A Comparative Study on TiO_2/Graphite–PEG and Graphite/Carbon Fibre-Paraffin Shape Stabilized Phase Change Materials for Thermal Energy Storage Applications

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ABSTRACT
Shape-stabilized phase change materials (SSPCMs) are promising candidates for latent heat thermal energy storage systems due to their high energy density and ability to prevent leakage during phase transitions. This study presents a comparative analysis of two SSPCM systems: TiO_2/graphite–polyethylene glycol (PEG) and graphite/carbon fibre/graphene–paraffin composites. Both composites were prepared by vacuum-assisted infiltration of molten PCMs into porous expanded graphite networks, with the addition of functional fillers to enhance structural integrity and thermal stability. Scanning electron microscopy (SEM) revealed distinct microstructural features for each system; TiO_2 nanoparticles were uniformly dispersed within the PEG matrix and anchored onto graphite surfaces, while carbon fibres and graphene nanoplatelets formed a hierarchical interconnected network within the paraffin-based composites. Differential scanning calorimetry (DSC) demonstrated that both systems preserved high latent heat storage capacities with slight shifts in phase transition temperatures compared to pure PCMs. Thermogravimetric analysis (TGA) showed improved thermal stability of the SSPCMs relative to neat PCMs, with filler composition significantly affecting degradation onset temperatures. In TiO_2/graphite–PEG composites, DSC analysis showed melting temperatures of 61.4-62.7 °C and solidification temperatures of 53.1-54.0 °C, with latent heats of 185-210 J g^{-1} depending on TiO_2 content. Graphite/carbon fibre/graphene–paraffin composites exhibited melting temperatures of 54.8-55.6 °C and solidification temperatures of 48.9-49.7 °C, with latent heats of 140-160 J g^{-1}. Thermogravimetric analysis revealed improved degradation onset temperatures: TiO_2/graphite-PEG composites showed higher thermal stability compared to pure PEG, while carbon fibre/graphene–paraffin composites exhibited enhanced thermal resistance relative to pure paraffin. The TiO_2/graphite-PEG composites exhibited higher latent heat capacities and enhanced thermal resistance, whereas the graphite/carbon fibre/graphene–paraffin composites provided superior mechanical reinforcement and phase change reliability. These findings offer insight into the design optimization of SSPCMs tailored for specific thermal management applications.

KEYWORDS: *Phase Change Materials, Hybrid Composites, Graphene, Nano titanium dioxide, Paraffin, Polyethylene Glycol, Thermal energy storage*

INTRODUCTION
Latent heat thermal energy storage (LHTES) systems are crucial for improving energy efficiency in applications ranging from renewable energy harvesting to building climate control (Stonehouse & Abeykoon, 2022; S. Wu et al., 2020; G. Yang et al., 2019). Organic phase change materials (PCMs), such as polyethylene glycol (PEG) and paraffin wax, are widely studied due to their high latent heat, chemical stability, and relatively safe handling properties (Kadoono & Ogura, 2014; Wong et al., 2023; Wuliu et al., 2021). However, the practical utilization of these materials is hindered by the inherent problem of PCM leakage during the solid-liquid phase change and limited thermal durability at elevated temperatures, which reduces system lifetime and effectiveness (Ouikhalfan et al., 2022).
Shape-stabilized phase change materials (SSPCMs) address these limitations by physically confining the PCM within a porous and rigid supporting matrix, which prevents leakage and maintains structural integrity throughout repeated thermal cycles (Falahatian et al., 2022). Expanded graphite (EG) is commonly used as such a matrix because of its highly porous structure, excellent thermal conductivity, and chemical compatibility with organic PCMs (Zhou et al., 2022). However, EG-based SSPCMs can benefit from the incorporation of functional fillers

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to improve thermal stability, mechanical strength, and phase change reliability.

Metal oxide nanoparticles, such as titanium dioxide (TiO_2), are known to enhance thermal stability and mechanical properties of SSPCMs due to their ceramic nature and strong interactions with polymeric matrices (Venkitaraj & Suresh, 2019; J. Zhang et al., 2024; Zou et al., 2022). On the other hand, carbon-based fillers such as carbon fibres and graphene nanoplatelets contribute to the formation of mechanically robust, conductive networks that can reduce phase separation and improve the overall durability of PCM composites (Chen et al., 2021; Mishra et al., 2022).

Although previous research has separately explored TiO_2-enhanced PEG/graphite composites and carbon fibre/graphene-modified paraffin composites, a direct comparative evaluation of these two SSPCM systems remains limited (Sun et al., 2021; Zou et al., 2022). A comprehensive understanding of how filler type, PCM matrix, and microstructural characteristics affect phase change behaviour and thermal stability is critical for advancing SSPCM design.

