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Advancements in Composite Phase Change Materials for Enhanced Thermal Energy Storage Applications

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Abstract

Balancing energy supply and demand remains a significant challenge in the current global landscape, underscoring the growing necessity for effective energy storage solutions. Phase Change Materials (PCMs), such as Paraffin Wax (PW), offer latent heat storage, a key component in advancing renewable energy sources. PCMs are increasingly recognized for their crucial role in enhancing renewable energy systems. Paraffin, a widely studied PCM, offers several desirable thermal properties, including high latent heat capacity, low supercooling, minimal vapor pressure, chemical stability, and an inherent tendency for self-nucleation. Despite these advantages, paraffins low thermal conductivity limits its thermal performance and broader application potential. This study aims to address the limitations of paraffin wax by incorporating varying concentrations of Expanded Graphite (EG) specifically, 3 wt%, 5 wt%, 7 wt%, and 9 wt%. The objective is to determine the effect of EG addition on the thermal characteristics of the resulting composite phase change material (CPCM). SEM, XRD, and FTIR were used to confirm the homogenous dispersion of EG within the PCM matrix. Differential Scanning Calorimetry (DSC) study revealed that adding EG raises both the melting temperature and the latent heat of the CPCM. Furthermore, Thermogravimetric Analysis (TGA) showed an increase in degradation temperature with the addition of EG, indicating increased thermal stability of the composite material.

Major Findings: *The inclusion of 7% optimized Expanded Graphite (EG) in paraffin wax leads to a remarkable 153% improvement in its thermal conductivity. Despite the addition of EG, the composite retains a substantial latent heat capacity, ensuring its effectiveness as a thermal energy storage material.*

Keywords: Additives, Composite Phase Change Materials, Expanded Graphite, Latent Heat Storage, Paraffin-based PCMs

1.0 Introduction

Rising global energy consumption and declining non-renewable energy sources provide a critical issue to securing a stable global energy supply[1]. Addressing this gap will require innovative solutions, with energy storage systems emerging as a feasible approach. Thermal Energy Storage Systems (TESS) that use PCMs are particularly appealing because of their ability to harness thermal energy *via* latent and sensible heat. Numerous types of PCM have been extensively researched, including organic and inorganic classes[2]. Paraffin wax is one example of an organic PCM. It has received a lot of attention because of its high energy density, low vapor pressure, chemical stability, thermal stability, low supercooling, non-toxicity, widespread availability, and inexpensive cost. However, the applicability of paraffin wax is limited because of its low thermal conductivity during phase transitions. Researchers have devised a variety of strategies to overcome this limitation, including the introduction of porous materials, high-conductivity particles (both metallic and non-metallic), and the incorporation of

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highly conductive metallic fins into PCMs. Currently, there is an increasing focus on improving PCM characteristics by the dispersion of highly conductive micro- and nanoparticles[3-13].

The distinctive structure of Expanded Graphite (EG) sets it apart from the other additions, excellent adsorption properties, high thermal conductivity (200 W/(m·K)), and low density (269 kg/m^3)[14]. Studies have shown substantial enhancements in thermal conductivity with the addition of EG to paraffin wax. For example, using the hot disc method, research has shown that adding EG can improve the thermal conductivity of paraffin wax increases from 0.1716 W/(m-K) to 0.472 W/(m-K)[15]. Further studies have explored the optimal mass fraction of EG to maximize thermal conductivity while maintaining other desirable PCM properties[16]. However, it's noted that exceeding the optimal EG mass percentage can lead to a reduction in thermal conductivity, attributed to the formation of voids and gases within the CPCM structure[17].

The goal of this research is to further this field by developing a novel CPCM using paraffin wax and EG at varying weight fractions (3%, 5%, 7%, and 9%). The study will look into the effects of EG addition on important thermophysical parameters such phase transition temperatures, latent heat of fusion, and thermal conductivity. The chemical compatibility and stability of composite PCM materials will be assessed using analytical techniques such as XRD and FTIR. While existing research provides useful insights, there is still a scarcity of thorough experimental data on the thermal conductivities and latent LHTES properties of these produced composite PCMs. The primary end application of this study is thermal energy storage systems; this application requires materials with high thermal conductivity and cost-effectiveness. In this context, the findings of our study contribute to the development of thermal conductive composites.

