



Thermal Characterization of Eicosane/Graphite nano-Composite-Based Phase Change Material

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ABSTRACT

Phase Change Materials (PCMs) have received much consideration as thermal energy storage systems due to their high storage capacity. However, their heat transfer rate is limited because of the low thermal conductivity. Incorporating of carbon-based nanoparticles into the matrix of PCMs with good dispersion can be an efficient way to solve their deficiency. In this research, graphite nanoparticles were homogeneously dispersed within the Eicosane PCM matrix to prepare a Nano-Enhanced PCM (NEPCM). The main objective is to determine the optimum amount of graphite to maximize the thermal properties of NEPCM composites. The Scanning Electron Microscopy (SEM) images of the prepared nanocomposites confirmed the excellent dispersion of graphite nanoparticles within the Eicosane layers through an ultrasonic bath-assisted homogenization procedure followed by solidification. In addition, Differential Scanning Calorimetry (DSC) and Thermal Conductivity Evaluation (TC) of the samples were conducted to determine their heat capacity and thermal diffusivity. The results illustrated that the more the number of graphite nanoparticles, the larger the number of collisions between graphite and Eicosane. As the nanoparticle content increased, the thermal conductivity and diffusivity were enhanced, as well. Numerically, the maximum thermal conductivity was 4.1 W/m K for the composite containing 10wt% graphite, 15.66 times that of the pure Eicosane. Furthermore, increasing crystal growth and reducing heat capacity for the large number of nanoparticles in the composite were discussed. The significantly improved thermal properties of the prepared NEPCMs with an optimal nanoparticle content could make them applicable for different thermal management applications.

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1. INTRODUCTION

Nowadays, energy storage has become a vital issue for future generations. Energy storage-based technologies have widely been used to prevent increase in energy costs, loss of fossil fuel resources, and environmental unsustainability [1-3]. Since thermal energy has been used extensively, reserving heat energy and advancing energy efficiency can be suggested to decrease environmental and energy problems. Hence, the latest proposed method is the utilization of heat-storage materials composed of adequate chemical stability, high latent heat, and outstanding thermal stability in industrial and homemade applications [4, 5].

PCMs, so-called latent heat-storage systems, could be efficiently utilized for stated applications due to high latent heat, high energy storage capacity, enormous diversity, and wide operating temperature [6, 7]. Several organic and inorganic materials, mainly salt hydrates, clathrates, paraffin, and polyethylene glycol, can be used as effective PCMs [8]. Through many pieces of research, scientists declared paraffin-based hydrocarbons as a major PCM containing saturated and straight-chain organic compounds. Among different kinds of

paraffin, Eicosane has been widely utilized due to its outstanding characteristics, such as excellent thermal stability, non-toxic, no phase separation during phase transformation, and good chemical stability. Therefore, Eicosane-based PCMs are a promising energy storage medium through a solid-liquid phase change at a low melting temperature. Despite its advantages, its performance has been restricted by its low thermal conductivity [9, 10].

Numerous studies have been conducted to prevail over the shortage of thermal conductivity of paraffin waxes, which suggested the introduction of different inorganic (nanoparticles of metal oxides) and organic (polymer) materials to PCMs [11]. A summary of recent works on PCMs modified with additives, mainly nanoparticles, is represented in Table 1.

Al Ghossein et al. [12] examined the effect of silver (Ag) nanoparticles on the thermal conductivity of Eicosane-based PCM. They found that an increasing thermal conductivity (k) trend emerges by adding Ag nanoparticles up to 2wt%. However, there is no monotonic relationship between filler amount and thermal conductivity at higher Ag loadings. Carbon-based materials, such as carbon fiber, single and multi-

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walled carbon nanotubes, and nano-graphite, have shown high thermal conductivity [20]. Nano-graphite has a two-dimension (2D) layer structure and a large length-diameter ratio. Moreover, it has a larger specific surface area than expanded graphite and exfoliated graphite, which enables it to create the network architecture within PCM and enhance the heat transfer rate [21]. For example, Fang et al. [13] studied the changes in thermal conductivity of Eicosane composite containing graphene nanoplatelet at various loadings, showing a 400% improvement in thermal conductivity by adding 10% filler.

Despite numerous studies on various types of paraffin as PCM, there is a lack of a complete study on the optimized thermal properties, primarily thermal conductivity, of an Eicosane-based PCM. In this study, the main aim is to find the optimum amount of nano-graphite, homogeneously dispersed within Eicosane matrix, to maximize the thermal properties of the composite for different PCM applications. Furthermore, the mechanism of heat transfer in the presence and absence of filler aggregation is discussed.

