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Enhanced Thermal Performance of Polyethylene Glycol-Based Phase Change Materials Using ZnO

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Abstract

Polyethylene Glycol (PEG), an organic Phase Change Material (PCM) known for its high latent heat capacity and biocompatibility, suffers from poor thermal conductivity. This research explored the addition of ZnO (at 8 and 12 wt.%) as an enhancer and SDBS as a surfactant, utilizing a two-step homogenization process at 80°C for 4 h. X-ray Diffraction (XRD) analysis confirmed that no chemical interactions or new phases occurred. Differential Scanning Calorimetry (DSC) findings revealed an enhancement in PEG's latent heat of PEG from 214 J/g to 238.3 J/g (with 8 wt.% ZnO) and 257.7 J/g (with 12 wt.% ZnO). The thermal conductivity improved by 28.2% and 30.3%, respectively, while Thermogravimetric Analysis (TGA) demonstrated increased thermal stability. The addition of ZnO can improve the conductivity and thermal stability, while the role of SDBS is significant in increasing the latent heat, making this composite a potential candidate for Thermal Energy Storage system (TES) applications.

Introduction

Global warming due to climate change continues to increase with an increase in CO_2 emissions [1]. The increase in global energy consumption and CO_2 emissions is primarily attributable to industrial activities that utilize power on a large scale. However, during industrial processes, a significant portion of the energy input is discharged in various forms as waste heat into the surrounding environment, resulting in substantial energy waste [2].

Waste heat is categorized into three distinct temperature ranges: high-temperature (exceeding 400°C), medium-temperature (ranging from to 100-400°C), and low-temperature (below 100°C). To enhance energy efficiency and minimize environmental impacts, Waste Heat Recovery (WHR) systems have been widely adopted across various industrial sectors [3], [4]. The integration of WHR systems with Thermal Energy Storage (TES) technology is a highly promising strategy for industrial energy management. Thermal storage is pivotal for addressing the discrepancy between the availability and demand for waste heat [5]. Among the various thermal storage methods, latent heat storage utilizing Phase Change Materials (PCMs) is particularly effective. In comparison to sensible heat storage, latent heat storage provides a higher energy density and more stable thermal charge–discharge performance [6]. PCMs can absorb and store substantial amounts of thermal energy. During the endothermic phase transition, these materials undergo a change from solid to liquid, while maintaining an approximately constant temperature. Conversely, during the exothermic process, they release stored heat as they transition from the liquid to the solid state at a specific temperature [7]. PCMs are classified into three categories: inorganic, organic, and eutectic [8] . Paraffin is a widely utilized organic PCMs owing to its high heat storage capacity, minimal volume change, non-toxic, non-corrosive, chemical and thermal stability [9]. However, paraffin is derived from petroleum hydrocarbons; consequently, it is sourced from non-renewable energy sources and has a carbon footprint as well as significant environmental impacts [10], [11]. Polyethylene Glycol (PEG) is another organic PCMs that exhibits properties comparable to those of paraffin, including substantial heat storage capacity, minimal volume change, non-toxicity, non-corrosivity and chemical stability. The advantage of PEG over paraffin is its natural biodegradability, which renders it more environmentally favorable [12].

PEG, like many organic PCMs, exhibits low thermal conductivity, which results in a reduced capacity for heat transfer [13]. It has been observed that the thermal conductivity increases as the molar mass of PEG increases from 1000 to 6000, subsequently decreases as the molar mass continues to increase up to 20000. The maximum thermal conductivity recorded was 0.34 W/m·K for PEG 6000. Therefore, it is necessary to enhance the thermal conductivity by incorporating fillers with high thermal conductivity [14]. Such as metal, metal oxide or carbon-based particles [15]. Researchers have extensively explored methods to augment the thermal conductivity of PEG by incorporating fillers. The addition of Al_2O_3 at mass fractions ranging from 1 to 5 wt.% alongside SDBS surfactant resulted in an increase in thermal conductivity of up to 52.09%, although it led to a 9.4% reduction in latent heat. After 1500 melting/freezing cycles, with no significant changes in thermal properties [16]. The incorporation of SiO_2 and Al_2O_3 nanoparticles (20 wt.%) also yielded a thermal conductivity enhancement of up to 111%, while the latent heat storage capacity decreased by 6.7% and 6.2%, respectively [17]. Another study investigated the use of metal oxide nanoparticles (Al_2O_3, CuO, TiO_2, and ZnO) in PEG via an ultrasonic method, revealing a thermal conductivity increase of up to 1.34 times, but with a latent heat reduction ranging from 1.0% to 5.1% [18]. The application of carbon-based fillers, such as Expanded Graphite (EG) and Wall-Mounted Carbon Nanotubes (WMCNT), results in a significant improvement in thermal conductivity. The addition of 2 wt.% WMCNT increased thermal conductivity by up to 55.80%, while EG achieved 29.44%. However, this enhancement was accompanied by a reduction in latent heat of 17.69% and 39.95%, respectively [19]. A similar trend was observed with the addition of 2 wt.% Graphene

