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Thermal conductivity and heat transfer performance enhancement of phase change materials (PCM) containing carbon additives for heat storage application

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ABSTRACT

In thermal storage system, a high thermal conductivity of Phase Change Materials (PCM) is required to complement the crystallization phenomenon of the PCM during the discharging process. In this study, PCM with carbon additives, Multi-walled Carbon nanotube, Graphite and Graphene, are manufactured and the thermal conductivity of the PCM is measured by the transient hot-wire method for thermal storage application. The thermal conductivity of the PCM is enhanced by adding the carbon additives, and the effect of Poly Vinyl Pyrrolidone (PVP) as a dispersion stabilizer on the thermal conductivity is evaluated. It is found that the heat transfer rate enhances up to 3.35 times in the case of Graphite at 5.0 vol%. It is finally concluded that Graphite is the most promising candidate for heat transfer enhancement of stearic acid among three carbon additives even though Graphene gives the highest thermal conductivity enhancement.

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Amélioration de la conductivité thermique et de la performance de transfert de chaleur de matériaux à changement de phase (PCM) contenant des additifs de carbone pour une application d'accumulation thermique

Mots clés : Nanotube carbone ; Graphite ; Taux de transfert de chaleur ; Conductivité thermique ; Accumulation thermique

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Nomenclature

k_{pure}	thermal conductivity of the pure stearic acid, $\text{Wm}^{-1}\text{K}^{-1}$
k	thermal conductivity of the nano PCMs, $\text{Wm}^{-1}\text{K}^{-1}$
n	sample count
\dot{Q}	heat transfer rate (W)
R_1	fixed resistance, Ω
R_2	fixed resistance, Ω
R_v	variable resistance, Ω
R_w	resistance of hot-wire, Ω
u	uncertainty
\bar{x}	sample mean
X	sample measurement error of thermal conductivity

1. Introduction

Thermal storage system has been paid attention from the view points of the efficient storage and utilization of thermal energy. The purpose of thermal storage system is to collect the excess heat source, to secure the heat source stably, and to supply it for resolving the time discordance of energy supply and demand by thermal storage. This system is applied in a variety of ways in the related fields (Zalba et al., 2003; Shin et al., 1987; Jesumathy et al., 2012; Chang et al., 1999; Meng et al., 2011; Padhmanabhan et al., 2011; Fumoto et al., 2013). Zalba et al. (2003) reviewed the thermal energy storage with phase change on materials, heat transfer and applications including 150 materials and 45 commercially available phase

change materials (PCM). Recently, Osterman et al. (2012) reviewed the PCMs based cooling technologies for free cooling applications, encapsulated PCM, air-conditioning and sorption cooling systems.

During the heat exchange in thermal storage system using the PCM, the latent heat is released from the PCM to the low-temperature tube (discharging process). However, during the discharging process, the crystallization of the PCM starts on the outer wall of the low-temperature tube. Due to the crystallization of the PCM, the heat transfer performance is reduced between the PCM and the tube. The reduction of thermal storage/release rate is just the reflection of the heat transfer performance degradation caused by the low thermal conductivity of the PCM.

The addition of nano particle into the base fluid has been studied and developed to improve the low thermal conductivity of the base fluid (Maxwell, 1873, Choi, 1995). According to the reports, the addition of the nanoparticles with a high thermal conductivity can recover the low thermal conductivity of the PCM. Especially some studies reported about the improvement of the thermal conductivity by adding Multi-walled Carbon nanotube (MWCNT) or Graphite on the PCM. The thermal conductivity of the Paraffin wax including MWCNT composites increased with the mass ratio of MWCNTs (Jifen et al., 2009) and the thermal conductivity of Paraffin wax was increased by impregnating porous graphite matrices. (Andrew et al., 2006) Also, the thermal conductivity of SA increased with increasing mass fraction of expanded graphite and carbon fiber (Ali et al., 2007).

In this study, Stearic Acid (SA) is chosen as the PCM for the thermal storage system application at the working temperature of 71 °C, MWCNT, Graphite and Graphene are used as the additives, and Poly Vinyl Pyrrolidone (PVP) is used as a dispersion stabilizer of carbon additives (Lee et al., 2008a,b). It

