

The Performance Enhancement of Paraffin as a PCM During the Solidification Process: Utilization of Graphene and Metal Oxide Nanoparticles

Babapoor, Aziz*⁺; Haghghi, Ali Reza*

Department of Chemical Engineering, University of Mohaghegh Ardabili, Ardabil, I.R. IRAN

Jokar, Seyyed Mohammad

Department of Chemical, Petroleum and Gas Engineering, Shiraz University of Technology, Shiraz, I.R. IRAN

Ahmadi Mezjin, Mehdi*

Department of Chemical Engineering, University of Mohaghegh Ardabili, Ardabil, I.R. IRAN

ABSTRACT: *The substitution of fossil fuels with renewable energies is a meaningful way to mitigate global warming and air pollution. Phase change materials could store and release a high amount of energy. The solidification phenomenon is an essential factor that should be considered for choosing Phase Change Materials (PCMs). In this work, attempts have been made to improve the thermophysical properties of paraffin as a PCM during the solidification process. 1-3 wt.% of Al₂O₃, CuO, TiO₂, and graphene nanoparticles were used during the solidification process. No reports had yet been made on the effect of graphene nanoparticles versus metal oxide nanoparticles on the thermal properties of Nanoparticle-Enhanced Phase Change Materials (NEPCMs). The DSC, TGA, SEM, and FT-IR analyses were done to investigate the transition temperature, nanoparticle distribution, and nanocomposites morphology, respectively. It was seen that the addition of nanoparticles could effectively increase the thermal conductivities of paraffin. The maximum and minimum increases were in thermal conductivities were recorded in samples with 3wt.% of graphene and 1wt.% of TiO₂. The results showed that selecting suitable nanocomposites depended on various parameters, such as the type of nanoparticles and the weight percentage of nanoparticles. The PCM nanocomposites can be used to control the thermal management of different systems. The results can be applied in thermal design and management concepts, especially in the solidification process.*

KEYWORDS: *Solidification; Phase Change Material (PCM); Nanoparticle; Thermal properties; Energy storage.*

* To whom correspondence should be addressed.

+ E-mail: babapoor@uma.ac.ir ; babapoor2006@yahoo.com

● Other Address: Energy Management Research Center (EMRC), University of Mohaghegh Ardabili, Ardabil, I.R. IRAN
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INTRODUCTION

The replacement of fossil fuels with energy from renewable resources is of great interest. One of the most effective ways is to store these types of renewable energy. Phase Change Materials (PCMs) have been introduced as a novel method for thermal energy storage [1]. In 1980, the PCM technology was developed by NASA and used in astronauts' clothes to control their body temperatures. Later, more applications of this technology in the textile industry were investigated by researchers [2-7].

The PCM stores the thermal energy during the melting process and reveals this heat energy during the solidification process. The use of PCM technology in the textile industry improves the thermal properties of a textile. These materials should have proper and optimized thermal, physical, kinetic, chemical, and economical properties for consideration as a PCM [7]. Paraffin is the most commonly used PCM. They are linear hydrocarbons with the formula C_nH_{2n+2} that are usually derived from refined crude oil. As the chain length increase, the paraffin's melting point and latent heat of the fusion increase as well. They are chemically inert and stable at temperatures below 500 °C with a low vapor pressure at the liquid phase and low volume change on melting. These properties make paraffin have a very long melt-freeze cycle. Furthermore, they are low-cost and available in a broad range of temperatures [1, 8-12]. The disadvantages of paraffin, however, are low thermal conductivity, incompatibility with plastic containers, and flammability [1, 13-21]. Different materials such as carbon fiber and metal oxide nanoparticles incorporated with paraffin could be used to compensate for the low thermal conductivity of PCM. Phase change materials composites such as other advanced composites; have many benefits such as elevated thermal conductivity, oxidation resistance, and stable form [22-31]. Composite Phase Change Materials (CPCMs) can not only overcome the defects of a single organic or inorganic PCM but also improve the application effect and application scope of PCM [32-35].

Ho et al. [36] investigated the effect of Al_2O_3 nanoparticles on the thermal properties of paraffin. They used nonionic surfactant and emulsion techniques to disperse nanoparticles. *Dong et al.* [37] used electrospinning technology to embed silver nanoparticles on poly (vinyl alcohol) (PVA) and poly (vinyl pyrrolidone) (PVP)

nanofibers. In another work, *Cai et al.* [38] used electrospinning to fabricate PA6/PEG4000 nanofibers and worked on electro-spun PA6/PEG-blended nanofiber. *Teng and Yu* [39] studied the enhancing effect of mixing nanoparticles with paraffin. It is approved that the combination of graphene with polymers could enhance their thermal conductivity [40-42]. *Malekpour et al.* [43] checked the role of defect density on graphene thermal conductivity. The results showed that graphene thermal conductivity will reduce from 1800 W/m K to 400 W/m K by changing defect density from 2.0×10^{10} 1/cm² to 1.8×10^{11} 1/cm². *Kant et al.* [33] studied the heat-transfer phenomenon during the melting process of PCM dispersed with graphene nano-particles. The results indicated that the addition of graphene nanoparticles could enhance the melting rate of the PCMs.