Nanoplatelets (GNP), which improved thermal conductivity by 146%, with a relatively smaller decrease in PEG latent heat of just 6.3% [20]. Several of the studies demonstrate an increase in thermal conductivity correlating with increasing mass concentration of fillers; however, this also impacts the reduction of latent heat. This phenomenon is attributed to the strong interaction between nanoparticles and PCM, influenced by van der Waals forces, particularly in the surface layer, which interferes with the PCM phase change process. Consequently, this reduces the latent heat that can be stored or released by the system [21]. Thus, although the thermal conductivity increased, the capacity of PCMs to store latent energy was diminished due to the influence of nanoparticle interactions and van der Waals forces. To address this issue, surfactants can be incorporated into PCMs, which reduces the surface tension between the nanoparticles and PCMs, thereby dispersing the PCMs and NPs, mitigating agglomeration, and enhancing their thermophysical properties [22].

Previous studies have shown that the addition of fillers can improve the thermal conductivity of PE. However, this improvement is frequently accompanied by a reduction in latent heat, potentially undermining PEG's role of PEG as an effective thermal energy storage medium. This study aims to enhance the thermal properties of PEG, such as latent heat, thermal stability, and thermal conductivity, by incorporating Zinc Oxide (ZnO) as an additive. To practically and tangibly assess the changes in thermal performance, ZnO mass fractions of 8% and 12% were selected as representative points. Sodium Dodecyl Benzene Sulfonate (SDBS) was used as a surfactant to improve the dispersion of ZnO within the PEG matrix. The homogenization process was conducted using a two-step method over a duration of 4 h at 80°C to ensure optimal dispersion. The thermophysical properties were evaluated using X-ray Diffraction (XRD), Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and Thermal Conductivity (TC) measurements to evaluate the influence of these additives on the performance of PEG as a thermal energy storage material.

Experimental Method

In this study, Polyethylene Glycol (PEG) with a molecular weight of 6000 and a melting temperature range of 58–63°C was obtained from Merck. Additionally, Zinc Oxide (ZnO) nanoparticles and Sodium Dodecyl Benzene Sulfonate (SDBS) surfactant were procured from Sigma-Aldrich, both of analytical grade, with purities exceeding 98%. PEG-ZnO composites with ZnO weight fractions of 8% and 12% and SDBS were synthesized using a two-step process: stirring and sonication [23]. The first step involved melting the PEG using a hot plate at 80°C. Once the PEG was completely melted, SDBS was added, and the mixture was stirred for 1 h at 1000 rpm. Subsequently, ZnO was added to the mixture, and stirring was continued for an additional 2 hours under the same conditions of 1000 rpm and 80°C. SDBS, with a ratio of 1:0.5 to the ZnO mass, was used to reduce agglomeration by lowering the surface tension between ZnO and the PCM, enhancing dispersion, and improving thermophysical properties [22]. However, an excessive surfactant ratio can reduce thermal conductivity and create air bubbles that are difficult to remove [24], [25]. Following this, sonication was performed at 80°C and 37 kHz for 1 h to further enhance the particle dispersion, as illustrated in Fig. 1. After achieving homogeneity, the PCM was molded. Analysis and characterization of the PCM were performed using various techniques. X-ray Diffraction (XRD) was employed to determine the crystal structure and phase, utilizing a SMARTLAB RIGAKU instrument with Cu Kα radiation (λ = 1.5418 Å). To explore the thermal properties of the material, Differential

Scanning Calorimetry (DSC) was conducted using a Polyma DSC 214 device, which facilitated the measurement of latent heat, melting point, and solidification point. The DSC tests were carried out at a heating and cooling rate of 10°C/min, spanning a temperature range from 25°C to 100°C. The thermal stability of the PCM was assessed through Thermogravimetric Analysis (TGA) with a PerkinElmer TGA 4000 instrument, operating at a heating rate of 10°C/min over a temperature range of 30°C to 600°C. Additionally, the Thermal Conductivity (TC) was evaluated using the hot-wire method with a QTM-500 instrument. To ensure that the results were reliable, the measurements were performed five times. The deviations were within ±5% of the thermal conductivity of the reference plate.