2. EXPERIMENTAL

2.1. Materials

The materials used to synthesize the NEPCMs in the current study are as follows: pure Eicosane wax with a melting temperature (T_m) of $\sim 38^\circ\text{C}$ was purchased from Parschemical Company. Nano graphite (with average particle sizes of 10-80 nm) and polyvinylpyrrolidone-40 (PVP) as the nanoparticle dispersant were supplied by IranNanotech.

2.2. Preparation of Eicosane/Nano-graphite PCM

Different amounts of nano-graphite (2.5, 5, 7.5 and 10 wt %) and PVP as the surfactant were added to the melted PCM, denoted by EG2.5, EG5, EG7.5, and EG10, respectively. The mixture was stirred vigorously for 30 minutes at 75°C to form a homogeneous mixture. The homogenization process of the mixture was facilitated using an ultrasonic bath for 2 hours at 90°C followed by solidification in a short time (about 5 min). This procedure helped prevent the graphite nanoparticles from precipitation and ensured that the nanoparticles were well dispersed within the PCM matrix. The obtained mixture was evaluated during the repeated melting/freezing cycle at 50 to 25°C and reverse order. A schematic representation of sample preparation is illustrated in Figure 1.

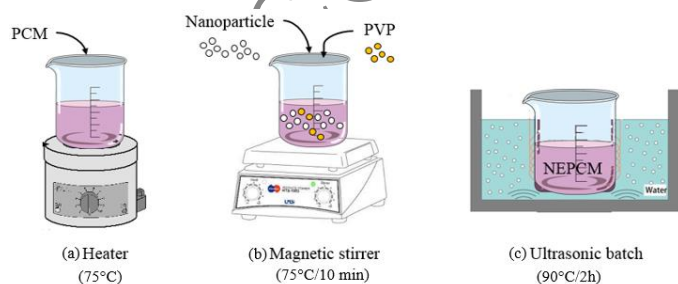


Figure 1. Schematic of preparation route of NEPCM samples.

2.3. Characterizations

The dispersion state of graphite nanoparticles into the synthesized NEPCMs was studied using scanning electron microscopy (SEM, Vega-TESCAN, Brno, Czech Republic),

operating at 20 kV using a secondary electron detector. Differential scanning calorimetry (DSC) analysis (Mettler Toledo, Switzerland) assessed the phase change characterizations of pure substances and NEPCMs under nitrogen coolant gas. The samples by weight of about 10 mg were added into a sealed aluminum pan. The thermal diffusivity (α) of each sample was measured with a Linseis LFA 500 Light Flash. The specific heat capacity (C_p) of NEPCM samples was determined with a Differential Scanning Calorimeter (DSC, Mettler Toledo, Switzerland). Also, the density of samples was measured using the test method ASTM D792. According to thermal diffusivity definition, thermal conductivity (k) of samples is calculated based on the following relation:

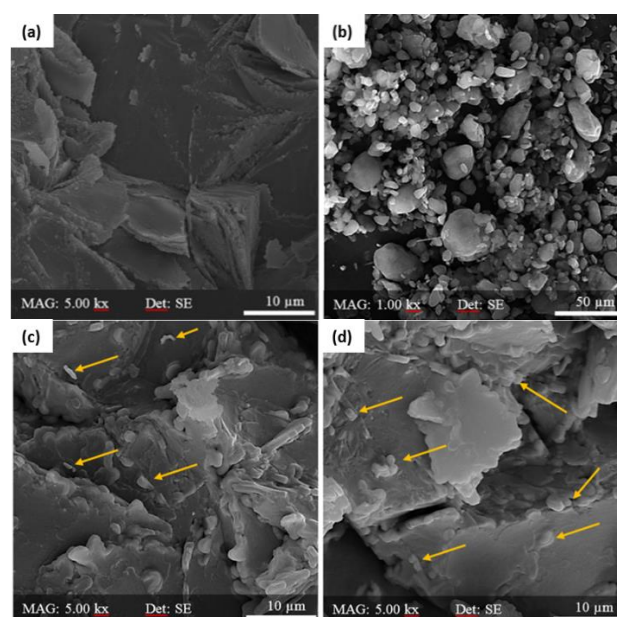
$$k_{NEPCM} = \alpha_{NEPCM} (\rho C_p)_{NEPCM} \quad (1)$$

The thermal behavior of all NEPCMs was examined in the temperature range of $0-70^\circ\text{C}$ at a heating rate of $5^\circ\text{C}/\text{min}$. All tests were conducted three times to ensure that the obtained results are reliable, and all the reported data is the averaged value with a deviation error within $\pm 5\%$.