2.0 Materials and Methods

2.1 Materials

In this study, commercial-grade paraffin wax serves as the base PCM, selected for its economic viability compared to other technical-grade paraffin. Commercial grade and technical grade paraffin wax, however, are found to have the same thermo physical characteristics. This PCM melts at a temperature of about 66° C. Table 1 presents the thermophysical properties of pure paraffin wax PCM and EG microparticles.

Table 1. Thermophysical Properties of Pure Paraffin Wax PCM and EG Microparticles

Properties	Materials	
	Paraffin wax	EG
Melting point (°C)	66.84 (Manufacturer)	3600
Density (Kg/m^3)	900	269
Latent heat (KJ/Kg)	204	-
Thermal conductivity (W/mk)	0.228	200
Specific heat(KJ/kg)	2.14	0.710

2.2 Synthesis of Composite PCM (CPCM)

Different ratios of EG particles are mixed with pure paraffin wax to form the EG or paraffin CPCM. This process involves heating and stirring the mixture of paraffin wax and EG at the melting temperature for 1 hour. The whole sample preparation step is depicted in Figure 1. The paraffin wax is initially melted at 68° C using a hot plate with a magnetic stirrer. To ensure the uniformity and prevent clumps formation, the CPCM is then subjected to

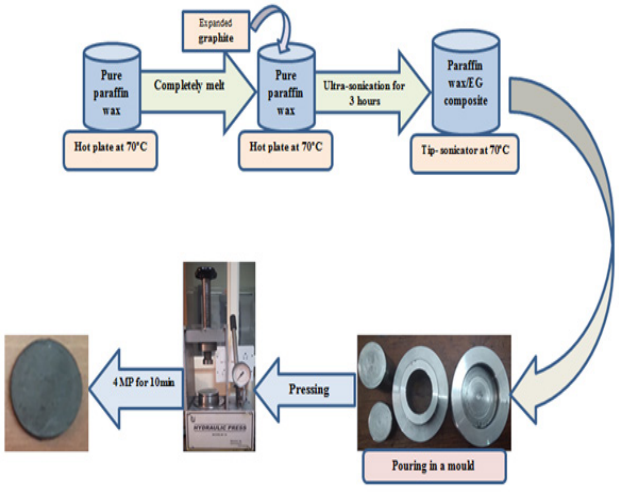


Figure 1. Synthesis step for preparing paraffin and EG composite.

tip sonication with pulse rate of 5 sec, 50Hz frequency and at a temperature of 80° C for two hours. PEG_0, PEG_3, PEG_5, PEG_7 and PEG_9 are the component percentage of paraffin wax and EG. PEG_0 contains pure paraffin wax; PEG_3, PEG_5, PEG_7 and PEG_9 with respect to 3 wt%, 5 wt%, 7 wt% and 9wt% of EG respectively. To measure thermal conductivity and perform FTIR analysis, the powder samples are compressed in a die to prepare the specimens. However special dies has been design to prepare the 23 mm diameter. Measured quality of prepared powder samples are placed in the dies and make pellet for C-therm conductivity analysis.

2.3 Characterization Techniques

Characterizing CPCMs involves employing various sophisticated techniques to explore their structural, chemical, and thermal properties comprehensively. These methods provide crucial insights into phase change behaviors, latent heat, thermal conductivity, and thermal stability. Here's a breakdown of the characterization techniques used in the study:

2.3.1 Scanning Electron Microscopy (SEM)

The microstructure of CPCMs was examined using a JEOL JCM 6000 Plus SEM instrument. This technique provides detailed surface and structural analysis, offering magnification ranges from 5x to 60,000x with a resolution as fine as 10 nm[18].

2.3.2 X-ray Diffraction (XRD)

XRD analysis was conducted using a Rigaku-Smart Lab X-ray diffractometer with CuKα (1.541 Å) radiation. This method is used to determine material properties such as crystal structure, crystallite size, and strain, typically over a scan range from 0° to 80°[19].