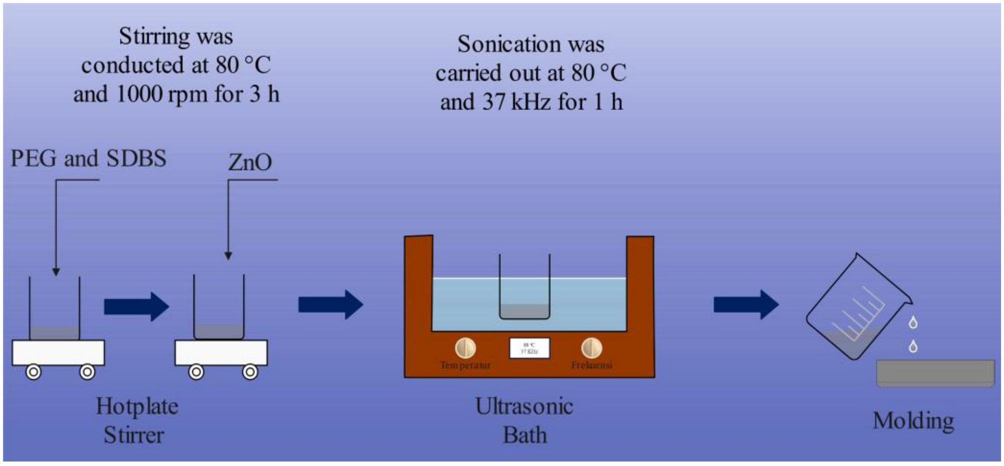


Figure 1. The synthesis process of PEG6000-ZnO composites.

Results and Discussion

X-Ray Diffraction

X-ray diffraction was employed to analyze the structure and phase purity of ZnO, PEG6000, as well as to confirm the structural stability of ZnO within the PEG6000 matrix. The relevant XRD patterns in the 10-90 Degree range of the synthesized samples are presented in Fig. 2. The diffraction ZnO peaks of (100), (002), (101), (102), (110), (103), (200), (112), (201), (004), (202, (104), (203) crystal planes were observed at 2theta 31.77, 34.42, 36.25, 47.54, 56.59, 62.86, 66.37, 67.95, 69.09, 72.47, 76.94, 81.39, and 89.61, respectively, which correspond to COD 2300450 data. This is consistent with the research [26][27][28].

The characteristic peaks of PEG6000 observed at 2θ angles of 19.18° and 23.24° indicate the presence of crystalline peaks within the polymetric. All the peaks distinctly exhibit hexagonal ZnO. The pronounced diffraction peaks of both composite materials underscore the highly crystalline nature of the samples. No additional impurity phases were detected in the composite within the detection limit of the instrument. A slight alteration in the XRD pattern of ZnO was observed at low PEG6000 concentrations (12 wt.% ZnO). The ZnO peak in the XRD pattern of the PCMs composite appeared with low intensity. These findings confirm that the sample comprises crystalline ZnO and PEG6000, consistent with the published literature. No peaks other than those of ZnO and PEG6000 were observed in the XRD patterns of the PCMs composites. Previous research has reported similar findings [18]. Indicating that the interaction between a PCM and nanoparticles is predominantly physical rather than chemical. Furthermore, the absence of new peaks or compounds in the XRD pattern corroborated the chemical compatibility of the nanoparticles with the PCM matrix, thereby enhancing the material properties without inducing undesirable chemical reactions [29]. Thermal cycling tests have been conducted in previous studies to assess the stability of materials against repeated heating and cooling processes, which could potentially impact the stability of their crystal structures in thermal energy storage systems [30]. These tests were conducted up to 100 cycles, and the results demonstrated that in the paraffin/Exfoliated Graphite (EG) composite with 10 wt.% EG, no chemical reaction occurred either before or after 100 thermal cycles, thereby confirming the chemical stability of the composite [31].