3. Results and discussion

3.1. SEM analysis

The morphology and surface structure of all samples, including graphite nanopowders, pure Eicosane, and the prepared nanocomposites, were analyzed by SEM images, as shown in Figure 2. In these micrographs, a laminar microstructure of the graphite nanoparticles (Figure 2a) and an excellent dispersion of nanoparticles between Eicosane layers (Figure 2b-2f) can be seen. However, some small aggregates are shown in the samples containing high nanoparticle concentration (above 5%) due to graphite's high surface energy. Moreover, good interaction between Eicosane and graphite causes the integrated structure of nanocomposites without any loose interface.



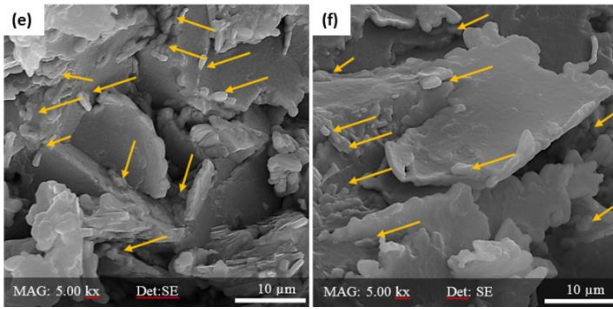


Figure 2. SEM images of (a) pristine Eicosane, (b) graphite nanopowders, (c) EG2.5, (d) EG5, (e) EG7.5, and (f) EG10 samples.

Table 1. Summary of the recent literature on modified PCMs with additives.

PCM material	Additive	Maximum thermal conductivity (k) [W/m K]	Main Finding	Ref.
Eicosane	Ag	0.5467	- The latent heat and the melting point of the samples decreased as the additives loading increased because of the reduction of the number of Eicosane molecules in the samples.	[12]
Eicosane	Graphene nanoplatelet (GNP)	2.1	- Decreased thermal interface resistance, attributed to the unique two-dimensional (2D) planar morphology of GNPs, was responsible for the improved performance of the composite.	[13]
n-docosane	Expanded-graphite	0.82	- The composite PCM with 10 wt% graphite was considered as form-stable allowing no leakage of melted paraffin throughout the liquid–solid phase change because of surface tension and capillary forces of graphite.	[14]
Paraffin	Nano-graphite	0.9362	- The graphite layers were randomly dispersed within the paraffin, and the thermal conductivity was gradually enhanced with the graphite content.	[15]
n-Eicosane	Expanded perlite & Carbon nanotube	-	- Compared to n-Eicosane/perlite composite, the utilization of carbon nanotubes improved the thermal conductivity without significantly affecting the compatibility of components, thermal stability, and thermal energy storage properties.	[16]
n-Eicosane	Mesoporous silica	1.171	- The composite demonstrated a shorter heat-charging period because of excellent thermal conduction by their interconnecting mesopores silica matrix.	[17]
n-Eicosane	Fe ₃ O ₄ @SiO ₂ @Cu	1.3926	- The composite microcapsule exhibited excellent heat transfer capability due to its high thermal conductivity and high thermal energy storage–release performance via suppressing super cooling.	[18]
n-Eicosane	Multilayer graphene	1.112	- The composite could effectively convert electricity into latent heat, in which its the electro-latent heat storage efficiency could exceed 59.9% at 1.9 V.	[19]

3.2. DSC analysis

PCM-based thermal energy storage systems have been designed to collect energy during melting. The heat flow triggers the melting process into the PCM. Since the main state of PCMs is solid at room temperature, heat transfer initially occurs by conduction, in which heat energy is transmitted through the molecules of PCMs. When temperature equals the melting temperature, in the melted part, convection would be the heat transfer mechanism that occurs to move fluid from warm regions to cold due to variation of PCM density. Thus, during the phase change, a transition period is observed in which energy is transferred

simultaneously through conduction and convection modes [22].

The DSC thermograms of pure Eicosane and NEPCM nanocomposites containing different concentrations of nanoparticles are shown in Figure 3 and Figure 4. Figure 3 presents the heating and cooling cycles of the pure Eicosane in the temperature range of 0°C to 70°C. The lower curve is related to the melting process, where PCM absorbs and stores thermal energy. The upper curve is associated with the freezing process where the PCM emits its stored energy to the ambience. The onset temperature is introduced as the baseline intersection with the melting curve [23]. The smaller peak (from 34 to 48°C) indicates solid-solid phase change due to the remodeling of the crystalline structure. The more significant height (48 to 62°C)

exhibits solid-liquid phase change and the latent heat amount [24].