2.3.3 Fourier Transform Infrared Spectroscopy (FTIR)

A Bruker-alpha-p FT-IR spectrometer was used to perform FTIR analysis, which helps identify functional groups and assess chemical interactions within the composite PCM. The spectral resolution of 4 cm^{-1} allows precise characterization in the 4000-400 cm^{-1} spectral range[20].

2.3.4 Thermal Gravimetric Analysis (TGA)

Thermal stability of the CPCMs was evaluated using a TGA Q500 instrument under a nitrogen atmosphere. The analysis involved monitoring weight changes as a function of temperature at a constant heating rate of 10° C/min, covering a temperature range from 30° C to 700° C.

2.3.5 Differential Scanning Calorimetry (DSC)

DSC analysis, conducted in a nitrogen environment using NETZSCH equipment, was utilized to investigate the thermal properties of the samples. Parameters such as latent heat, specific heat capacity, and melting temperature were measured by heating the samples at a rate of 5°C/min across a temperature range of 30° C to 300° C.

2.3.6 Thermal Conductivity Measurement

Thermal conductivity of the CPCMs is measured using a C-Therm thermal conductivity analyzer, providing crucial data on heat transfer properties. These characterization methods collectively provide a detailed understanding of the CPCM's performance and suitability for TES applications. Each technique contributes specific insights crucial for optimizing the design and application of CPCMs in energy storage systems.

3.0 Result and Discussion

3.1 SEM Analysis

Figure 2(a) and 2(b) show the SEM images of paraffin wax or expanded graphite with of 3% and 9% weight fraction under 1000X magnification. In this figure, the black area corresponds to the saturated Expanded Graphite (EG), while the white area represents the paraffin wax. The observations indicate that EG is surrounded by paraffin wax. The SEM images of the PCMs reveal that paraffin wax is absorbed into the pores of EG and encased by graphite flakes, a process facilitated by capillary action and surface tension[21]. This phenomenon also understands as the wettability of paraffin wax on the surface of EG is good hence it is effortless infiltrate paraffin wax into the pore structure of expanded graphite[22]. Although graphite was traditionally thought to be naturally hydrophilic, the phase boundary between paraffin and expanded graphite merged compactly, indicating the strong wettability[23].

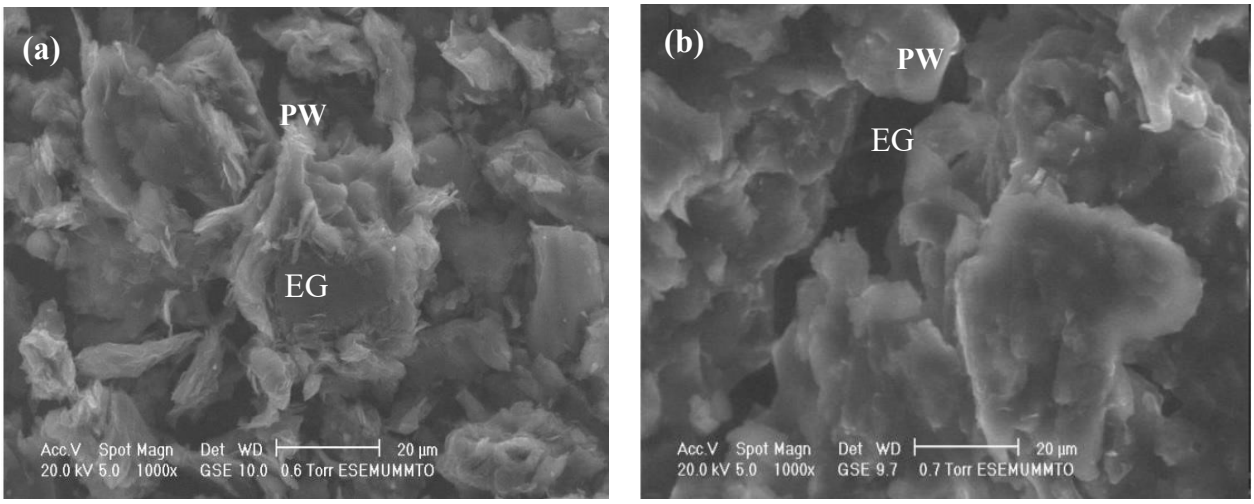


Figure 2(a-b). SEM images of the prepared composite phase change materials.