Xin et al. [44] studied the thermal conductivities of graphene with different defect densities. They showed that defect-free graphene at 2200 °C has the highest thermal conductivity (3.55 W/m K), and graphene morphology also affects the grouping thermal conductivity. *Shen et al.* [45] investigated the impacts of size and number of nanographene layers on the thermal conductivities of the nanocomposites. Results showed that increases in size and the number of nanographene layers could elevate the thermal conductivity of PCMs. *Chu et al.* [46] reported that the lower thermal conductivity could be observed in a nanographene composite with higher wrinkles. The effects of the graphene layers on nanographene composites were studied in the works prepared by *Kim et al.* as well as *Hao Wang et al.* [47]. *Mohammadi Khoshraj et al.* [48] prepared PCM (butyl palmitate) composites as a microencapsulated form (MPCM) for use in gypsum wall applications. About 65 wt.% of MPCMs was butyl palmitate with 70.6 J/g of latent heat energy, which indicates the applicability of this synthesis MPCMs for thermal energy storage in gypsum walls.

Singh et al. [49] used polyethylene glycol (PEG-1000) as a phase change material. They used aluminum and carbon fins to increase the thermal conductivity of PCMs. The aluminum and carbon fins could enhance the thermal conductivity by 40 times and 33 times, respectively. *Li et al.* [50] approved the proper effects of Three-Dimensional Graphene (3DC) and discrete Graphene Flakes (GP) to improve the thermal conductivity, the heat of fusion, and shape stability of PCMs.

Table 1: The thermo-physical properties of the paraffin wax.

Thermo-physical properties	Units	Magnitude
Melting/ Solidification T	K	300.7
Latent heat fusion	KJ/kg	206
Thermal conductivity S-L	W/m K	0.18 - 0.19
Specific heat S-L	KJ/kg K	1.8 - 2.4
Density	kg/m ³	789 – 750

Table 1: The specification of nanoparticles.

Nanoparticle	CuO	Graphene	Al ₂ O ₃	TiO ₂
Purity	99%	99.5%	99%	99%
Color	Black	Black	White	White
Average particle size (APS) (nm)	<50	4-20 (Thickness)	80	20
Specific surface area (SSA) (m ² /g)	80<	-	10>	10-45
Diameter (μm)	-	5-10	-	-
Morphology	Almost spherical	-	Almost spherical	Almost spherical
Bulk density (g/cm ³)	0.79	-	0.79	-
Real density (g/cm ³)	6.4	-	6.4	-

Despite the various researches, there is an essential gap in the study of the thermal properties of nanocomposites during the solidification process. No reports yet were made on the effect of graphene nanoparticles versus metal oxide nanoparticles on the thermal properties of nanoparticle-enhanced phase change materials. In this work, paraffin is used as a PCM and TiO₂, CuO, Al₂O₃, and graphene oxide nanoparticles as thermal conductivity enhancement materials during the solidification process. The metal oxide nanoparticles have been selected because of their high thermal conductivities. On the other, they could be properly dispersed and stabilized in melted paraffin. DSC, SEM, TGA, and FT-IR tests were carried out to determine the properties of the nanocomposite, such as temperature variations, solidification heat, and morphology of nanocomposites, respectively.

EXPERIMENTAL SECTION

Liquid and solid paraffin waxes, Sodium Dodecyl Sulfate (SDS), and TiO₂, CuO, Al₂O₃, and graphene oxide nanoparticles were purchased from Merck & Co., Inc., SDS prevents the agglomeration of nanoparticles within

paraffin. A uniform dispersion could be obtained by SDS surfactants. The metal oxide nanoparticles have been dispersed and stabilized in melted paraffin. The specifications of the paraffin wax and nanoparticles were presented in Tables 1 and 2. A fan was applied to create cooling air for adjusting the solidification zone and solidification temperature range.

Material synthesis

A mixture of liquid-solid paraffin waxes containing 70 wt.% solid and 30 wt.% liquid were prepared as a base blend. In this regard, the solid paraffin was heated up to the melting point and then mixed with liquid paraffin. The nanoparticles were then distributed in paraffin uniformly. The nanocomposite could be prepared by mixing the nanoparticle, base blend, and SDS in an ultrasonic bath for 120 min at a constant temperature higher than the paraffin melting point. SDS was used as a surfactant to prevent the agglomeration of nanoparticles in PCM. Finally, the solid nanocomposite was made by mixture cooling down to room temperature. Table 3 shows the list of synthesized nanocomposites.

Table 2: The list of synthesized nanocomposites.