Furthermore, the X-ray diffraction patterns of the prepared ZnO nanoparticles were analyzed. The Rietveld refinement results are shown in Fig. 3 and Williamson-Hall analysis in Fig. 4. The parameters are listed in Table 1. Subsequently, the crystallite size was determined using the following Scherrer and Williamson-Hall equations: (1 and 2) [32].

$$D = \frac{K\lambda}{\beta \cos\theta} \tag{1}$$

$$\beta_{HKL} \cos\theta = \frac{K\lambda}{D} + 4\varepsilon \sin\theta \tag{2}$$

In this context, β_{HKL} represents the full width at half maximum (FWHM), λ denotes the wavelength, D indicates the crystallite size, θ is the Bragg angle, and k is the Scherrer constant. Utilizing the Williamson-Hall (WH) plot technique allows for the determination of both the average crystallite size and the microstrain (ε), which signifies the lattice distortion within the particle [32], [33].

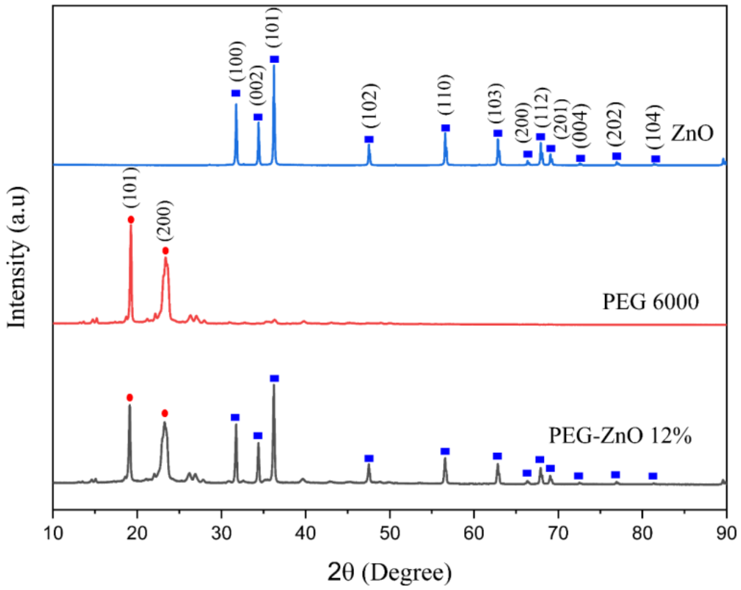


Figure 2. XRD pattern of ZnO, PEG6000 and PEG-ZnO 12%.

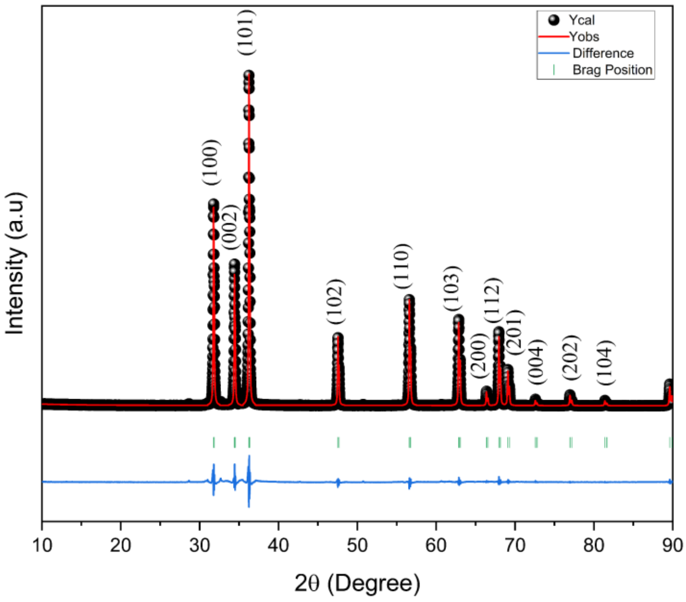


Figure 3. Rietveld refinement analysis of ZnO material based on XRD pattern.