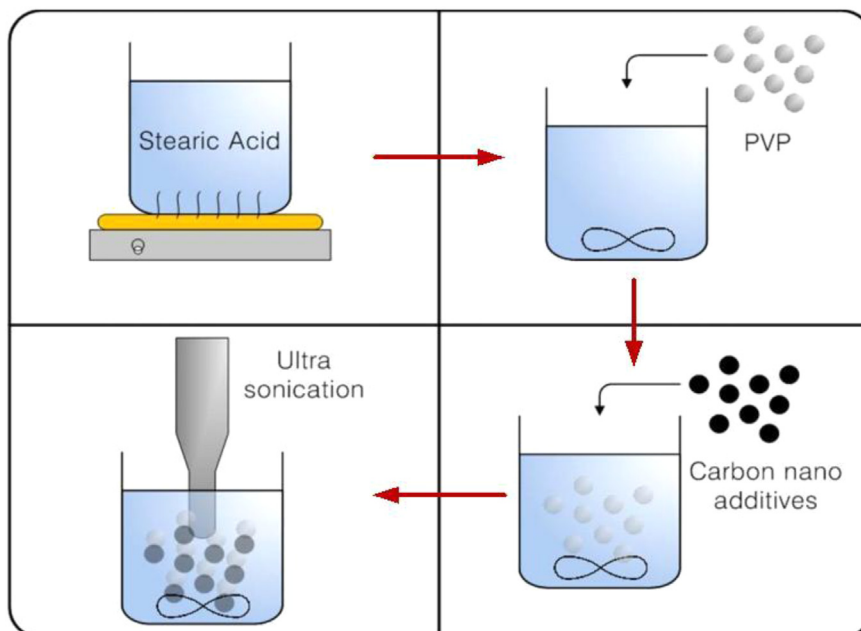


Fig. 1 – Manufacturing procedure for PCMs.

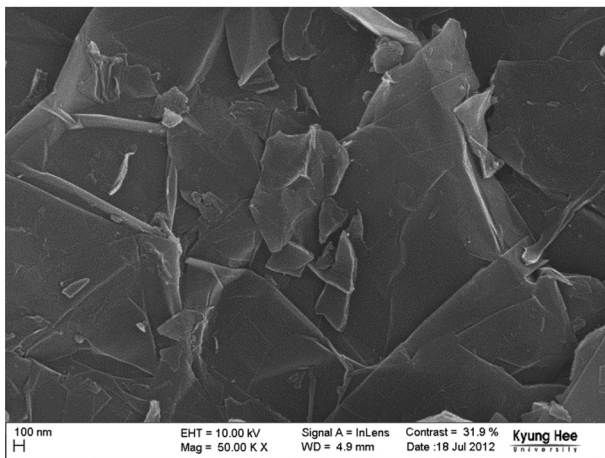
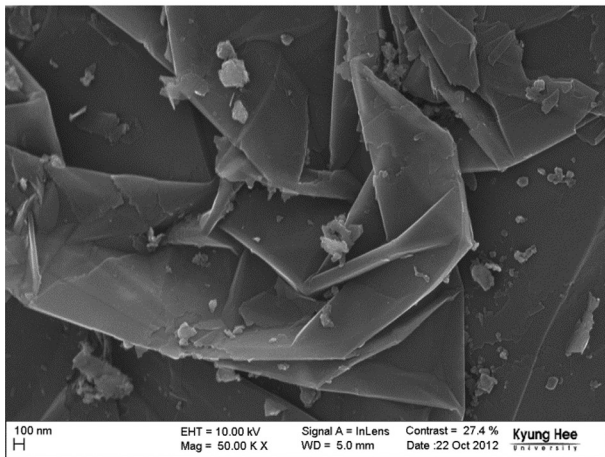
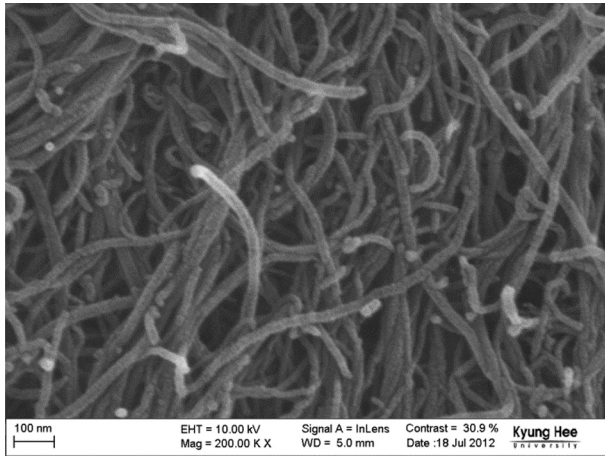


Fig. 2 – FE-SEM images of MWCNT (above), Graphite flake (middle) and Graphene nano-powders (below) used in this study.

has been reported that the addition of the carbon additives does not affect the solidification point of the nano PCMs while it does a little the melting point. (Li et al., 2013a). After making the PCMs, the dispersion stability in liquid state is evaluated. The thermal conductivity of the PCMs depending on the concentration of the carbon additives is measured in liquid state using the transient hot-wire method. Also, to verify the effect of the thermal conductivity enhancement on the heat

Table 1 – Properties of materials.