Sample name	Compounds	Weight percent (wt.%)
P	Paraffin (Base blend)	-
Cu-1	Paraffin + CuO + SDS	1
Cu-2	Paraffin + CuO + SDS	2
Cu-3	Paraffin + CuO + SDS	3
Ti-1	Paraffin + TiO ₂ + SDS	1
Ti-2	Paraffin + TiO ₂ + SDS	2
Ti-3	Paraffin + TiO ₂ + SDS	3
Al-1	Paraffin + Al ₂ O ₃ + SDS	1
Al-2	Paraffin + Al ₂ O ₃ + SDS	2
Al-3	Paraffin + Al ₂ O ₃ + SDS	3
G-1	Paraffin + Graphene + SDS	1
G-2	Paraffin + Graphene + SDS	2
G-3	Paraffin + Graphene + SDS	3

Evaluation and characterization of samples

The infrared spectroscopy tests were done on a Bruker Tensor 27 FT-IR with a spectral range of 4000 to 400 cm⁻¹ and wavenumber accuracy of 0.1cm⁻¹. It is equipped with accessories such as a KBr pellet maker and ATR cell. The Scanning Electron Microscope (SEM) Leo 1430-VP and differential scanning calorimetry (DSC) PT10 with the range of 100°C-350°C were used for the analysis of the samples. The ultrasonic bath (Lab750 model) at a fixed frequency of 22 kHz and a maximum power of 750 W was utilized for uniform sample preparation. The thermal conductivity of the PCM samples was calculated using a digital conductivity meter (Sahand Co., Iran, absolute thermal conductivity accuracy: ±3%), with a Pt/platinized electrode that is based on the transient hot-wire method. The measurements were repeated six times for each sample at a constant temperature, and the mean value is reported.

RESULTS AND DISCUSSIONS

Surface morphology

The sample SEM images were employed to examine the distribution of nanoparticles in paraffin. The homogeneity of nanoparticles exerts a significant impact on their thermal properties. Figs. 1-4 show the SEM images of pure nanoparticles and nanocomposites containing 1 wt.%, 2 wt.%, and 3 wt.% of different nanoparticles. The uniform distribution of nanoparticles within paraffin can be seen in these figures.

Thermal analysis

The results of DSC tests for paraffin in the temperature range of 15-110 °C were shown in Fig. 5. The onset, peak, and offset temperatures are presented in this figure. The lower curve (flow curve) is related to the melting process in which the PCM absorbs and stores thermal energy, whereas the upper one (return curve) belongs to the solidification process in which the PCM reveals its stored energy. It should be taken into account that the solidification process takes place at a practically fixed temperature for the bulk of the PCM due to high heat transported through convection in the liquid phase. After solidification of the whole liquid PCM, the extra heat is rejected from the solid PCM. It could be seen that the solidification temperature is lower than the melting temperature. Furthermore, the solidification process is faster than the melting process.

Fig. 6 shows the transition temperatures of the paraffin and nanocomposites during the solidification process (return curve of Fig. 5). The Ti-2 and G-1 samples have the highest, and lowest onset temperatures (50.8 °C, 47.9 °C) compared to the paraffin onset temperature (47.7 °C), respectively. The Cu-3 with a peak temperature of 42.9 °C and Ti-1 and Ti-3 samples with a peak temperature of 37.9 °C have the highest and lowest peak temperatures compared with the paraffin onset temperature (37.3 °C), respectively. The G-1 and Cu-samples have the highest and Ti-3 samples have the lowest offset temperatures

