes, at a much less cost and thus feasible for applications. They also come with high dimensional aspect ratios and that can be tailored to different sizes. We report here on the measured thermal conductivities of the composites materials, as well as the specific heats and heats of fusion as a function of mass fraction up to 6%. Composites of paraffin and graphite nanoplatelets with diameters in the 1-15 μm range and thicknesses in the 6-15 nm range were prepared. For 6% weight fraction with the 15μm diameter nanoplatelets the thermal conductivity of the composite is found to be 0.78 W/mK representing ~250% increase on the pure paraffin thermal conductivity measured at 0.22 W/mK. We have found that the thermal conductivity is enhanced at even lower weight fraction though, with the addition of 1% weight fraction of nanoplatelets of 15μm diameter (k=0.44 W/mK, ~100% increase). In addition, the heat of fusion has been found to reduce up to 10% when increasing weight fraction, but to remain very close to the pure value, within 2%, for low weight fractions. Some preliminary efforts were undertaken to compare our findings in thermal conductivity with theoretical predictions within the effective medium approach.

**EXPERIMENTAL**

**Materials**

Paraffin with melting temperature of 53 °C was provided by PCM products and the graphite nanoplatelets xGnP were provided by XG Sciences Inc. The graphite nanoplatelets are ultrathin particles of graphite that can also be thought of as short stacks of graphene sheets made with thickness ranging from a few nanometers and diameters ranging a few the microns. In this report we present data for three different types of nanoplatelets: Grade M with thickness of 6-8nm and diameter of 5μm (M5) and diameter of 15μm (M15) and typical surface area of 120 to 150 m²/g, grade H with thickness of 15nm and diameter of 5μm (H5) and typical surface area of 50 to 80 m²/g. Finally we also report on nanoplatelets (grade C) that typically consist of aggregates of sub-micron platelets having particle diameter of less than two microns and typical thickness of a few nanometers (C750) with surface area of 750 m²/g.

**NP enhanced PCM preparation**

Paraffin wax was melted at 70°C and the nanoplatelets were added to the liquid paraffin under stirring at 0.5, 1, 2, 3, 4 and 6 % wt. The liquid composite was magnetically stirred for 30 min.

When discussing the thermal properties of the enhanced phase change material, in the literature, both weight (mass) and volume fractions are used. In order to facilitate comparisons, the conversion between the volume and mass fractions for a two-component system is given below:

\[
\phi_{vol} = \frac{\phi_{wt} \rho_m}{\phi_{wt} \rho_m + \phi_{wt} \rho_d + (1-\phi_{wt} \rho_d)} \tag{1}
\]

with \(\rho_m=914 \text{ kg/m}^3\) and \(\rho_d=2200 \text{ kg/m}^3\) are the densities of the paraffin matrix and the dispersed component and \(\phi_{vol}\) and \(\phi_{wt}\) denote the volume and mass fractions, respectively.

**Measurements of the thermal properties**

For the thermal conductivity measurement we used a modified enthalpy-based water bath method, described in detail elsewhere (Lagaris et al, in press). Contrary to the T-History method, we use only one cylindrical tube having length of 25 cm and external diameter and thickness of 1.2 cm and 3 mm, respectively. The tube was a of Borosilicate glass Duran® having thermal conductivity of 1.2W/(mK) at 90°C and a density of 2.23 gr/cm³, while the specific heat (not provided from the producer) was evaluated as a weighted average of the specific heat of its constituents, i.e. 81% SiO₂, 13% B₂O₃, 4% Na₂O+K₂O and 2% Al₂O₃. Two NTC 5mm thermistors of 14 KOhms purchased from Mouser Corporation UK were used
for the temperature measurements, one at the center of the tube containing the PCM composite and the second one attached at the tube’s external surface. The temperature acquisition was obtained by means of the MCC USB Data Acquisition card. The tube with the PCM was initially equilibrated in a water bath at a certain temperature (well above the melting point), to subsequently being immersed into a cold water bath with a constant temperature at 10 °C. Taking into account the geometry of the tube, i.e. that its length is approximately 20 times longer than its diameter, we may assume that we are dealing with 1-Dimensional transient heat conduction problem in cylindrical coordinates. By nonlinear minimization procedure the thermal conductivity can be evaluated by adjusting the numerical predictions with the experimental data (Lagaris et al, in press).