This study undertakes a systematic comparison between TiO_2/graphite–PEG and graphite/carbon fibre/graphene–paraffin composites prepared via vacuum infiltration methods. Their microstructures, phase transition characteristics, and thermal stabilities are investigated through SEM, DSC, and TGA analyses. The outcomes elucidate the influence of filler chemistry and morphology on SSPCM performance, providing valuable guidance for tailoring materials to specific energy storage needs.

MATERIALS AND METHODS

Polyethylene glycol (PEG, molecular weight ~8000 g/mol) and commercial-grade paraffin wax were selected as the organic PCMs. Expanded graphite was prepared by thermal exfoliation of natural flake graphite at high temperature, resulting in a lightweight, highly porous network with interconnected pores suitable for PCM impregnation.

Titanium dioxide nanoparticles (~25 nm diameter) were used as ceramic fillers for the PEG-based composites. For the paraffin-based system, carbon fibres (3 mm length) and graphene nanoplatelets were employed to form a multi-scale reinforcing network within the graphite matrix.

The TiO_2/graphite–PEG composites were fabricated by first melting PEG at 80 °C and dispersing TiO_2 nanoparticles at weight fractions ranging from 1 to 5%. The dispersion was achieved by mechanical stirring to ensure uniform distribution. Expanded graphite samples were subjected to a vacuum to remove trapped air and then infiltrated with the molten PEG/TiO_2 mixture. The composite was cooled to room temperature to solidify the PCM within the graphite pores.

Similarly, the graphite/carbon fibre/graphene–paraffin composites were prepared by mixing expanded graphite, carbon fibre, and graphene nanoplatelets in targeted weight ratios. Molten paraffin wax was added under stirring, and vacuum-assisted infiltration was performed to achieve homogeneous filling of the graphite network. The composites were cooled to solidify the paraffin and fix the filler arrangement.

Microstructural analysis was conducted using scanning electron microscopy (SEM) to observe the dispersion and interaction of fillers within the PCM matrix and the graphite network. A Field Emission Gun Scanning Electron Microscope (FEG-SEM, Oxford Instruments) was employed to examine the microstructure of samples with thicknesses between 8 mm and 10 mm; non-conductive samples were sputter-coated with a 6.0 nm thick gold/palladium layer (80:20 ratio) using a 40 mA current for up to 5 minutes to ensure electrical conductivity and prevent static charge buildup during electron irradiation.

Phase change behaviour was assessed via differential scanning calorimetry (DSC), measuring melting and solidification temperatures as well as latent heat of fusion. Differential Scanning Calorimetry (DSC) was performed on samples weighing 2–10 mg sealed in aluminium pots, heated from –30°C to 120°C at a rate of 10°C/min, with thermal properties analyzed and data processed by TA Universal Analysis 2000 software.

Thermal stability was evaluated using thermogravimetric analysis (TGA) under nitrogen atmosphere, identifying degradation onset temperatures and mass loss profiles. Thermogravimetric analysis (TGA) was conducted on approximately 10 mg samples using a Q500 analyzer with a heating rate of 10°C/min from 30°C to 500°C under an inert atmosphere, and data were analyzed using TA Universal Analysis software.

RESULTS AND DISCUSSION

Microstructure

The scanning electron microscopy (SEM) micrographs in Figure 1 reveal distinct morphological features of various carbon-based and composite materials, enabling a clear comparison of their surface structures and potential implications for functional performance. In the upper-left panel, the graphene sample exhibits a characteristic sheet-like morphology with irregular wrinkling and folding. These thin, planar flakes, outlined by the highlighted regions, indicate the two-dimensional nature of the graphene structure, which is consistent with its high aspect ratio and large surface area. Such morphology is expected to enhance electrical conductivity and facilitate electron transport when integrated into composite systems (Fan et al., 2014; Xia et al., 2019).

The upper-right panel reveals the microstructure of carbon fibre, characterized by its long, continuous filamentous morphology. The fibres exhibit significant surface roughness and a highly entangled arrangement, which are critical features for enhancing mechanical interlocking within polymer matrices (Jiang et al., 2024). This roughened, fibrillar surface increases the effective contact area between the fibres and the surrounding matrix, promoting stronger interfacial adhesion (X. Wu et al., 2021). The entanglement and microfibrillar features observed are indicative of a highly irregular surface, which can influence phenomena such as wettability and adhesion at the microscopic scale.