3.2 FT-IR Analysis

Figure 3 shows the FT-IR spectra of prepared paraffin or EG composite PCMs sample. The main characteristics adsorption bands are observed in between 1460-1600 cm^{-1} and 3200-3600 cm^{-1} corresponding to the stretching vibrations of C-O, physically adsorbed water, C==O, $-CH_2$,$-CH_3$ and –OH groups for the entire samples[24]. The hydroxyl (OH) groups are generally present in the regions 3600 to 3500 cm^{-1}, the amino (NH) groups at 3400 to 3300 cm^{-1}, and C-H groups are present at 2850 to 3350 cm^{-1}, it proves there are no hydroxyl or amino groups present in PW. The peaks found at 1400.54 and 1472.45 cm^{-1} indicate the presence of hydrate elements (H_2O)[25]. The sharp peaks found in the region of 1300 to 1600 cm^{-1} indicate the presence of hydrate (H_2O) elements. As paraffin wax belongs to an alkane family, it consists only of a single bond with the absence of double and triple bonds. The peak observed between 2850 cm^{-1} and 3300 cm^{-1} indicates the presence of C-H groups. The graph also shows distinct C-H stretching vibrations at 1462.70 cm^{-1}, 2848.98 cm^{-1}, and 2917.46 cm^{-1}, along with C-H bands at 2958.23 cm^{-1} and 3130.72 cm^{-1}. Additionally, the peaks at 728.92 cm^{-1} and 719.60 cm^{-1} confirm the presence of aliphatic organic carbon compounds. Notably, the results reveal that all characteristic peaks detected in the pure PCM samples are also evident in the prepared composites. This indicates that the EG particles are physically integrated into the PCM matrix without participating in any chemical reactions. As a result, the PCM samples maintain excellent chemical stability, even with the inclusion of EG.

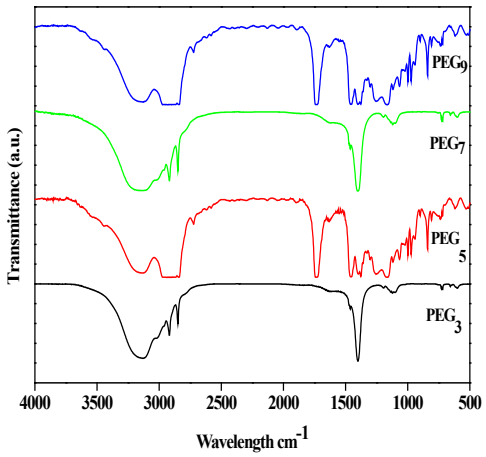


Figure 3. FTIR plots of composite PCM samples.

3.3 XRD Analysis

To ascertain the chemical reaction between the PCM and EG particle in the region of 0-65°, XRD examination is carried out on the generated composite PCM samples. Figure 4 show that all of the manufactured PCM composites exhibit prominent peaks at approximately 21.84 degrees. Another peak at 24.21° also noticed in pure paraffin samples and this peak is also observed in all the composite samples. Since there is no significant peak observed after 65, the 2θ value has been taken up to 60° C.

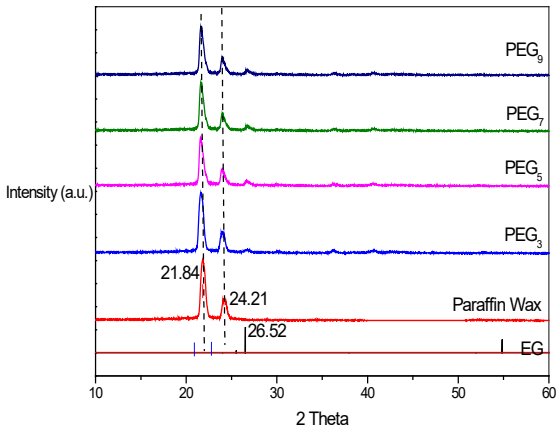


Figure 4. XRD pattern of pure paraffin and micro EG composite.