The specific heat capacity and heat of fusion were measured by the Modulated Differential Scanning Calorimetry (MDSC) method. MDSC differs from the standard DSC in that it uses two simultaneous heating rates- a linear heating rate that provides information similar to standard DSC, and a sinusoidal or modulated heating rate that permits the simultaneous measurement of the sample’s heat capacity.

RESULTS AND DISCUSSION

The measured thermal conductivities as a function of weight fraction of the various graphite nanoplatelets can be seen in Figure 1. There is an overall increase in the thermal conductivity upon the nanoplatelets additions, although the effect is not linear and it is different for each particular type of nanoparticle. The largest increase can be seen to occur for the M15 sample in the low weight fraction regime up to 1% and at 6% weight fraction. The thermal conductivity is increased 100% to the value of 0.44 W/mK from the 0.22 W/mK for pure paraffin for 1% weight fraction (~0.4% volume fraction) and almost 250% at 6% weight fraction (~2.6% volume fraction). The thermal conductivity does not increase linearly. The increase observed though is similar to that found in the literature for a similar sized graphite nano-platelet of 15μm diameter (Xia et al, 2010, Kim and Drzal, 2009). Samples M5 and M15 differ only in the diameter, sample H5 has the same diameter as M5 but smaller overall surface area. Samples with M5 also show significant increase in the thermal conductivity of the order of 60% to 100% for weight fractions between 2 and 6% (~0.8% and 2.5% volume fraction). The composite with C750, the nanoplatelets with the smaller size, more spherical shape and the larger surface area has less effect on the thermal conductivity than the rest, a small increase of ~13%, rather constant for the whole range studied. At the higher end studied, 6% weight fraction (~2.5% volume fraction) for samples M5 and H5, the enhancement is leveling off or reduced in contrast to M15 in which the thermal conductivity is greatly enhanced.

Figure 1 Thermal conductivity of paraffin wax with graphite nanoplatelets as a function of weight fraction with different sizes, aspect ratios and surface area: (squares, M15) d=15 μm and t=6-8 nm, Surface Area=120-150 m²/g; (rombus, M5) d=5μm and t=6-8 nm, Surface Area=120-150 m²/g; (crosses, H5) d=5μm t=15 nm, Surface Area=50-80 m²/g; (triangle C750) d<2μm, thickness a few nm and surface area=750 m²/g.
In Figure 2 the heat of fusion is shown as a function of the weight fraction for the different nanoplatelets. It can be seen that for the small weight fraction of 1% that the thermal conductivity, in the case of samples M15, has increased 100%, the heat of fusion has only been reduced slightly (~2%). The heat of fusion is gradually reduced with the increase of weight fraction to about 10% for a 6% weight fraction. Similar trend is seen for the samples with C750 nanoplatelets. Interestingly, the M5 nanoplatelets seem to leave the heat of fusion largely unaffected up to 6% weight fraction.

**Figure 2** Heat of fusion as a function of weight fraction of (a) (squares, M15) diameter=15 μm thickness=6-8 nm, Surface Area=120-150 m²/g, (b) (M5) diameter=5 μm thickness=6-8nm, Surface Area=120-150 m²/g, (c) (triangles C750) d<2 μm, thickness a few nm and surface area=750 m²/g.

Similarly, the specific heat capacitance $C_p$ (Figure 3) shows negligible change as a function of weight fraction for the liquid phase for the M15 at the 1% weight fraction.