Stearic acid	Purity (%)	95.0
	Melting point (°C)	64.0–71.0
	Latent heat capacity (kJ kg ⁻¹)	203
	Thermal conductivity (Wm ⁻¹ K ⁻¹)	0.33/0.17 (Solid/Liquid)
Multi-walled carbon nanotube	Purity (%)	>95
	Average diameter (nm)	25
	Density (gm ⁻³)	2.1
Graphene	Thermal conductivity (Wm ⁻¹ K ⁻¹)	–3000
	Purity (%)	>99.5
	Average thickness (nm)	7
	Density (gm ⁻³)	2.2
Graphite	Thermal conductivity (Wm ⁻¹ K ⁻¹)	–5000
	Purity (%)	>95
	Average thickness (µm)	5
	Density (gm ⁻³)	2.25
	Thermal conductivity (Wm ⁻¹ K ⁻¹)	–200

transfer performance, it is applied for the real thermal storage system during the discharging phase.

The objectives of this study are to measure the thermal conductivity of the PCMs and to study the effect of the dispersion stabilizer of carbon additives (MWCNT, Graphite and Graphene) on the thermal conductivity enhancement for thermal energy storage application at a high working temperature of 71 °C. The effect of carbon additives on the heat transfer performance is also confirmed for the thermal storage application.

2. Experiment

2.1. Preparation of the PCM

The PCMs are prepared by the following four-step method. Fig. 1 shows the schematic diagram of manufacturing procedure for PCMs. First, the Stearic acid (supplied by DAE JUNG Chemical & Metal) is heated to make it in liquid state. Second, the PVP (supplied by Aldrich Chem.) as a dispersion stabilizer is added into fully melted SA and well dispersed using a stirring machine. Third, the carbon nanoparticles are added into the mixture prepared by step 2. Fig. 2 shows the FE-SEM images of the MWCNT (supplied by CNT CO., LTD.), Graphite flake (supplied by SGL GROUP) and the Graphene nano powder (supplied by Enanotec) used in this study. Then, finally the PCMs are made by using an ultrasonic disruptor for 2 h. The carbon additives concentration range of PCMs is from 0.001 to 0.1 vol%. Tables 1 and 2 show the properties of materials and

Table 2 – Experimental conditions.

Phase change material	Stearic acid	
Carbon additives	Multi-walled carbon nanotube Graphite Graphene	
Dispersion stabilizer	Poly Vinyl Pyrrolidone (PVP)	
Fluid temp.	71 °C	
Total vol. of sample	600 mL	
Ultra sonication	Time	120 min
	Frequency	20 kHz
	Power	350 W

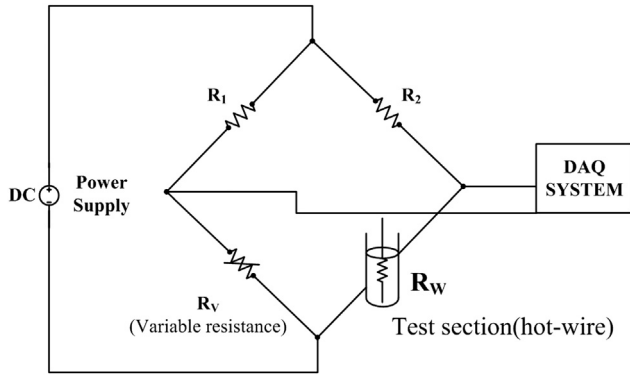


Fig. 3 – Schematic diagram of the transient hot-wire method.

the detailed experimental conditions conducted in the present study, respectively. To evaluate the dispersion stability in liquid state, the PCMs are observed in the thermal chamber at a constant temperature of 71 °C. It has been reported that the addition of the carbon additives does not affect the solidification point of the nano PCMs while it does a little the melting point. (Li et al., 2013a).

2.2. Transient hot-wire method

The transient hot-wire method is used to measure the thermal conductivity of the PCMs in this study. In the transient hot wire method, the time rate of change in the electric resistance of metal-wire is measured by its temperature dependence. The time rate of temperature change in metal-wire during the electric heating process varies with the thermal conductivity of the surrounding fluids which are PCMs in this study. The rate of heat dissipation to the fluid depends on the thermal conductivity of the fluid. In contrast to the steady state measurement method, the transient hot-wire method can be free from the effect of natural convection due to its promptness in the measurement (Lee et al., 2008a,b).

Figs. 3 and 4 show the schematic diagrams of the transient hot-wire method and the test section of the sensor, respectively. The experimental setup consists of the Wheatstone bridge circuit in connection with the power supply, high accuracy standard resistors, a high accuracy variable resistor, and the test section including the Pt-wire exposed in the test fluids. The test section (left part in Fig. 4) is inserted into the cylinder (right part in Fig. 4), which contains the test fluid. The Pt-wire (Teflon-insulated platinum wire) of 190 mm in length and 50 μm in diameter is used as the metal wire. The Pt-wire is coated with the Teflon layer to prevent the current leakage to the test solution (Hong et al., 2011; Nagasaka and Nagashima, 1981).