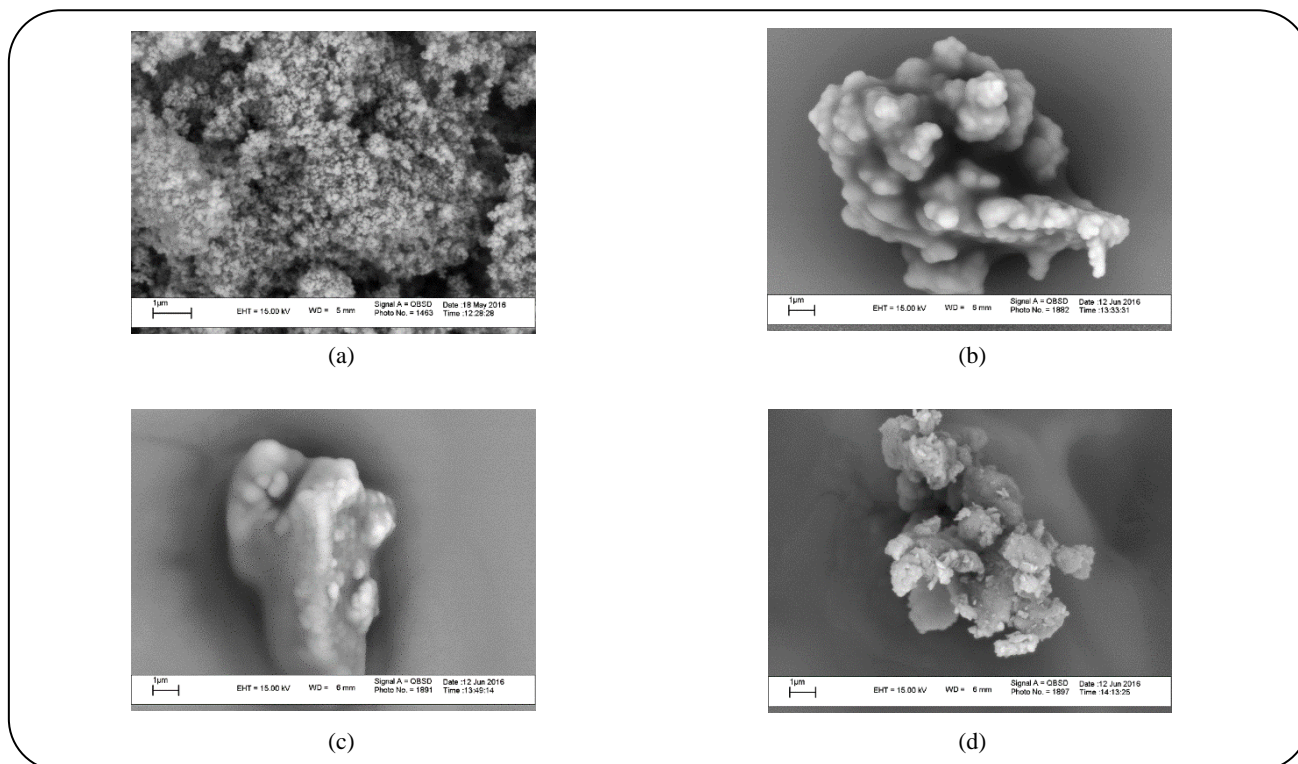


Fig. 1: The SEM images of a) TiO₂ nanoparticles b) TiO₂ nanocomposites (1wt.%) c) TiO₂ nanocomposites (2wt.%) d) TiO₂ nanocomposites (3wt.%).

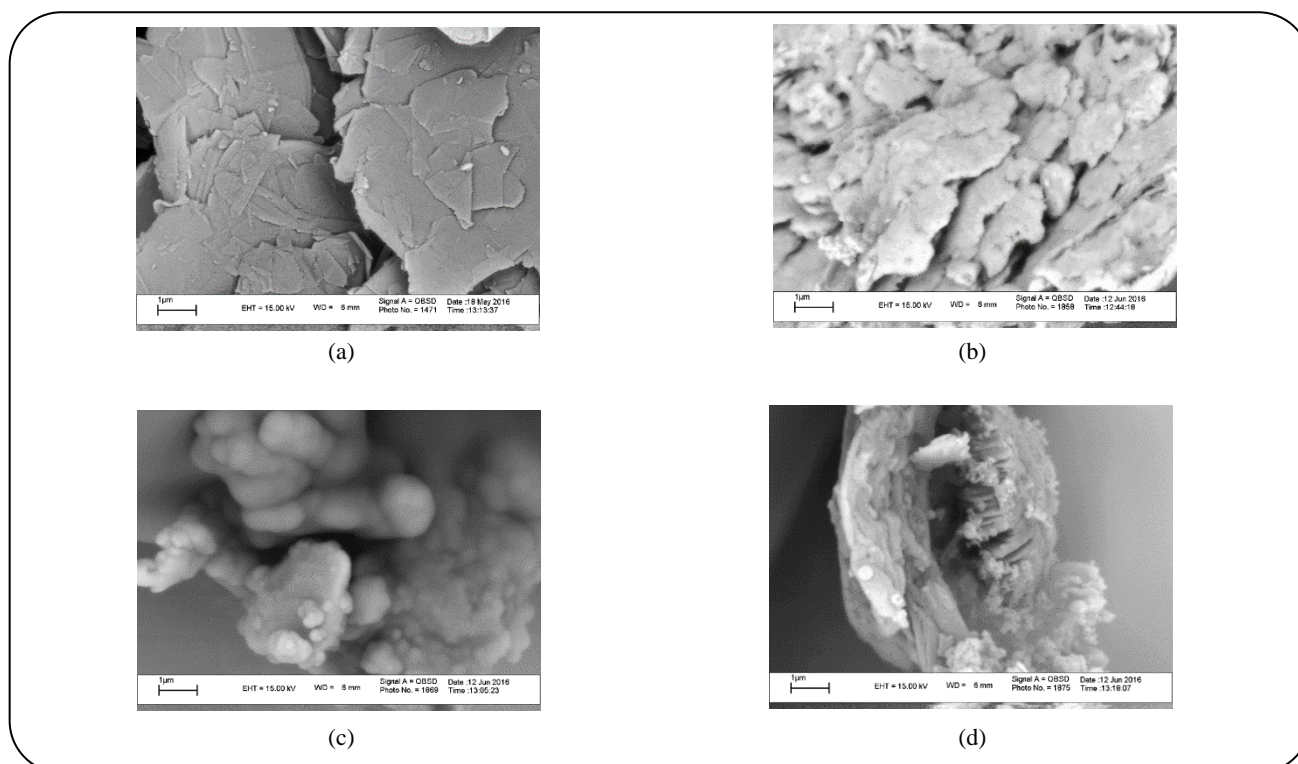


Fig. 2: The SEM images of a) Graphene nanoparticles b) Graphene nanocomposites (1wt.%) c) Graphene nanocomposites (2wt.%) d) Graphene nanocomposites (3wt.%).