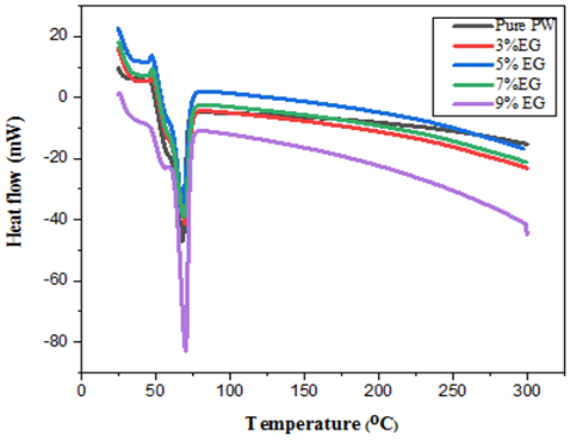


Figure 5. Typical DSC curve of the PCMs.

The standard peak for expanded graphite is also shown in Figure 4 and drawn according to JCPDS No. 41e1487. The peak 26.52° corresponds to EG be observing as a small peak in the entire prepared PCM/EG composite sample, which indicates the formation of homogeneous.

When the percentage of EG in CPCM increases, the sharp peak associated with paraffin wax shifts to the lower angle. The crystallographic lattice plane distance fluctuation is the cause of this effect[26]. Full Width with Half Maximum (FWHM) values varies from 0.57989 to 0.288 radians; D-spacing varies from 4.09054 to 2.2147cm. The crystallite size is increases with increase the percentage of EG, it varies from 73.34% to 182.21%. It is important to note that there are no additional peaks in the PCM/EG composite. In the prepared composite PCM, the doped particles are positioned as interstitial atoms within the PCM particles. It has been established that there is no chemical reaction between the components as a result of this arrangement, which produces a homogenous composite.

3.4 DSC Analysis

Figure 5 displays the DSC curves for pure PCM and the PCM/EG compositions at 3%, 5%, 7%, and 9%. All curves contain two phase transitions, one roughly at around 46° C and the other at around 68° C. In DSC results, two distinct endothermic peaks were identified during the heating process.

The solid-liquid phase transition, or heterogeneously nucleated rotator-liquid transition, is shown by the peak that is more intense at higher temperatures. Conversely, the homogenous solid-solid phase transition is associated with the peak at a lower temperature that has a lesser intensity[27]. The total thermal capacity of the PCMs is considerably increased by both of these phase shifts.

The phase transition of each sample exhibits minor variations with the addition of different EG loadings. Meanwhile, the latent heat is determined by integrating the endothermic peak observed in the DSC curve.

Table 2. Melting temperature and latent heat capacity of pure paraffin and composite PCMs

Samples	Increase in Melting Temperature (T_m) (%)	Increase in Latent heat Capacity (ΔH) (%)
PEG_0	----	---
PEG_3	2.77	25.62
PEG_5	3.21	52.14
PEG_7	3.41	103.5
PEG_9	3.86	170.89

The addition of EG content enhances the latent heat capacity of each sample. Table 2 presents the calculated latent heat and phase transition temperatures of the prepared composite PCMs. The phase transition temperature of CPCM is enhanced by 2.77%, 3.21%, 3.41%, and 3.89% for 3%, 5%, 7%, and 9% EG respectively, compared to pure paraffin wax. The melting point temperature has shown a slight increase, attributed to the higher porosity of the composite PCM samples, which results in increased surface tension and greater capillary force[28].