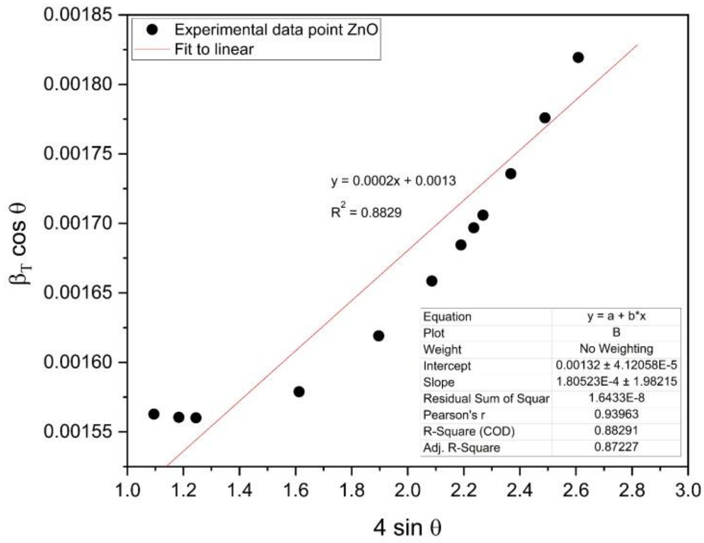


Figure 4. Williamson-Hall plot of ZnO material.

Differential Scanning Calorimetry (DSC)

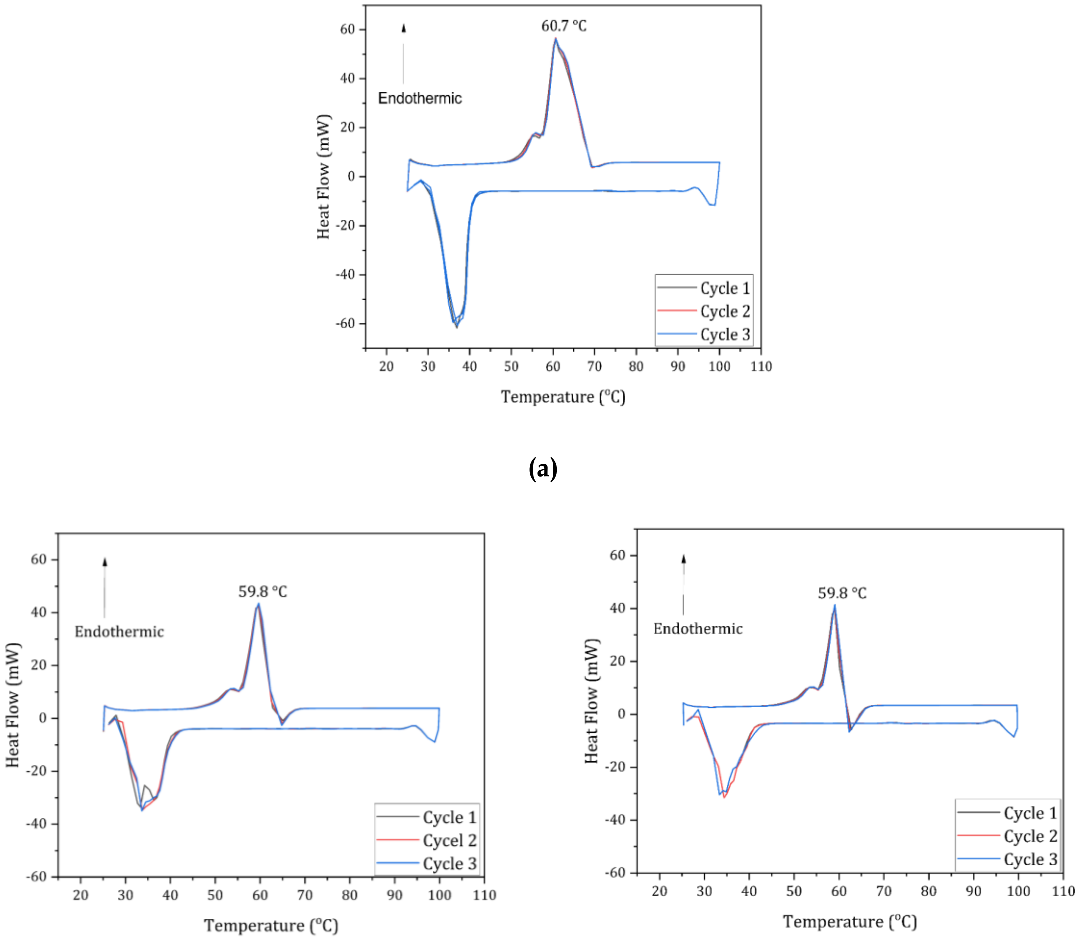
Figure 5(a-c) illustrates the DSC analysis, a common method for determining the thermal characteristics of PCMs, such as latent heat, melting point, and solidification point. When PEG absorbs thermal energy, it raises the energy levels of its atoms or molecules, enhancing their vibrational states. At the melting temperature, the atomic bonds become weaker, leading to a transition from solid to liquid (endothermic). As the material releases energy to its surroundings, the molecules lose energy and reorganize into a solid phase (exothermic) [34]. Figure 5(a) demonstrate that during the endothermic cycling of pristine PEG6000, a peak was observed at 60.7°C, which was considered the primary peak representing the solid-liquid phase change with a latent heat of 214 J/g. Figures 5(b) and 5(c) demonstrate that with the addition of 8 and 12 wt.% ZnO, the latent heat increased to 238.3 and 257.7 J/g, respectively. Compared to previous studies utilizing ZnO particles in PEG without SBBS, a reduction in latent heat of 3.13% was observed at a mass ratio of 2 wt.% [18]. Similar trends were observed with the incorporation of SiO_2 and Al_2O_3 particles in PEG without SBBS, demonstrating a decrease in the latent heat with increasing particle mass by 6.73% and 6.22%, respectively, at a mass ratio of 20 wt.% [17]. In this study, the increase in latent heat is consistent with the findings of previous studies, where the increase in latent heat was also caused by a more uniform particle distribution owing to the use of SDBS. As a result, the ZnO are distributed more evenly and exhibit a high surface area, thereby improving their interaction efficiency with the surrounding material [22]. This uniform distribution arises because SDBS changes the surface properties of the particles by using electrostatic forces to create repulsive forces between the particles. In addition, SDBS forms a physical barrier between the particles, thereby reducing the formation of agglomerations, usually caused by van der Waals forces [35][36]. Consequently, a greater amount of ZnO with an increased surface area was available for interaction. Additionally, a higher concentration of ZnO provides more particles that enhance the heat transfer, thereby accelerating the melting process. Moreover, it optimizes energy absorption and enhances latent heat capacity, particularly at higher nanoparticle mass ratios [37]. In accordance with this, the findings of this study demonstrate significant potential for application within TES systems. Concerning TES applications, several studies have indicated that latent heat losses are considered acceptable if they fall within the range of 10-20% [38].