In the lower-left panel, graphite is presented as large, compact aggregates composed of stacked platelets. The morphology displays tightly layered structures with relatively low edge compared to more exfoliated carbon forms. This compact platelet stacking is indicative of a stable layered arrangement, which contributes to the material's inherent structural integrity. The reduced surface area and limited edge sites suggest lower chemical reactivity at the particle surfaces compared to more dispersed carbon allotropes (Tian et al., 2016).

The lower-right panel illustrates the composite of graphite and TiO_2 nanoparticles, where graphite retains its plate-like morphology and is decorated with uniformly dispersed, near-spherical TiO_2 aggregates. The nearly homogeneous distribution of TiO_2 on graphite surfaces suggests effective surface coverage, potentially modifying the surface chemistry and optical properties of the composite (Venkitaraj & Suresh, 2019; Zou et al., 2022). The intimate contact between TiO_2 nanoparticles and graphite platelets may enable combined functionalities related to surface interactions, such as enhanced photocatalytic activity or UV absorption, stemming from the distinct properties of each phase (J. Zhang et al., 2024).

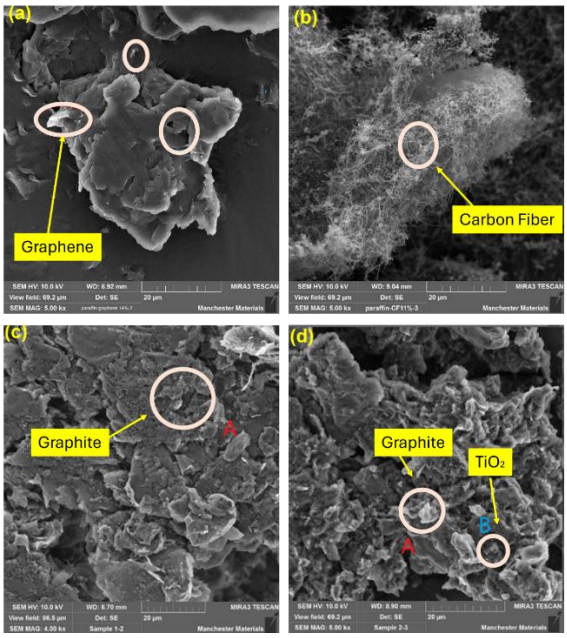

Figure 1. SEM images of (a). Graphene-Paraffin, (b). Carbon Fiber-Paraffin, (c). 35% Graphite 5% TiO_2 PEG and (d). 30% Graphite 10% TiO_2 PEG hybrid composites.

In contrast, the graphite/carbon fibre/graphene–paraffin composites exhibited a hierarchical structure formed by the interplay of carbon fibres bridging larger graphite pores and graphene nanoplatelets

coating the graphite surfaces and paraffin interfaces. This interconnected carbon network appeared to minimize PCM leakage pathways by physically confining paraffin within the graphite pores and DSC measurements showed that the melting temperatures of PEG in the TiO_2/graphite–PEG composites remained close to that of pure PEG, with only minor shifts (approximately 1–2 °C) towards lower temperatures as TiO_2 content increased. The latent heat of fusion for these composites was high, ranging from 185 to 210 J/g depending on filler loading, though a slight reduction was noted at higher TiO_2 concentrations due to the dilution of PCM by the ceramic filler. The narrow melting and solidification peaks indicated stable and reproducible phase transitions, with minimal supercooling effects observed (J. Zhang et al., 2022, 2024)

Thermal Stability

TGA analysis under a nitrogen atmosphere revealed distinct thermal behaviours for the enhancing the composite's structural cohesion (Ohayon-Lavi et al., 2021; W. Yang & Kim, 2023a; D. Zhang et al., 2018). The nanoscopic graphene sheets contributed to sealing microvoids, potentially promoting uniform phase transitions and mitigating paraffin migration (W. Yang & Kim, 2023b).

Phase Transition Behaviour

For the graphite/carbon fibre/graphene–paraffin composites, paraffin melting temperatures were similarly maintained near pure paraffin values, with small decreases of about 1 °C attributed to the presence of fillers. The latent heat ranged between 140 and 160 J/g, with a slight decrease at increasing graphene content above 2 wt%. Notably, the presence of graphene nanoplatelets sharpened the melting and solidification peaks, suggesting enhanced nucleation and improved crystallization kinetics during cooling cycles, which is beneficial for consistent thermal cycling (Chen et al., 2021; Wong et al., 2024). TiO_2/graphite–PEG and graphite/carbon fibre/graphene–paraffin composites as shown in Table 1.