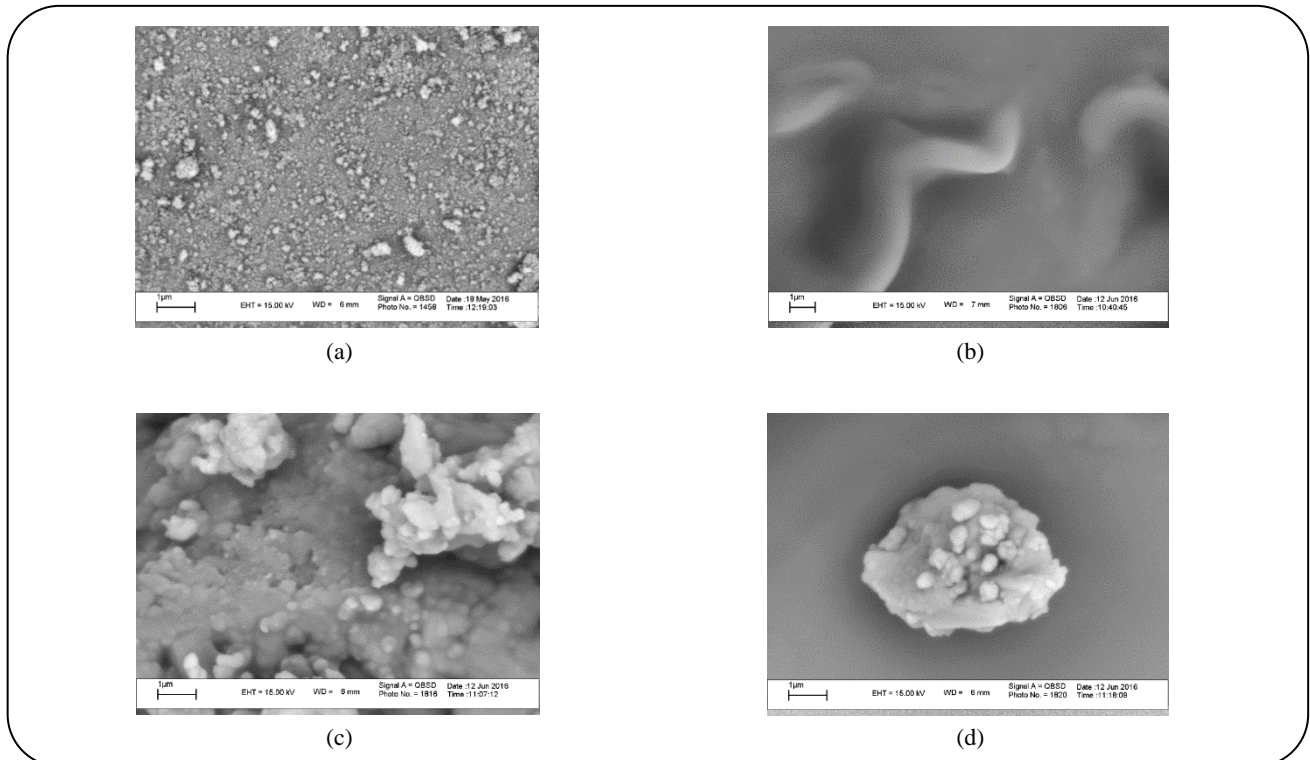


Fig. 3: The SEM images of a) Al_2O_3 nanoparticles b) Al_2O_3 nanocomposites (1wt.%) c) Al_2O_3 nanocomposites (2wt.%) d) Al_2O_3 nanocomposites (3wt.%).