Table 1. Data on lattice parameters obtained from Rietveld refinement analysis and the crystallite size of ZnO.

No	Parameter	ZnO
1	Space Group	P63mc
2	a (Å)	3.23
3	b (Å)	3.23
4	c (Å)	5.20
5	V ($Å^3$)	47.61
6	Crystallite size Scherrer (nm)	83.06
7	Crystallite size WH (nm)	144.81
8	ε(%)	0.0011

Thermogravimetric Analysis (TGA)

Evaluating the thermal stability of Phase Change Materials (PCMs) is essential for determining their suitability in Thermal Energy Storage (TES) systems. In this study, Thermogravimetric Analysis (TGA) was employed to assess pure PEG6000 and its composites with zinc oxide (ZnO). Figure 6(a) shows that the initial degradation temperature of pure PEG6000 was 371.64°C, and it completely decomposed without leaving any residue. The DTG curve indicates that the peak decomposition temperature of pure PEG6000 was 416°C, with a maximum mass loss rate of 3.19%/min. Figures 6(b) and 6(c) show the analysis results with the addition of 8 and 12 wt.%ZnO.



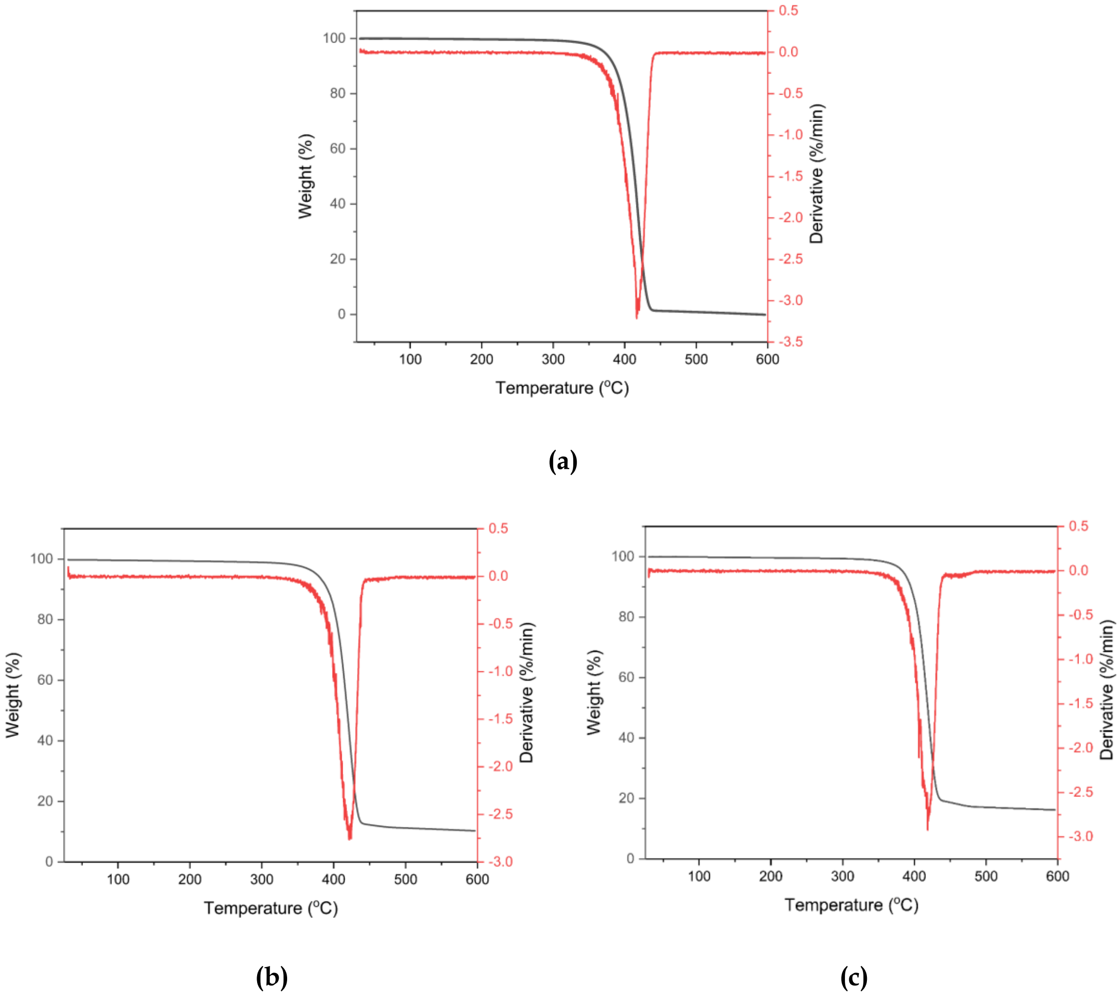
(a)

(b)

(c)

Figure 5. Results of the Differential Scanning Calorimetry (DSC) analysis. (a) PEG6000 (b) PEG-ZnO 8% (c) PEG-ZnO 12%.

The initial degradation temperature rose to 376.99°C and 384.35°C for PEG-ZnO 8 and 12%, respectively. This implies that the incorporation of ZnO enhanced the thermal stability of PEG6000, making it more resistant to high temperatures. At 600°C, the residues left by PEG-ZnO 8 and 12% were 10.3% and 16.3%, respectively. Additionally, there was a shift in the decomposition peak temperature in the DTG curve to 420°C for PEG-ZnO 8%, and a slight decrease to 418°C for PEG-ZnO 12%. The mass loss rate also decreased to 2.70%/min for PEG-ZnO 8% but slightly increased to 2.90%/min for PEG-ZnO 12%. This finding is consistent with previous research showing that the addition of particles such as TiO_2, improves the thermal stability. Mass loss occurred at higher temperatures (240°C) than that of pure PEG (180°C) [39]. A similar trend was observed with the addition of Cu, where pure PEG began to degrade in the temperature range of 300–410°C, while PEG/Cu degraded at slightly higher temperatures, namely 315–410°C [40]. The findings in this study show that the addition of ZnO not only delays the onset of thermal degradation but also changes the overall degradation characteristics of PEG6000. ZnO acts as a thermal barrier by forming a protective layer on the surface of the material, which effectively delays evaporation during the degradation process. This layer helps stabilise the temperature during mass loss and reduces the heat transfer from the environment into the PCM. Furthermore, ZnO inhibits the decomposition of volatile compounds within PCM, thereby enhancing their resistance to elevated temperatures and prolonging their lifespan.[39][41] [29].



(a)

(b)

(c)

Figure 6. TGA and DTG analysis of (a) PEG6000 (b) PEG-ZnO 8% and (c) PEG-ZnO 12%.