Figure 4 shows the melting curve of NEPCM nanocomposites and pure Eicosane. The NEPCM with 2.5 wt% graphite had the highest onset temperature at 48.9°C, while the pure paraffin had an onset temperature of about 48.0°C. Therefore, the insignificant influence of the graphite nanoparticles on the composite melting point is observed. All samples have determined the melting points at about 53±0.2°C. According to the investigation of nanoparticle effects on Eicosane in the literature [25], Eicosane's melting temperature and thermal capacity are not affected by nanoparticles, but its thermal conductivity changes, which are in agreement with the current results.

The area under the melting peak is attributed to the enthalpy. The enthalpy value is expected to increase slightly after the small addition of nanoparticles due to the presence of different molecular interactions (van der Waals) between nanoparticles and the PCM matrix [20]. However, the results of this study demonstrated that increasing nanoparticle concentration decreases the amount of latent heat in NEPCMs. As reported by Warzoha et al. [26], adding different fillers in paraffin-based PCMs leads to increasing or decreasing phase change enthalpy based on its impact on paraffin crystallinity. On the one hand, the phase change enthalpy of paraffin increased significantly by adding graphite due to an increase in NEPCM crystallinity; on the other hand, its value decreased with the addition of TiO₂ nanoparticles as a result of reduced NEPCM crystallinity. Table 1 reports the values of the enthalpy and thermal conductivity of all samples. The data indicated that a large amount of graphite caused a reduction in melt enthalpy due to reducing Eicosane mass and declining NEPCM crystallinity [26]. However, it predicts better molecular heat transfer due to the presence of nanoparticles in NEPCM samples.

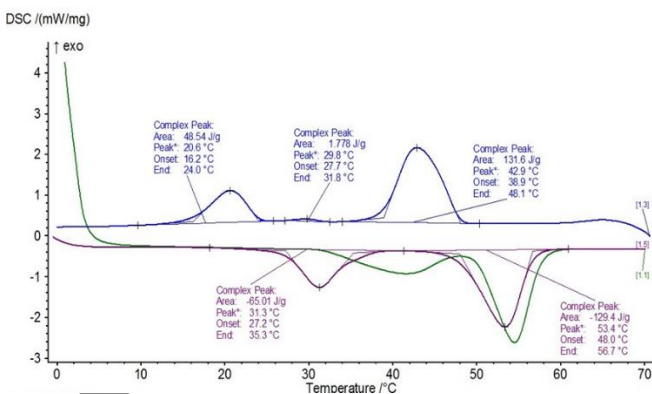


Figure 3. DSC graph of pure Eicosane.

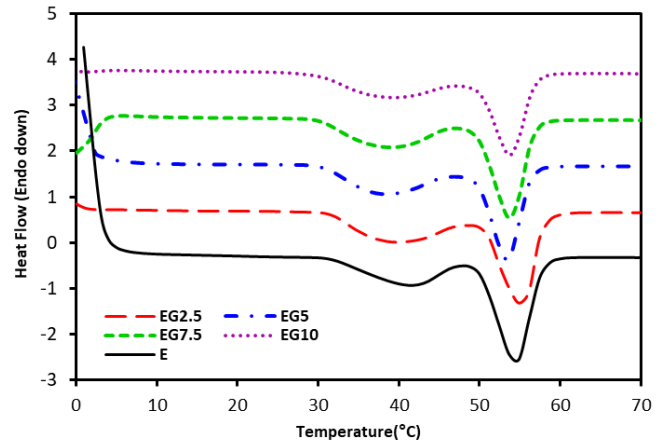


Figure 4. DSC graph of Eicosane-based samples; pristine PCM and NEPCMs at different mass fraction of graphite nanoparticles.

Table 2. Enthalpy, thermal conductivity and density of pure Eicosane PCM and NEPCM samples.

Sample	Enthalpy (J/g)	Thermal Conductivity (W/m K)	Density (g/cm ³)
E	194.4	0.30	0.9207
EG2.5	179.2	0.51	0.9703
EG5	168.3	0.48	0.9708
EG7.5	148.7	0.40	0.9908
EG10	140.8	4.7	1.0388

3.3. Specific heat capacity of NPCMs

A proper phase change material should have a high specific heat capacity to be able to store more quantity of heat per unit volume [22]. The specific heat capacity of nanocomposites depends on the type, size, shape, concentration, state of dispersion and distribution, and crystalline structure of the nanoparticles [27].