2.3. Uncertainty analysis

The uncertainty analysis is carried out by the GUM-method to verify the measurement system (Labudova and Vozarova, 2002).

The uncertainty $u(x_i)$ can be expressed as

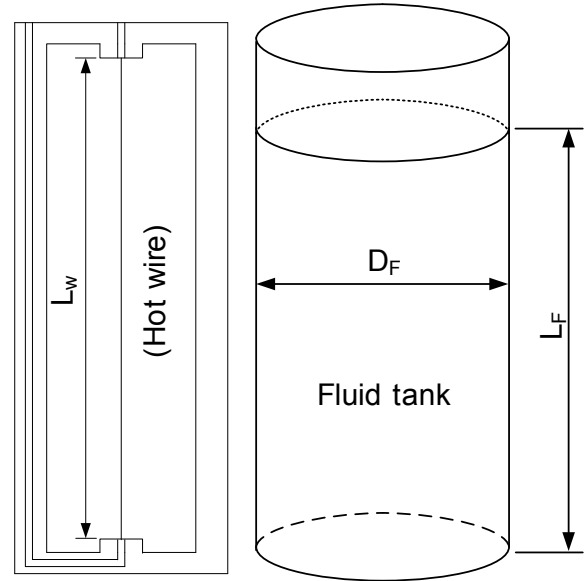


Fig. 4 – Test section for the transient hot-wire method.

$$u(x_i) = \sqrt{\frac{1}{n(n-1)} \sum_{k=1}^n (X_{i,k} - \bar{X}_i)^2} \quad (1)$$

where x_i , $u(x_i)$, n , X and \bar{X} are sample mean, standard uncertainty associated with the input estimate, sample count, sample measurement error of thermal conductivity and average sample measurement error of thermal conductivity, respectively.

The verification of the transient hot-wire setup is done by comparing the measured thermal conductivities of a test sample with that from literature. The Ethylene Glycol (EG) is used as the test sample prior to the main experiments. Table 3 shows the experimental verification results of the transient hot-wire method and the maximum standard uncertainty obtained by the statistical analysis is estimated as 0.08%

Table 3 – Experimental verification of transient hot-wire method.

Fluid	Ethylene glycol	
Literature value (71 °C)	k_{EG} (Wm ⁻¹ K ⁻¹) 0.2694	
Test no.	Measurement values	Experimental error (%)
1	0.2641	-1.9784
2	0.2721	0.9873
3	0.2690	-0.1521
4	0.2700	0.1983
5	0.2704	0.3635
⋮	⋮	⋮
29	0.2737	1.6109
30	0.2732	1.4120
Mean	0.2751	1.8590
u	0.000821	
u (%)	0.08	

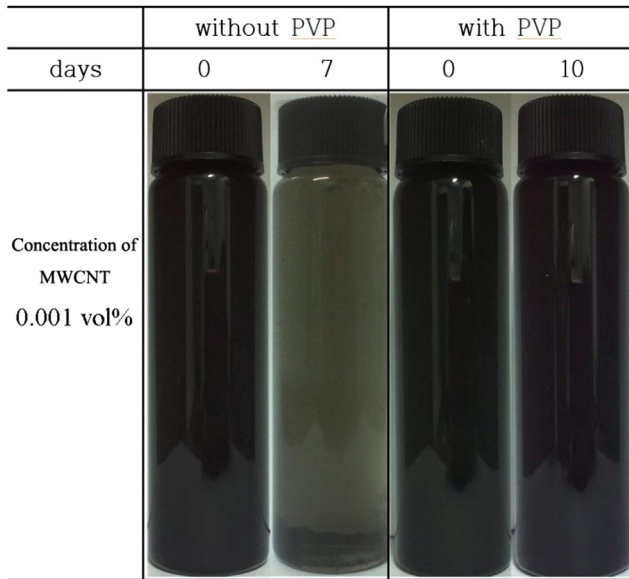


Fig. 5 – Comparison of dispersion stability for SA + MWCNT with and without PVP.

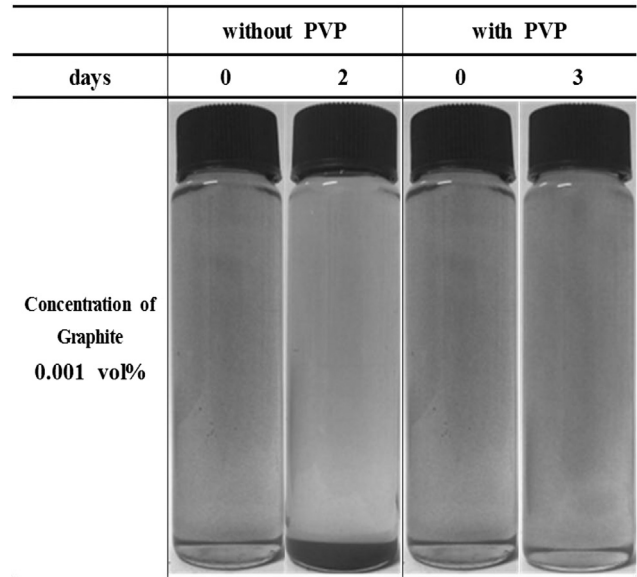


Fig. 7 – Comparison of dispersion stability for SA + Graphite with and without PVP.

