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Article

PEG 4000/rGO-Based Phase Change Material Composites: Synthesis Routes, Thermal Performance, and Research Gaps

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Abstract

Polyethylene glycol 4000 (PEG 4000) is an organic phase change material (PCM) with potential for use in thermal energy storage due to its suitable transition temperature range, good chemical stability, and relatively high latent heat. The use of PEG 4000 is still limited by its low thermal conductivity and leakage when the material is in the liquid phase. This review discusses the development of PEG 4000 composites with graphene and reduced graphene oxide (rGO) via blending, solvent-assisted processes, impregnation into porous scaffolds, and encapsulation. The analysis focuses on the relationship between material composition, scaffold structure, and process conditions on latent heat, thermal conductivity, shape stability, and thermal cycling stability. The results indicate that dispersed graphene/rGO can form heat conduction pathways, while porous structures are capable of retaining PEG through capillary forces and interfacial interactions. Increasing the amount of support material does not always result in better thermal performance, as it can reduce the active PEG fraction and affect crystallinity. Impregnation into a porous framework shows potential for producing composites with high PCM content and good dimensional stability. Meanwhile, blending and encapsulation offer advantages in terms of process simplicity and material integration. Differences in PEG type, filler, composition, and testing methods mean that results across studies cannot yet be directly compared. A comparison of commercial rGO and biomass-based rGO in a PEG 4000 system under equivalent conditions is still needed to evaluate the effect of the material source on composite performance.

Keywords: Phase change material (PCM), PEG 4000, graphene, reduced graphene oxide (rGO), thermal conductivity, latent heat, impregnation

1. Introduction

Thermal energy storage is one approach that can be used to increase the utilization of renewable energy and industrial waste heat. In latent heat storage systems, phase change materials (PCMs) absorb and release energy when undergoing a reversible phase transition within a specific temperature range. During this process, most of the energy is stored in the form of latent heat, so the change in the material's temperature is relatively small compared to the amount of energy absorbed or released [1]–[8].

Polyethylene glycol (PEG) is one of the organic PCMs that has been extensively developed due to its good chemical stability, low volatility,

ease of processing, and melting point that varies with its molecular weight. PEG 4000 has thermal characteristics suitable for energy storage in the mid-temperature range. However, PEG's thermal conductivity is relatively low, and the material may leak once it exceeds its melting point. These conditions necessitate the use of conductive fillers, porous support materials, or encapsulation structures to maintain its shape while improving heat transfer [9]–[15].

Graphene-based materials are widely used as thermal reinforcement components because they can form heat conduction pathways even at relatively low concentrations. Graphene, graphene oxide (GO), and reduced graphene oxide (rGO) have different structures and surface properties, so their applications must be distinguished. rGO is obtained by reducing GO, a process that removes some of the oxygen groups; however, the resulting structure may still contain defects and residual oxygen groups. The degree of reduction, the number of layers, and the surface chemistry of rGO are influenced by the synthesis method used [1], [2], [16]. In PCM composites, graphene derivatives can be used as dispersed fillers or formed into three-dimensional porous frameworks. These porous structures not only facilitate heat transfer but also help retain liquid PEG through capillary forces, surface tension, wettability, and interfacial interactions [1], [2], [17]–[21].

The thermal performance of PEG–graphene/rGO composites is influenced by the shape and arrangement of the materials within the composite. Chen et al. reported temperature-dependent thermal rectification in a binary eicosane/PEG 4000 system embedded in an rGO aerogel [22]. This phenomenon stems from the design of a specific composite system and does not represent the general characteristics of all PCMs. In other PEG/graphene systems, the parameters most frequently studied include thermal conductivity, latent heat, resistance to leakage, dimensional stability, photothermal conversion, and thermal cycle stability [17]–[21], [23].

Increasing the filler fraction does not always result in an overall improvement in performance. A conductive filler network can accelerate heat transfer, but an excessive amount of non-PCM material can reduce the active PEG fraction. Strong interactions between PEG and the carrier material can also affect the crystallization process, leading to a decrease in latent heat. Therefore, the evaluation of PCM composites must simultaneously consider thermal conductivity, latent heat, PCM content, dimensional stability, and cycle stability [1], [2], [5]–[7].