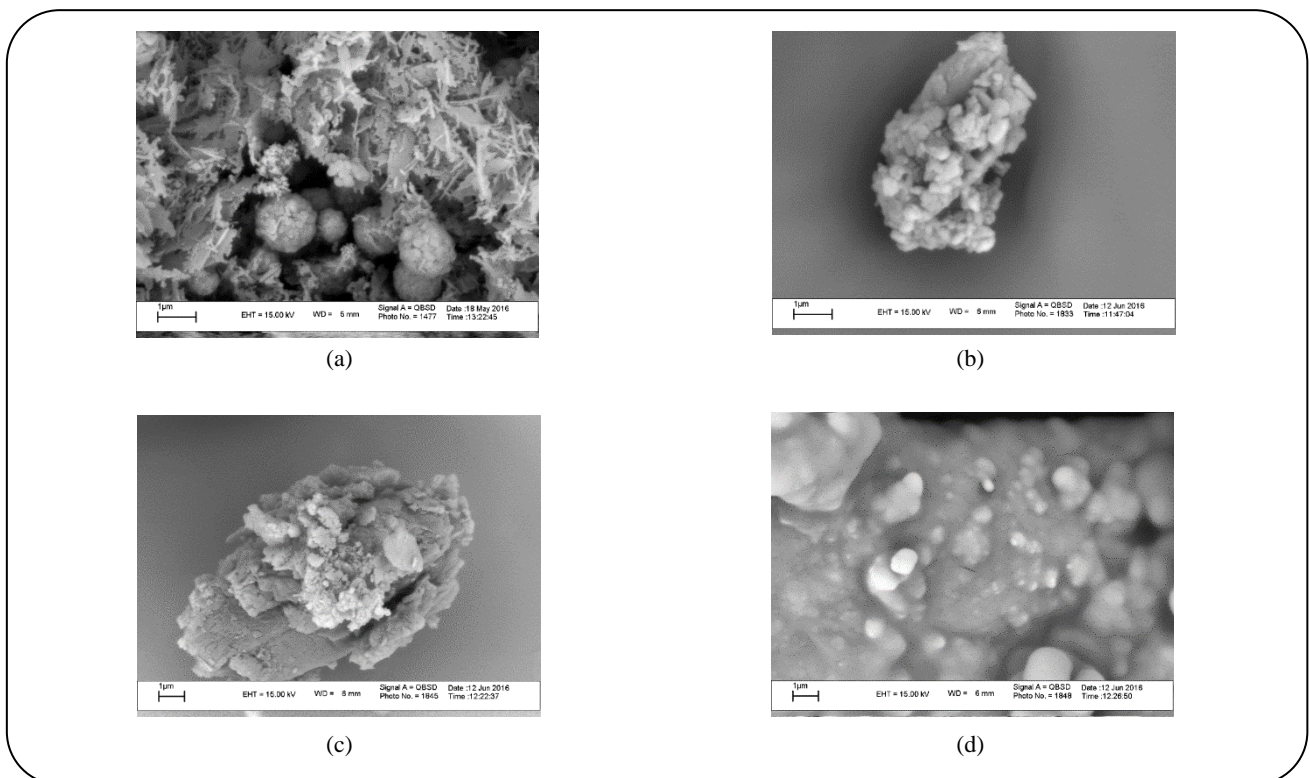


Fig. 4: The SEM images of a) CuO nanoparticles b) CuO nanocomposites (1wt.%) c) CuO nanocomposites (2wt.%) d) CuO nanocomposites (3wt.%).

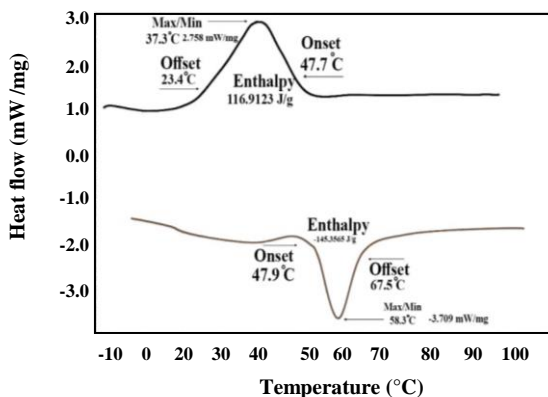


Fig. 2: DSC results of paraffin sample.

(30 °C, 24.8 °C) compared with the paraffin onset temperature (23.4 °C), respectively. Al-3 has the highest onset temperature (48.9 °C) among the Al₂O₃ nanocomposite samples. Al-2 has the highest offset and peak temperatures (29.5 °C and 40.5 °C) among the Al₂O₃ nanocomposite samples. Ti-2 has the highest onset and peak temperatures (50.8 °C and 42.2 °C) among the TiO₂ nanocomposite samples. Ti-1 has the highest offset temperature (25.4 °C) among the TiO₂ nanocomposite samples. Cu-3 has the highest onset and peak temperatures (50.3 °C and 42.9 °C) among the CuO nanocomposite samples. Cu-1 has the highest offset temperature (30 °C) among the CuO nanocomposite samples. G-3 has the highest onset, offset, and peak temperatures (49.9 °C, 30 °C, and 41.7 °C) among the graphene nanocomposite samples. The increase in nanoparticle percentage causes rising in these temperatures, which can be originated from proper size and homogeneous distribution of particles. Combining nanoparticles with paraffin leads to an increase in the onset temperature and allowed the solidification process to appear at elevated temperatures.

Fig. 7 shows the difference between the onset and offset temperatures of the samples during the solidification process. The maximum and minimum temperature differences belong to Ti-2 and G-2 (-25.4 °C and -18.9 °C).

Fig. 8 shows the enthalpy of samples in the solidification process. The Ti-2 and Al-2 samples have the highest and lowest enthalpies (163.555 J/g, 80.7969 J/g) compared with the paraffin enthalpy (116.9123 J/g), respectively. The enthalpy is an important factor that

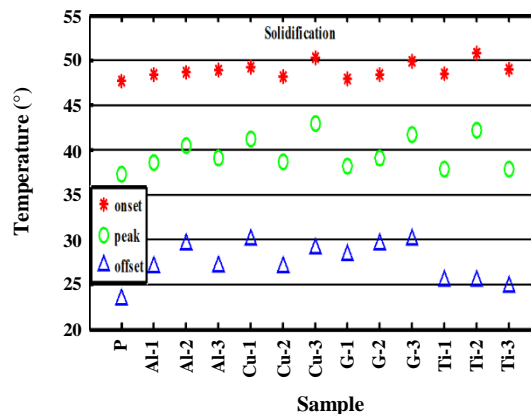


Fig. 6: The transition temperatures of paraffin and nanocomposites during the solidification process.

directly affects the thermal properties of PCM as an energy source. Therefore, the enthalpy of nanocomposites should not be much lower than paraffin. In general, adding nanoparticles can reduce the enthalpy value of the material. On the other hand, the nanoparticles increase the thermal conductivity of the PCMs. In fact, the addition of the nanoparticles tends to undesirably decrease the energy storage capacity because they usually do not contribute to the latent heat of solidification. The latent heat is expected to decrease non-linearly as the nanoparticle content increases. Therefore, the nanoparticles need to be optimized to improve the thermal conductivity without dramatically reducing enthalpy.