3. Results and discussion

3.1. Compatibility of nano PCMs

Fig. 5 shows the comparisons of the dispersion stability for SA + MWCNT with and without PVP. The concentration of MWCNT is 0.001 vol% and the ratio of MWCNT and PVP is set to be 1:5 (Lee et al., 2008a,b). In the case without PVP, MWCNT nanoparticles are coagulated each other and sedimented after 7 days. On the other hand, in the case with PVP, the dispersion of SA + MWCNT is well maintained for 10 days.

Fig. 6 shows the comparisons for the dispersion stability of SA + Graphene with and without PVP. The concentration of

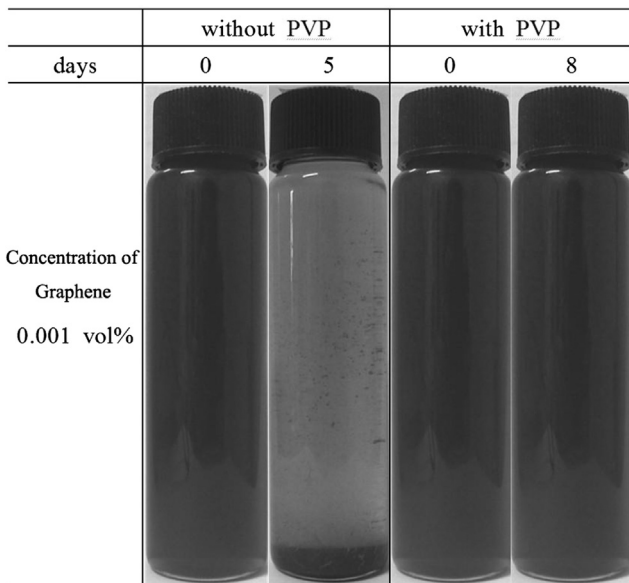


Fig. 6 – Comparison of dispersion stability for SA + Graphene with and without PVP.

Graphene and the ratio of Graphene and PVP are same as MWCNT case. In the case without PVP, the Graphene nanoparticles are sedimented to the bottom in 5 days. In contrast, in the case with PVP, the dispersion of SA + Graphene is maintained for 8 days.

Fig. 7 shows the comparisons of the dispersion stability for SA + Graphite with and without PVP. The concentration of Graphite and the ratio of PVP is same as other cases. After 2 days, in the case without PVP, most of Graphite nanoparticles are sedimented. However, in the case with PVP, the dispersion of SA + Graphite is maintained at least 3 days.

Based on these three cases, it is found that the dispersion stability becomes higher in the case with PVP than that without PVP. Generally, MWCNT, Graphite and Graphene particles are tangled and wound around each other and it is

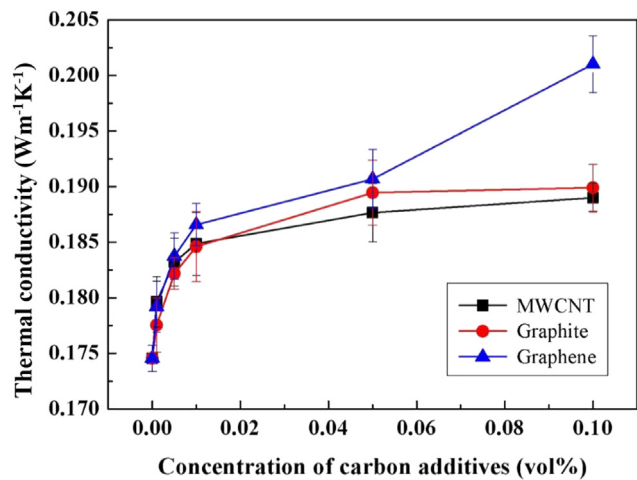


Fig. 8 – Variation of thermal conductivity as a function of the concentration of carbon additives at liquid state without PVP.

difficult to apply their fine powders to many commercial area because of their strong cohesion. These cohesions are caused by intermolecular force such as van der waals force (Lee et al., 2008a,b). In this case, the surfactant helps MWCNT, Graphite and Graphene to be well dispersed inducing the electrostatic repulsive force or the steric repulsive force by coated onto the particle surface. The PVP as a dispersion stabilizer, which is a nonionic surfactant, promotes the dispersion of carbon additives having a hydrophobic property in the organic solvent.