Figure 5 shows the specific heat capacity profile of pure Eicosane and its nano-enhanced forms determined by DSC. The specific heat capacity of the NEPCM composite depends on the specific heat capacity of the nanoparticles and pure Eicosane, which causes the overall thermal absorbance in the composite. Since the nano-graphite has a lower heat capacity than Eicosane, adding it to the Eicosane matrix decreases C_p of the nanocomposite [28]. Therefore, as the mass portion of nanoparticles increases or the mass portion of paraffin decreases, the specific heat capacity of composites is reduced due to the much lower specific heat of the nanomaterials than the pure paraffin.

Also, Figure 5 shows that increasing graphite concentration to 2.5% leads to the reduction of C_p of the nanocomposite. The amount of C_p reduction is strongly influenced by the surface energy of the nanoparticles [24]. Adding 5 and 7.5 wt% graphite did not reduce heat capacity compared to the 2.5wt% graphite. Despite the independence of heat capacity with respect to network formation in the system [29], graphite aggregation at higher concentrations acts as a heterogeneous nucleation agent, which is advantageous for the crystallization growth of Eicosane [30]. Hence, the competition between this phenomenon and heat capacity reduction due to the presence of nanoparticles causes the specific heat capacity to remain almost constant. For the sample EG10, heat capacity reduction may overcome the

influence of crystallization growth, leading to the reduction of C_p of the sample.

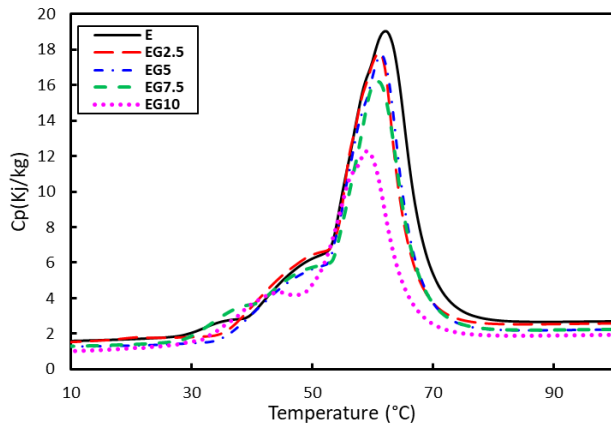


Figure 5. Specific heat capacity of pure Eicosane PCM and NEPCM samples containing graphite nanoparticles.

Thermal diffusivity in a material introduces heat conductivity ability compared to thermal energy storage. Therefore, a quantity, so-called "thermal diffusion coefficient", which is the ratio of the heat transferred by conduction mechanism to the heat stored by the unit volume of the substance, is represented by the symbol α correlated in Eq. 2.

$$\alpha = k / \rho C_p \quad (2)$$

According to the above equation, the thermal diffusion coefficient is inversely related to heat capacity and directly related to thermal conductivity. The high thermal conductivity intensified heat transmission through the material, representing high thermal diffusivity.

The values of the thermal diffusivity of the samples are reported in Figure 6. Adding graphite up to 7.5wt% increased the thermal diffusivity by 1.8 times, while 10 wt% graphite enhanced that by approximately 27 times compared to pure Eicosane. This significant increase in thermal diffusivity of NEPCM at this loading level may be due to the formation of a percolation network [24]. This also can be explained by trapping graphite in Eicosane's crystalline structures, enhancing the effective contact surface between the nanoparticles and the PCM [20]. Thus, increasing the collisions between Eicosane and graphite increases the thermal diffusivity and conductivity of nanocomposites.

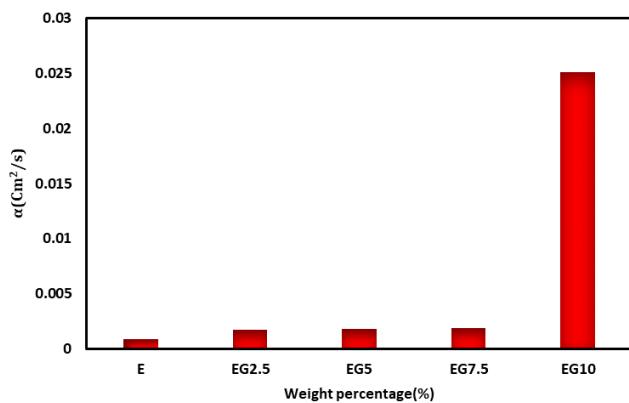


Figure 6. Thermal diffusivity for pure Eicosane PCM and NEPCM samples with different mass fractions of graphite nanoparticles at 25°C.