The processes of thermal degradation taking place in some samples were studied comparatively as the TGA curves (Fig. 9). At 700 °C, the thermal degradation for "P, Ti-1, Al-1, Cu-1 and G-1" samples are 99.7%, 79.7%, 97%, 98.1% and 96%, respectively. As can be seen in Fig. 9, the degradation process can be divided into three temperature zones. For "P" sample, the first temperature zone starts at 270 °C. This is while the thermal degradation of nanocomposites has proven to start at 280 °C-320 °C. The protective effect of nanoparticle film leads to an increase in thermal stability of nanocomposite at higher temperatures. The second temperature zone is limited between 320 °C and 410 °C. At temperatures higher than 350 °C the slope of the degradation curve for nanocomposites is greater than that for pure paraffin. This could result in a higher lifetime at elevated temperatures for the nanocomposites. At 500 °C the samples are marked as degraded (3rd zone).

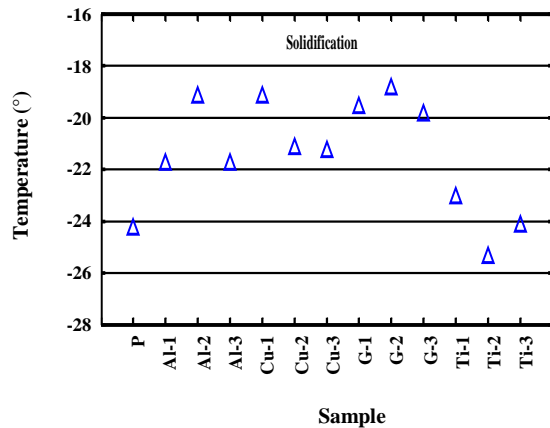


Fig. 7: The difference between the onset and offset temperatures of samples during the solidification process.

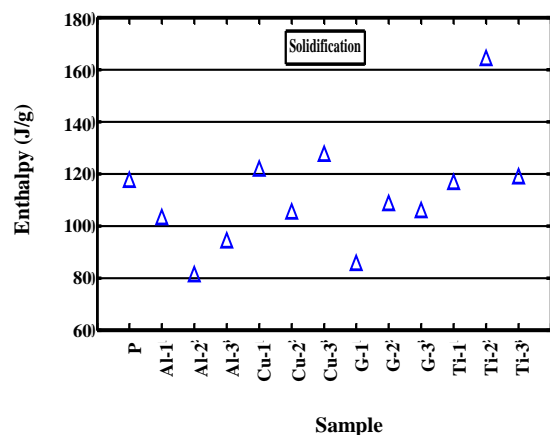


Fig. 8: Enthalpy of samples in the solidification process.

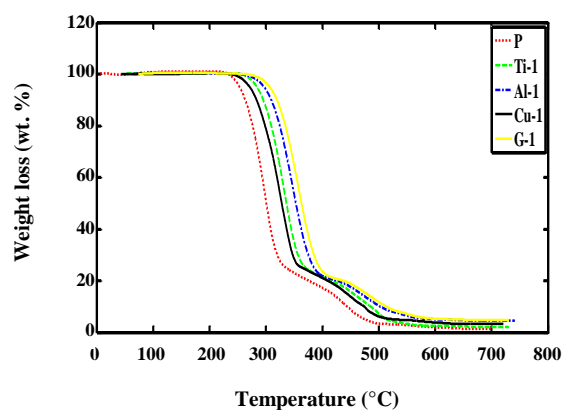


Fig. 9: TGA results of some samples.

Thermal conductivity

Figure 10 shows the results of the thermal conductivity of the tested samples. The addition of nanoparticles could potentially improve the thermal properties of paraffin. The thermal conductivity of nanographene is exceptionally high. The thermal conductivity of G-3 enhances by increasing the weight percentages of graphene oxide nanoparticles. Therefore, the highest thermal conductivity is observed in a sample with 3 wt.% of graphene (G-3). The minimum increase in thermal conductivities was recorded in the sample with 1wt.% of TiO_2 . It should be noted that the effect of nanoparticles on solidification viscosity is essential. The addition of nanoparticles changes the dynamic properties of the material. Namely, along with an increase in thermal conductivity, a significant increase in the viscosity of the samples is observed, which leads to a change in the hydrodynamics of the melt flow. The increase in the nanoparticles' volume fraction causes rising in thermal conductivity and viscosity. Therefore, the interaction of these two effects does not lead to an intensification of the solidification process [51, 52].