3.2. Thermal conductivity at liquid state

Fig. 8 shows the variation of the thermal conductivity of the PCMs depending on the concentration of carbon additives without PVP. The thermal conductivity of the nano PCMs are measured in 2 h after preparation. It is found that as the concentration of carbon additives increases, the thermal conductivity of the PCMs without PVP increases and the case of 0.1 vol% shows the highest value. Fig. 9 shows the corresponding effective thermal conductivity ratio of the PCMs without PVP as a function of the concentration of carbon additives. In the case of 0.1 vol% of Graphene, the enhancement ratio of the thermal conductivity is 15.2% and those of Graphite and MWCNT cases are 8.79%, 8.27%, respectively. Li et al. (2013b) measured the thermal conductivity of the PCMs without PVP at solid state by the Hot Disk thermal constants analyzer, and found that the thermal conductivity of the solid PCMs also increased with increasing the concentration of the carbon additives. For example, the thermal conductivity of pure stearic acid is about $0.26 \text{ W m}^{-1} \text{ K}^{-1}$, and it increases to 0.45, 0.35 and $0.75 \text{ W m}^{-1} \text{ K}^{-1}$ respectively for the MWCNT-based, Graphene-based and Graphite-based nanocomposites with the content of 1.0% additives.

Fig. 10 shows the variation of the thermal conductivity of the PCMs as a function of concentration of carbon additives with PVP. It is found that as the concentration of carbon additives increases, the thermal conductivity of the PCMs with PVP increases and the case of 0.1 vol% shows the highest value. Fig. 11 shows the corresponding effective thermal

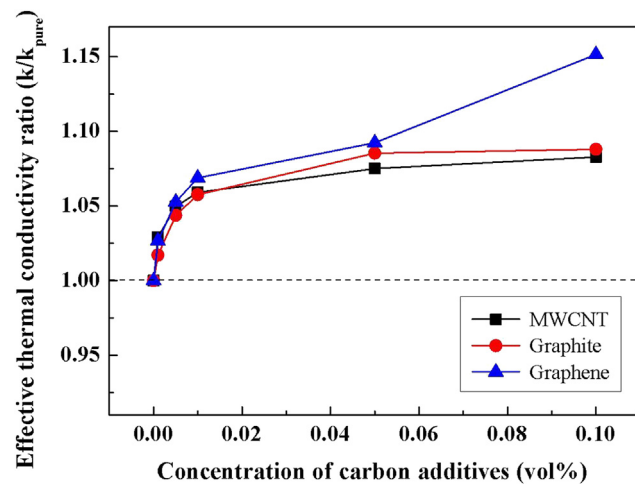


Fig. 9 – Effective thermal conductivity ratio as a function of the concentration of carbon additives at liquid state without PVP.

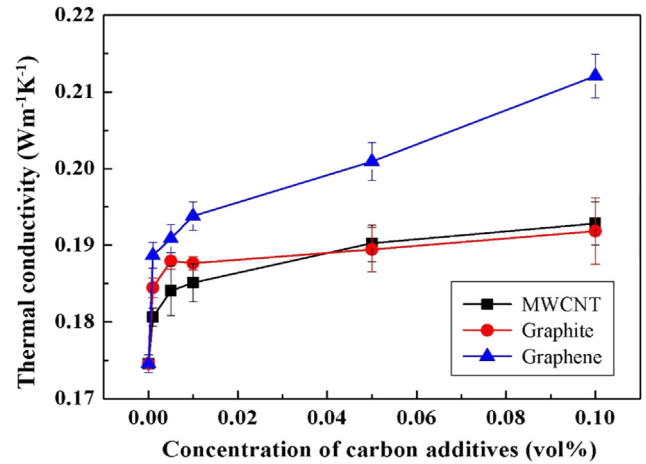


Fig. 10 – Variation of thermal conductivity as a function of the concentration of carbon additives at liquid state with PVP.

conductivity ratio of the PCMs with PVP as a function of the concentration of carbon additives. In the case of 0.1 vol% of Graphene, the enhancement ratio of the thermal conductivity is 21.5% and those of MWCNT and Graphite cases are 10.5%, 9.91%, respectively.

By comparing Fig. 9 with Fig. 11, it is found that the effective thermal conductivity ratio in the case with PVP is higher than that without the PVP. This is because the addition of the PVP improves the dispersion stability of the nanoparticles as explained in Section 3.1. In addition, the case of Graphene shows a higher thermal conductivity than that of MWCNT and Graphite, especially in high concentrations.