3.4. Thermal conductivity measurement

Figure 7 illustrates changes in the latent heat and thermal conductivity of composites as a function of nano-graphite percentage. Dispersion of nano-graphite layers in different directions in the Eicosane-based matrix creates a framework that promotes heat transfer in NEPCMs. This enhancement is related to the higher thermal conductivity of graphite (25-470 W/mK) compared to Eicosane. Additionally, in the presence of nanoparticles, a layer of melt Eicosane may form on the graphite surface, increasing interfacial heat conduction and decreasing interfacial thermal resistance [31]. Although NEPCM's thermal conductivity increases above the percolation threshold of graphite particles [21], its values are strongly related to the dispersion state of the suspension. If the particles stick to each other and form clusters resulting in precipitation, this will drastically reduce the thermal conductivity of the composite. Thus, thermal conductivity enhancement in this work confirms the proper dispersion of nanofillers without significant precipitation. Numerically, the thermal conductivity was increased by 1550% at a graphite loading of 10 wt%. The improvement of the thermal diffusivity is greater (Figure 6) than that in thermal conductivity, indicating the major impact of C_p on the thermal properties of PCMs. According to Figure 7, continuous increment in the thermal conductivity of NEPCM samples is accompanied by a gradual reeducation in the latent heat of the composite by enhancing graphite nanoparticle content. Accordingly, the appropriate nanoparticle content must be carefully selected based on the desired PCM application.

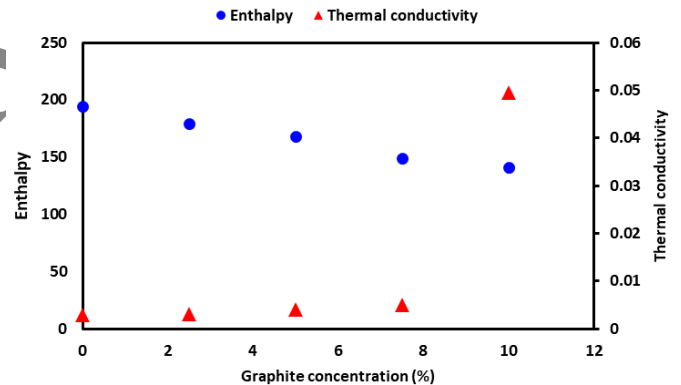


Figure 7. The enthalpy and thermal conductivity of the nanocomposite as a function of nano-graphite.

4. Conclusion

The high thermal energy storage capacity of PCMs makes them applicable for different purposes, especially thermal management applications. However, their low thermal conductivity reduces the heating rate, resulting in lower system efficiency. In this study, we have shown that incorporating graphite nanoparticles in Eicosane-based PCMs can improve the thermal properties of NEPCMs, primarily their thermal conductivity. An ultrasonic bath-assisted homogenization procedure followed by solidification was harnessed to prepare homogenous PCM composites. The results exhibited that good dispersion of lamellar layers of graphite nanoparticles within the Eicosane matrix was responsible for the improved thermal properties. Although small aggregates at high nanoparticle loadings were observed, they had no impact on the heat transfer rate. However, aggregation of big nanoparticles affects the specific heat capacity due to its effect on the crystallization

growth of Eicosane. Moreover, as the content of graphite nanoparticles increased, the thermal diffusivity was more affected than thermal conductivity. With the addition of graphite nanoparticles up to the optimal amount (10 wt%), the thermal conductivity and diffusivity of the composite PCM reached their maximum values of 4.7 W/m K and 0.025 cm²/s, approximately 15.5 and 27 times those of the pure n-Eicosane, respectively. Due to the significant improvement in thermal conductivity of the PCM containing the optimal amount of nano-graphite, such composite PCMs could be promising in various applications, mainly aero-space systems and thermal management electronics.

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Nomenclature

Ag	Silver
C_p	Specific heat capacity
DSC	Differential scanning calorimetry
GNP	Graphene nanoplatelet
k	Thermal conductivity
NEPCM	Nano-enhanced phase change material
PCM	Phase change material
PVP	Polyvinylpyrrolidone
TC	Thermal conductivity evaluation
Greek Symbol	
α	Thermal diffusivity

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