Table 3. Experimental and theoretical latent heat values of prepared composites

Samples	Experimental Latent Heat Values (ΔH) kJ/kg	Theoretical Latent Heat Values (ΔH) kJ/kg
PEG_0	225.6	225.6
PEG_3	283.4	274.8
PEG_5	343.35	326.05
PEG_7	459.13	426.9
PEG_9	611.15	556.14

Meanwhile, the latent heat capacity of the CPCM has been found to be enhanced by 25.62%, 52.14%, 103.5%, and 170.89% for 3%, 5%, 7%, and 9% EG content, respectively, compared to pure paraffin wax. Table 3 presents the experimental and theoretical latent heat values of prepared composites. Based on the obtained analysis results, it can be concluded that the enhancement of latent heat of the PCMs would be addresses by the attractive interactions between paraffin molecules and the EG surface. On the other hand, the crystallinity of paraffin on the composite PCMs must be considered on the account of the interactions between paraffin and EG. As known, the crystallinity of organic PCMs has significant influence on the latent heat of the composite. The increased crystallinity observed in the CPCMs is attributed to the role of EG as a heterogeneous nucleation agent during the solidification of paraffin. The presence of EG promotes nucleation, accelerating the development of a more ordered crystalline structure within the paraffin matrix. This effect improves the latent heat storage capacity of the CPCMs. In this study, the addition of EG at low concentrations (≤10 wt%) in paraffin-based CPCMs aims to enhance their thermal properties without compromising latent heat storage capacity per unit mass.

3.5 Thermal Gravimetric Analysis

Assessing the long-term reliability and durability of paraffin or Expanded Graphite (EG) composites is pivotal when considering their potential applications in heat storage systems. This evaluation is essential to verify the sustained performance of these composites over prolonged operational durations[29]. Figure 6 displays the thermal stability profile of the prepared composites as measured by using a Thermal Gravimetric Analysis (TGA). One

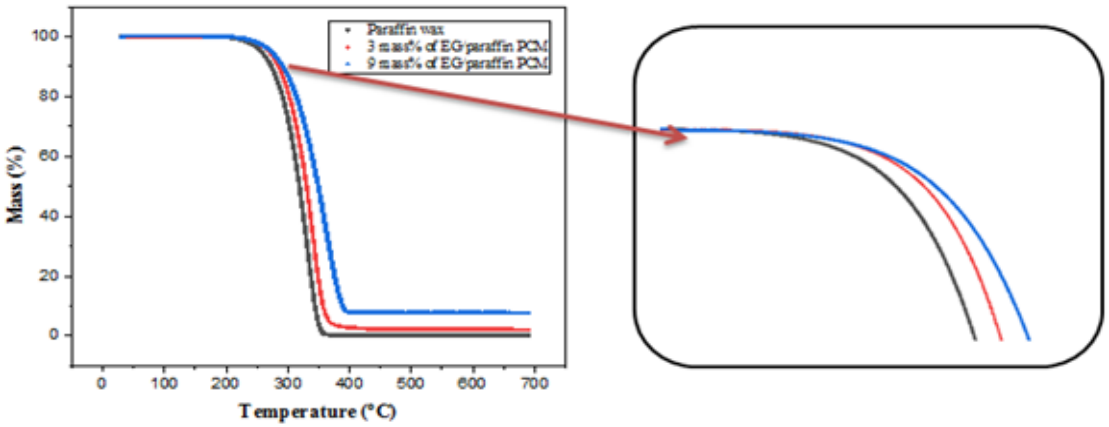


Figure 6. TGA results of pure PCM and composite PCM samples.

notable observation is that the weight loss curves exhibit uniform degradation behavior, with each composite showing a consistent one-step degradation process. The paraffin or EG composites commence evaporation at around 200° C and undergo full decomposition at approximately around 360° C.

The thermal degradation temperatures of pure paraffin wax and paraffin wax with 3% EG and 9% EG are found to be at 202° C, 224.07° C and 238.76° C, respectively. All samples including paraffin wax completely degraded around 350 to 400° C. The onset degradation temperature of the pure paraffin, paraffin with 3% EG and 9% EG samples are found at 359° C, 374° C and 398.38° C respectively. The result shows degradation temperatures of CPCMs higher than pure paraffin wax, which is more around 4.2% and 10.9% for 3wt% and 9wt% of EG, respectively. It indicates that an EG based PCM composition can be safely operated and can maintain the stability at temperatures up to 250° C. It concludes that the developed composite PCMs can serve as an effective material in thermal storage mediums for medium or low-temperature applications. The results of this study show that, within their operating temperature range, the CPCMs show outstanding thermal stability.