FT-IR analysis

Figs. 11 and 12 show the results of the FT-IR analysis of the samples. In G-3 sample, the peaks at 874.19 cm^{-1} , 1378.92 cm^{-1} , and 1463.97 cm^{-1} belong to graphene nanoparticles, and the peak at 2931.10 cm^{-1} belongs to paraffin (Fig. 11). In Cu-3, the peak at 723.14 cm^{-1} belongs to CuO nanoparticles, and the peaks at 2850.33 cm^{-1} and 2919.91 cm^{-1} belong to paraffin (Fig. 12). Since the spectra of the composites are similar to those of their compositions (paraffin and graphene nanoparticles), this indicates the formation of the nanocomposite. The paraffin peaks are at 1377.55 cm^{-1} , and 1465.15 cm^{-1} which is assigned as the stretching vibration of C-H. The peaks at 1375 cm^{-1} , and 1455 cm^{-1} are related to CH_2 bending vibration [53]. The absorption bands at 1076 cm^{-1} , and 1288 cm^{-1} are representative of C-O. The bands at 1398 cm^{-1} , 1739 cm^{-1} and 1058.35 cm^{-1} are related to C=C, C=O, and O-H, respectively [54].

CONCLUSIONS

CuO, Al_2O_3 , TiO_2 , and graphene nanoparticles were combined with paraffin to produce phase change materials composites. Sodium Dodecyl Sulfate (SDS) was used as a stabilizer in these compounds. DSC, SEM, TGA

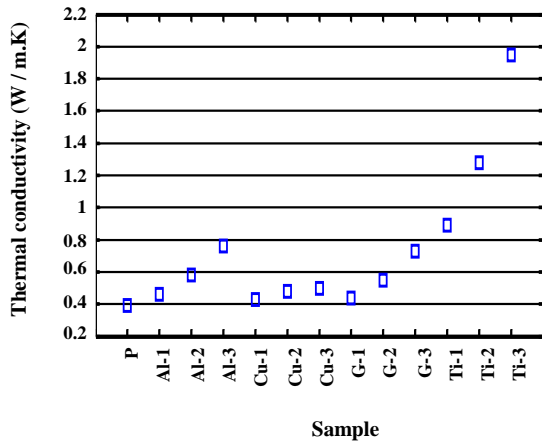


Fig. 10: Thermal conductivity of the samples.

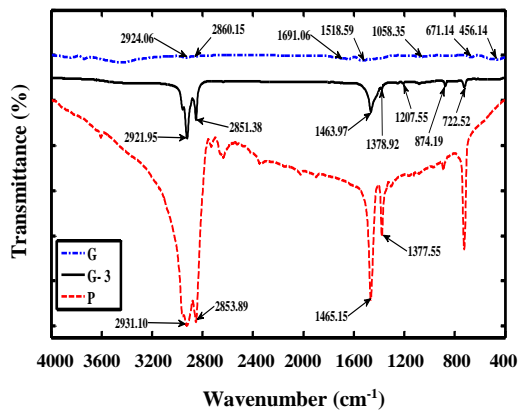


Fig. 11: The results of the FTIR analysis for graphene nanoparticles (G), graphene nanocomposite (G-3) and paraffin (P).

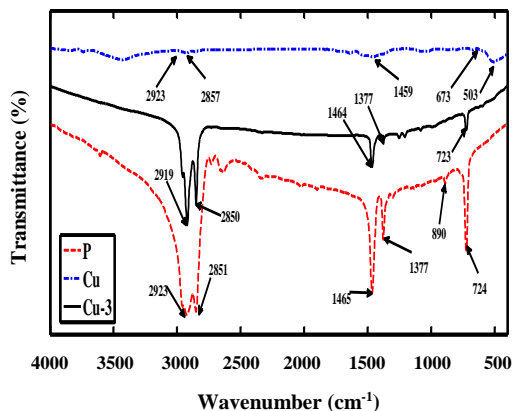


Fig. 12: The results of the FTIR analysis for CuO nanoparticles (Cu), CuO nanocomposite (Cu-3) and paraffin (P).

and FT-IR tests were performed to investigate the characteristics and thermal properties of the nanocomposites and their morphological structure. The SEM tests approved the uniform distribution of nanoparticles in prepared samples. The FT-IR results showed that no chemical reaction occurs between the pure paraffin and nanoparticles, and the nano enhancement phase change materials are the result of the dispersion of nanoparticles in paraffin. The DSC results showed that the highest and lowest latent heat during the solidification process belongs to the nanocomposites with 2wt.% of TiO₂ and 2wt.% of Al₂O₃, respectively. Furthermore, it was seen that the addition of nanoparticles could effectively increase the thermal conductivities of paraffin. The maximum and minimum increase in the thermal conductivities were recorded in samples with 3wt.% of graphene and 1wt.% of TiO₂. The protective effect of nanoparticle film leads to an increase in thermal stability of nanocomposite at higher temperatures. The experimental results showed that the selection of suitable nanocomposites depends on various parameters, such as the type of nanoparticles, the weight percentage of nanoparticles, etc. The PCM nanocomposites can be used to control the thermal management of different systems.

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