In general, there are several models to explain the mechanism of thermal conductivity enhancement of nano-fluids; Brownian motion, micro convection cell model, liquid layering theory and ballistic heat transport (Kleinstreuer and Feng, 2011; Wang and Mujumdar, 2007). However these theories are limited to explain the behavior of the thermal conductivity enhancement of MWCNT, Graphite and

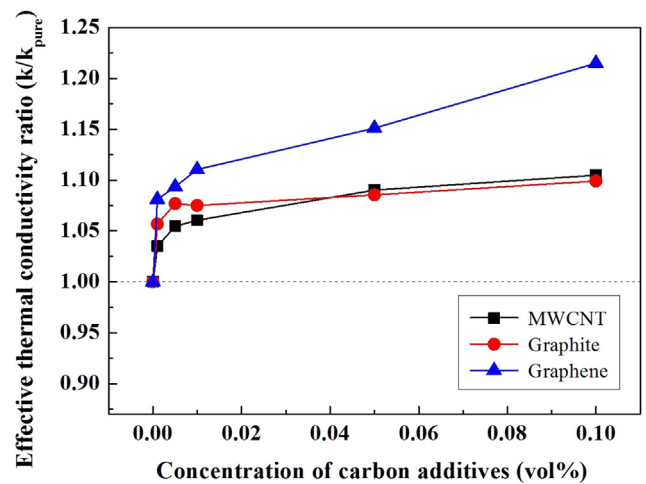


Fig. 11 – Effective thermal conductivity ratio as a function of the concentration of carbon additives at liquid state with PVP.

Graphene nanofluids. The shape and size of the nanoparticles play important roles in defining the physics of the heat conduction. Due to the high aspect ratio of MWCNTs, the interaction between them is considered to be highly probable forming a chain structure. Also the flake structure in two-dimensional lattice of Graphene is the chain structure similarly to MWCNT (Gupta et al., 2011). This chain formation leads to the percolation model (Venkata Sastry et al., 2008). According to the model, MWCNT having a high aspect ratio and Graphite or Graphene being a planar sheet form long chains of interconnected networks which act as conducting paths. The formation of a low heat flow resistance by the percolation network could potentially explain the enhancement of the thermal conductivity (Hu et al., 2004). It is found that the thermal conductivity of the PCM with Graphene is much higher than those with MWCNT and Graphite even though they have a similar percolation network structure. This is because the thermal conductivity of Graphene is approximately 1.5 times higher than those of MWCNT and Graphite.

3.3. Performance evaluation of the PCMs during the discharging phase

The increase of thermal conductivity of the PCM indicates that the carbon additives and the PVP, which can promote the uniform distribution of the carbon additives within the PCM, can play a beneficial role in enhancing the heat transfer performance of stearic acid. Experimental setup and data reduction process for heat transfer performance evaluation are explained in detail in Lee et al. (2008a,b). In this paper, the experimental test section and procedure are just briefly summarized. The test unit mainly consists of a heat storage

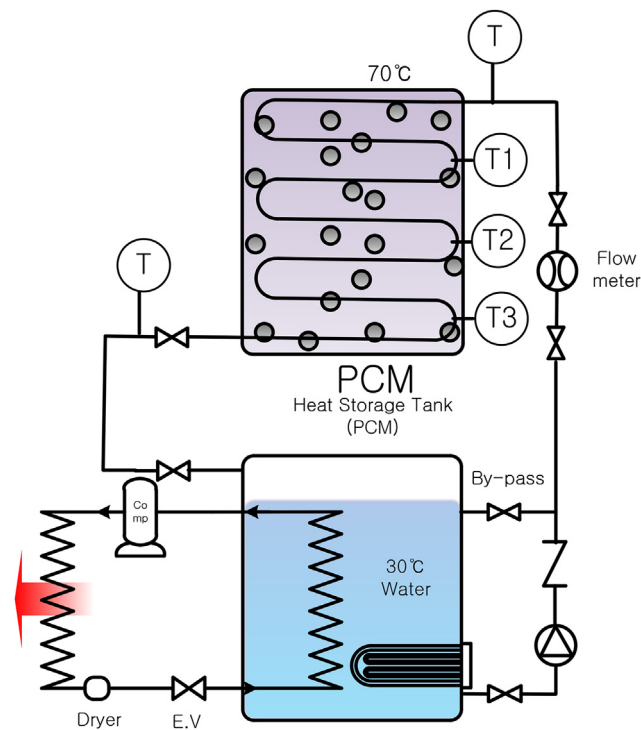


Fig. 12 – Schematic diagram of the experimental apparatus during the solidification process.

tank containing energy storage material and a thermostatic water bath as shown in Fig. 12. The heat storage tank is filled with the PCMs, and its working temperature is automatically controlled by the thermostatic bath with an accuracy of ± 0.1 °C with the help of an electrical heater and a compressor chiller. The heat storage tank and thermostatic water baths are insulated in order to reduce the heat losses to the ambient. The thermostatic water bath works as a heat sink during the discharging phase. To identify the heat transfer performance of the latent heat storage by the PCMs, a flow meter with an accuracy of 1.5% is used to measure the mass flow of the circulated cooling water. Three thermocouples are placed in the heat storage tank to measure the temperature of PCMs during the discharging process, and two thermocouples are utilized to measure the inlet and outlet water temperatures. The measurement error of temperature is within ± 0.5 °C by using four-wire type PT100 platinum resistance sensors.