**Figure 3** Specific heat capacitance as a function of weight fraction of (a) liquid at 70°C and (b) solid at 20°C. Squares: M15, Rhombus: M5, Triangles: C750.
In the solid phase the respective change is of the order of 2% at the 1% weight concentration, increasing to 14% at the maximum weight fraction studied.

**CFD SIMULATIONS**

CFD simulations of the heat transfer process on a fin and tube exchanger (HE) were developed to assess the influence of the enhancement of the thermal properties of the PCM over the solidification/melting process. The simulations were developed for a HE with cooper tubes and aluminium fins with a single circuit six meters long. The tubes have 8.6 mm of internal diameter and an external diameter of 10 mm and are distributed in a square arrangement with the distance between the tube centers being 52.5 mm. The fin pitch is 10 mm and fin thickness is 0.3 mm. The PCM is placed around the tubes and the fins, water is the heat transfer fluid (HTF) and circulates through the tubes. The simulations were performed for a solidification/melting cycle of A53 and A53+1%NP, for a constant flow rate of the HTF 2.8 l/min, during the solidification the water inlet temperature was set at 47.2 ºC (five degrees below A53 solidus temperature) and then set to 58.6 ºC over the melting process (five degrees above A53 liquidus temperature). The simulations were initialized with the PCM fully melted at a uniform temperature of 55 ºC. The water flow is fully turbulent, the average velocity of the HTF inside the tubes is 0.8 m/s, the correspondent Re number is approximately 10000. The numerical simulations were developed with Ansys Fluent 17.1 software, the code solves the mass, momentum and energy transport equations for constant density. The turbulence was modelled by the two equations k-epsilon standard model and the energy equation is solved in the form of total enthalpy. The thermal properties of copper, aluminium, HTF and the PCMs considered in the CFD calculations are presented in Table 1. The simulations convergence criterion was $10^{-6}$ for the residuals of the mass, momentum, and turbulence equations and $10^{-12}$ for the solution of the energy equation.

**Table 1. Material Properties**

| Property            | HTF  | A53  | A53 + 1% NP | Cooper | Aluminium |
|---------------------|------|------|-------------|--------|-----------|
| $\rho$ [kg/m3]      | 992.6| 800  | 805         | 8978   | 2719      |
| $C_p$ [J/kgK]       | 4182 | 2300 | 2335        | 381    | 871       |
| $k$ [W/mK]          | 0.60 | 0.22 | 0.44        | 387.6  | 202.4     |
| $\mu$ [kg/m.s]      | 6.6 E-4 | 7.0 E-3 | 7.0 E-3 | -      | -         |
| Latent heat [kJ/kg] | -    | 195  | 192         | -      | -         |
| Solidus Temperature [ºC]| - | 52.2 | 52.2     | -      | -         |
| Liquidus Temperature [ºC]| - | 53.6 | 53.6      | -      | -         |

Figure 4 shows the time progress of the PCM liquid mass fraction and the heat transfer rate for the HTF over the solidification/melting cycle of A53 and A53+1%NP and Figure 5 presents the heat transfer rate to the HTF over the solidification and the melting process for both simulations. Despite the high density of tubes and fins of the HE arrangement the CFD results clearly show the advantages of enhancing the A53 thermal conductivity. The heat transfer rate is higher along the entire solidification/melting cycle for the enhanced A53 comparatively to simple A53 and both process (solidification and melting) are faster.
CONCLUSION

In conclusion, nanoplatelets of expanded graphite mixed in paraffin were found to increase the thermal conductivity at relative low weight concentration; 1% and 6% weight fraction of the M15 type, 15µm in diameter and 6-8nm thick graphite nanoparticles, increased the thermal conductivity by 100% to 250% respectively. At the same time the heat of fusion was only slightly reduced from 2-10% for the 1 and 6% weight fraction respectively. CFD simulations predict better thermal performances for the nano-composite material compared to the pure PCM. We believe that the present results provide an efficient and easy way for improving the thermal properties of PCMs, suitable for thermal storage applications.
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