Thermal Conductivity

PCMs are widely used in heat-storage applications. However, PCMs have a significant limitation in the form of low thermal conductivity, which hinders the process of heat charging and discharging and reduces the system efficiency [42]. Thermal conductivity is facilitated by two main mechanisms: transfer by electrons and phonons [43]. In non-metallic PCMs, heat transfer is mainly due to phonons, which are lattice vibrations that carry heat energy [44]. Table 2 shows findings of thermal conductivity measurements. The tests indicated that the thermal conductivity of pure PEG6000 was only 0.295 W/m·K. To overcome this limitation, ZnO has been used which has a high thermal conductivity of 29 W/m·K [45]. Incorporating 8 wt.% ZnO led to a 26.44% improvement in the thermal conductivity of the PCMs. However, the augmentation in thermal conductivity with the addition of 12 wt.% ZnO was marginally higher at 28.14% compared to the 8 wt.% ZnO addition. The enhancement in thermal conductivity was further facilitated by the incorporation of SDBS as a surfactant, which played a crucial role in achieving a uniform dispersion of ZnO particles within the PEG matrix. As explained in the previous paragraph, this uniform distribution arises because SDBS changes the surface properties of the particles using electrostatic forces to create repulsive forces between particles. In addition, SDBS forms a physical barrier between the particles, thereby reducing the formation of agglomerations, usually caused by van der Waals forces [35][36]. Consequently, this improved dispersion minimises the formation of nanoparticle clusters that could obstruct the heat flow path, ultimately enhancing the thermal conductivity of the PCM composite [46]. Improved thermal conductivity enables faster responses in TES systems by accelerating the melting and solidification processes. Previous investigations revealed that the temperature of pure PEG increased from 27.2 to 41.6°C, whereas the PEG/AlN composite with Al_2O_3 added experienced a temperature rise from 26.9 to 46.5°C after 100 s. This resulted in a 36.1% enhancement in the heat storage rate of the PEG/AlN-Al_2O_3 composite, which was attributed to the substantial improvement in PEG's thermal conductivity due to the inclusion of the Al_2O_3 additive and AlN framework [47].

Table 2. Thermal conductivity of PEG6000, PEG-ZnO and previous data.

No	Sampel	Thermal conductivity (W/m·K)
1	PEG6000	0.295
2	PEG6000	0.25 [48]
3	PEG6000	0.297 [49]
4	PEG-ZnO 8%	0.373
5	PEG-ZnO 12%	0.378
6	PEG-Al_2O_3 10%	0.39 [50]

Conclusion

In this study, the thermophysical behaviour of PEG-based composites was comprehensively investigated by incorporating 8 and 12 wt.% ZnO with SDBS as a surfactant. Several key findings were obtained from these results. First, the addition of SDBS was effective in enhancing the thermophysical properties of the PEG-based PCMs. DSC analysis showed an increase in latent heat from 214 J/g (pristine PEG 6000) to 238.3 J/g (8 wt.% ZnO) and 257.7 J/g (12 wt.% ZnO). This finding contrasts with those of previous studies, which typically reported a decrease in the latent heat with the addition of fillers. Meanwhile, the TGA results indicated an increase in the decomposition temperature from 416°C (pristine PEG 6000) to 420°C and 418°C for 8 and 12 wt.% ZnO, respectively, suggesting improved thermal stability. Thermal conductivity tests also revealed significant improvements: 28.2% for 8 wt.% ZnO and 30.3% for 12 wt.%. XRD analysis confirmed that there were no significant changes in the crystalline structure of PEG6000 after the addition of ZnO and SDBS, indicating that the structural integrity of the composite was maintained. Overall, the findings demonstrate that incorporating ZnO and SDBS into the PEG6000 matrix effectively enhances the thermal conductivity, latent heat capacity, and thermal stability without altering the fundamental crystal structure. Improved thermal conductivity enables faster responses in TES systems by accelerating the melting and solidification processes, whereas a higher latent heat capacity allows for greater energy storage. The developed PEG-ZnO composites show great potential for low-temperature TES applications (below 100°C), particularly in WHR systems across industrial sectors such as manufacturing, HVAC systems, internal combustion engines and residential energy systems. Future research should focus on optimising the formulation, testing the long-term thermal cycling stability, and evaluating the real-world performance to ensure practical feasibility.

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