Table 1. TGA data for PCM hybrid composites

Carbon Fibre (wt.%)	Graphene (wt.%)	Graphite (wt.%)	TiO_2 (wt.%)	Onset Temp (°C)	Temp at 5% Loss (°C)	First Degradation Peak	Final Degradation Temp (°C)	Final Mass Residual (%)
4	–	–	–	164.50	206.40	–	359.99	2.173
8	–	–	–	192.06	240.68	–	342.50	7.956
–	4	–	–	172.14	216.55	–	358.22	2.530
–	8	–	–	169.55	216.57	–	376.89	6.147
–	–	35	5	-	-	80.00	270.00	59–65
–	–	30	10	-	-	72.91	292.12	51.83

For the graphite/carbon fibre/graphene–paraffin composites, the onset temperature varied with filler type and loading: 164.50 °C (4 wt.% carbon fibre), 192.06 °C (8 wt.% carbon fibre), 172.14 °C (4 wt.% graphene), and 169.55 °C (8 wt.% graphene). The second peak (temperature at 5% loss) followed the same trend, increasing from 206.40 °C to 240.68 °C for carbon fibre, and from 216.55 °C to 216.57 °C for graphene. The higher loadings of carbon fibre produced the most notable improvements in thermal stability, accompanied by greater final mass residuals (7.956% for 8 wt.% carbon fibre versus 2.173% for 4 wt.%). Graphene-filled samples exhibited higher final degradation temperatures (376.89 °C for 8 wt.% graphene) compared to carbon fibre, suggesting that the continuous carbon network formed by graphene can further delay degradation by restricting oxygen diffusion and dispersing heat more evenly (Jiang et al., 2024; X. Wu et al., 2021). The continuous carbon filler network likely impedes thermal decomposition by restricting oxygen diffusion and dispersing heat more evenly throughout the composite (Zhu et al., 2023).

For the TiO_2/graphite–PEG composites, the first degradation peak (onset) occurred at 80.00 °C. for the graphite 35 wt.%–TiO_2 5 wt.% sample and 72.91 °C for the graphite 30 wt.%–TiO_2 10 wt.% sample. The second degradation peak (corresponding to 5% weight loss) was observed at 270.00 °C and 292.12 °C, respectively. Both composites exhibited earlier degradation onset temperatures compared to neat PEG, which began degrading around 310 °C; however, they demonstrated enhanced thermal stability through increased char residue and sooner major degradation, attributed to the barrier and heat dissipation effects of TiO_2 nanoparticles and porous structure after adding the fillers (Sun et al., 2021; J. Zhang et al., 2024). Final mass residuals were 59–65% and 51.83%, indicating the significant char yield retention of these formulations. This enhancement is attributed to the physical barrier effect and heat dissipation properties of TiO_2 nanoparticles, which slow the release of volatile degradation products.

Comparative Assessment

The TiO_2/graphite–PEG system demonstrates superior latent heat capacity, consistent with PEG's inherently higher heat of fusion compared to paraffin. Furthermore, the ceramic TiO_2 fillers confer improved high-temperature stability, extending the operational range of the PCM. However, increased TiO_2 content reduces PCM loading due to filler volume occupation, which may limit total energy storage.

In contrast, the graphite/carbon fibre/graphene–paraffin composites offer enhanced mechanical reinforcement and phase change reliability due to the multi-scale carbon network. This filler structure effectively suppresses leakage and stabilizes phase transitions, although the overall latent heat is somewhat lower than the PEG-based system.

Choosing between these SSPCMs involves trade-offs: the PEG–TiO_2 composites are preferable where high energy density and thermal endurance are paramount, whereas paraffin–carbon fibre/graphene composites are better suited to applications prioritizing mechanical stability and cycle life.

CONCLUSIONS

This work presents a detailed comparative analysis of two shape-stabilized phase change material systems aimed at thermal energy storage applications. TiO_2/graphite–PEG composites exhibited high latent heat capacities and improved thermal stability, facilitated by ceramic nanoparticle reinforcement within the porous graphite network. Conversely, graphite/carbon fibre/graphene–paraffin composites formed a hierarchical carbonaceous structure that enhanced mechanical integrity and stabilized phase change behaviour, though with slightly reduced latent heat storage.

SEM analysis revealed distinct filler dispersion and matrix interactions in each system, which correlated with the observed phase transition and thermal stability properties. The enhanced thermal resistance in TiO_2-containing composites and the superior structural cohesion in carbon fibre/graphene-filled paraffin composites demonstrate the importance of filler type and morphology in SSPCM design. These findings inform the selection and engineering of SSPCMs for specific thermal management requirements, enabling optimized performance in applications ranging from building temperature regulation to renewable energy systems.

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