3.6 Thermal Conductivity Measurement

The thermal conductivity of the prepared composite samples was assessed using a C-Therm thermal conductivity analyzer, as shown in Figure 7. The baseline thermal conductivity of pure paraffin wax is measured at 0.22 W/mK. By incorporating Expanded Graphite (EG) into the paraffin wax at concentrations of 3%, 5%, 7%, and 9%, the thermal conductivity improves significantly, with increases of 136%, 145%, 153%, and 159%, respectively.

Table 4. Increase in thermal conductivity (W/m-K) values of prepared composites

Samples	Increase in Thermal Conductivity (W/m-K)	Increase in Thermal Conductivity (%)
PEG_0	0.22	-
PEG_3	0.521	136
PEG_5	0.54	145
PEG_7	0.552	153
PEG_9	0.571	159

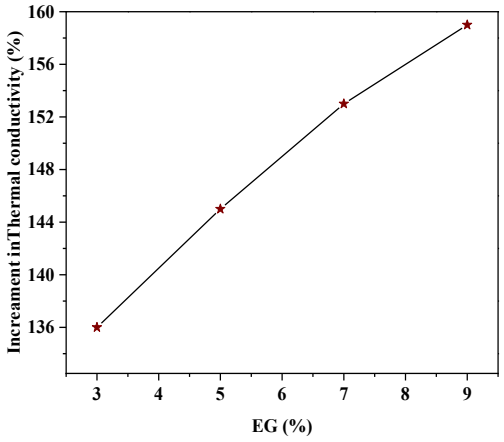


Figure 7. Thermal conductivity enhancement ratio of composite PCMs.

The percentage enhancements in thermal conductivity (k) of the Composite Phase Change Materials (CPCMs) were calculated using the following equation:

$$Thermalconductivity[K](\%)\frac{Kcpcm - Kpcm}{Kpcm} \quad (2)$$

According to the results, the thermal conductivity increases non-linearly with the mass percentage of EG addition. Hence the percentage of EG particle is selected from 0 to 10 wt. %. The increased heat conductivity of CPCM improves PCM heat transfer performance and speeds up the rates of melting and solidification as well as energy storage and release during phase transitions. EG has the advantage of low price, good adsorption performance, and stable chemical properties[30,31]. The rich pore structure and high surface area also allow EG to have a larger internal space, and a higher thermal conductivity[32-34].

4.0 Conclusion

Researchers utilized several analytical techniques to evaluate the Composite Phase Change Materials (CPCMs).

SEM imaging confirmed the good surface morphology and effective embedding of paraffin within the additive's pores. XRD analysis revealed no new crystallographic formations post-addition of the additive. FT-IR analysis affirmed that the original chemical nature of paraffin wax, composed solely of carbon and hydrogen, remained unaltered. DSC analysis demonstrated that the addition of nanoparticles enhanced latent heat and specific heat capacities. TGA analysis indicated that carbon additives significantly elevated the working temperature of paraffin wax, thereby enhancing its thermal stability. Incorporating high-conductivity particles enhanced the thermal conductivity of pure PCMs, improving heat transfer performance in latent heat thermal storage systems. Based on our experimental results and analysis, the optimum weight percentage was determined to be 7 wt. %. This value was identified as optimal because it provided the best balance thermal conductivity, mechanical properties and cost-effectiveness. Additionally, exceeding this weight percentage was observed to lead to agglomeration, degradation of structural properties, while lower percentages did not achieve the desired improvement in thermal conductivity.

In conclusion, the incorporation of a small mass fraction of EG particles considerably improves the thermal properties of paraffin wax. This enhancement indicates that EG or paraffin composite PCMs have strong potential for application in efficient thermal energy storage systems, promising increased thermal conductivity and stability that are critical for advanced energy solutions.

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