Fig. 13 shows the heat transfer rate during the heat discharging process versus the concentration of the carbon additives. In the cases of Graphite and Graphene, the heat transfer rate increases as the concentration of carbon additives increases. In the case of MWCNT, the heat transfer rate increases with increasing the concentration until 1.0 vol%, after then it starts to decrease. It is considered that as the concentration of MWCNT increases over 1.0 vol%, the viscosity rises rapidly. As a result the natural convection effect on the process of the discharging phase decreases leading to the reduction of the heat transfer rate. It is found that the heat transfer rates enhance up to 2.22 times higher than the pure stearic acid for MWCNT (at 1.0 vol%), and 3.35 and 3.15 time for Graphite (at 5.0 vol%) and Graphene (at 5.0 vol%), respectively. The different results obtained from MWCNT-based, Graphite-based, and Graphene-based PCMs may be attributed to the combined effects caused by the enhancement of thermal conductivity of PCM in solid state and the restraint of natural convection of PCM in liquid state due to the addition of additives in phase change nanocomposites. The MWCNT and Graphene have low-dimensional structures according to the arrangement of carbon atoms when compared with Graphite as shown in Fig. 2. The SEM photograph shows that MWCNT matrix is composed of numerous individual multi-wall carbon tubes. Although the

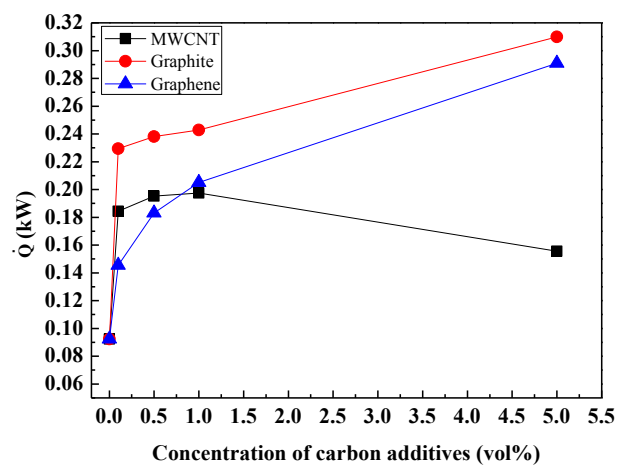


Fig. 13 – Heat transfer rate as a function of concentration of carbon additives.

additive of Graphite has a relatively low thermal conductivity compared with Graphene, a continuous and effective heat transfer matrix is more easily formed in the Graphite-based nanocomposite due to the fact that the Graphite additive is a very porous matrix and it has relatively high thermal conductivity in different heat transfer directions because of its structure. For the additive of Graphite, it appears that the increasing magnitude of thermal conductivity is always higher than the decreasing magnitude of natural convection in the liquid solution of stearic acid during the discharging process. It also appears that the restraint of natural convection has a more strong influence on the heat transfer of stearic acid in comparison with the thermal conductivity enhancement when the MWCNT and Graphene are employed as additives. Therefore, it is finally concluded that Graphite is the most promising additive for heat transfer enhancement of stearic acid among three carbon additives tested in the present study even though Graphene gives the highest thermal conductivity enhancement. This result agrees very well with the results from the differential scanning calorimeter (DSC) analysis for three different carbon additives (Li et al., 2013b).

4. Conclusions

In this study, the PCMs (Stearic acid + carbon additives) were prepared to enhance the thermal conductivity of SA as the PCM. The dispersion stability was evaluated and the thermal conductivity of the PCMs was measured by the transient hot-wire method and compared with the pure SA depending on the concentration of carbon additives. Furthermore, the effect of carbon additives on the heat transfer rate was evaluated. The results from the present study are summarized as follows;

- 1) In the case of MWCNT, the dispersion stability of PCM is maintained at least for 10 days by adding PVP as a dispersion stabilizer. In the case of Graphite and Graphene, the dispersion of PCM lasts for 3 and 8 days, respectively. The good dispersion stability of the PCM could be obtained by adding PVP in both cases.
- 2) At liquid state, it is found that the thermal conductivity of the nano PCMs increases with increasing the concentrations of MWCNT, Graphite and Graphene particles. The PCM with PVP gives a higher thermal conductivity than that without PVP. Especially, in the case of Graphene of 0.1 vol%, the thermal conductivity is enhanced up to 21.5%.
- 3) It is finally concluded that Graphite is the most promising additive for heat transfer enhancement of stearic acid among three carbon additives tested in the present study even though Graphene gives the highest thermal conductivity enhancement.

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