Based on these conditions, this review compares four synthesis routes used for PEG 4000–graphene/rGO-based PCM composites: melt blending, solvent casting, impregnation into a porous scaffold, and encapsulation. The comparison is based on material composition, process conditions, and reported thermal parameters. Studies using PEG 4000 with graphene/rGO were used as the primary reference, while other PEG systems were used as supporting data when they provided relevant information regarding synthesis mechanisms or heat transfer. The discussion regarding commercial rGO and biomass-based carbon materials focused on identifying research gaps, as comparative data under equivalent PEG 4000 conditions remain limited.

2. Material and Method

This article employs a literature review method to analyze the synthesis and thermal performance of phase change material (PCM) composites using PEG 4000-based materials and additives such as graphene, graphene oxide (GO), reduced graphene oxide (rGO), as well as relevant porous matrix materials. Subsequently, the literature was selected and utilized based on the topic of PEG 4000-based PCMs, the relationship between the support material and PEG retention or heat transfer, the completeness of bibliographic information, and the availability of data on synthesis or thermal characteristics.

2.1 Theoretical Basis of PEG 4000 and Graphene-Based Additives

2.1.1 PEG 4000 as A Heat Storage Medium

PEG stores and releases thermal energy primarily through changes in enthalpy during phase transitions. The molecular weight of PEG affects the transition temperature range, so not all types of PEG can be assumed to have the same thermal properties. Therefore, the composite design aims to maintain the highest possible fraction of active PEG while still providing sufficient structural integrity and enhanced heat transfer [4], [24].

2.2 Graphene, GO and rGO Function

Graphene derivatives serve two primary functions in PCM composites. Dispersed carbon

nanosheets or fillers can reduce the effective thermal resistance by forming heat conduction pathways. Meanwhile, graphene/rGO aerogels, which have a self-supporting structure, can form a porous framework to retain liquid PEG and reduce leakage. The balance between pore size, pore connectivity, surface chemistry, wettability, and PEG content determines whether the support material primarily enhances heat transfer, shape stability, or both [1], [2], [6], [16], [17], [19]–[21], [25], [26].

2.3 Relathionship Between Thermal Conduktivity and Latent Heat

Thermal conductivity and latent heat must be analyzed simultaneously because an increase in one parameter is not always accompanied by an increase in the other. Conductive fillers can accelerate heat transfer, but an excessively high filler fraction can reduce the amount of active PCM or limit the crystallization process, thereby decreasing the enthalpy. In porous materials, the PEG content can remain high; however, the interaction between PEG and the pore surface can also affect crystal formation. Therefore, the evaluation of composites must simultaneously consider PEG content, latent heat, thermal conductivity, dimensional stability, cycle stability, and process complexity [5], [9], [11]–[14], [27].

2.4 Synthesis Route

2.4.1 Melt Blending Methods

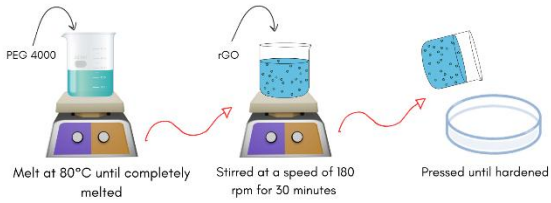

Fig. 1. The melt blending method PEG 4000 composites with graphene/rGO fillers. The main steps include melting the PEG, adding the filler, the dispersion process, and composite formation. Source: Author's illustration adapted from Ref. [10], [11].

The blending method is attractive because the process is relatively simple and does not require the separate fabrication of a porous framework. PEG is melted or dissolved, and then the conductive filler is dispersed through stirring, sonication, or compounding before the mixture is solidified or dried. Research on PEG 4000/graphene nanoplatelets shows that carbon fillers can enhance heat transfer while maintaining reasonably good latent heat, but agglomeration and changes in crystallinity caused by the fillers still need to be taken into account [10], [11]. PEG/PBAT composites with AlN and CNT fillers also demonstrate that the compounding process and filler interface modification can be used to control heat transfer in PEG-based polymer PCM systems [28].

2.5 Solvent Casting Methods

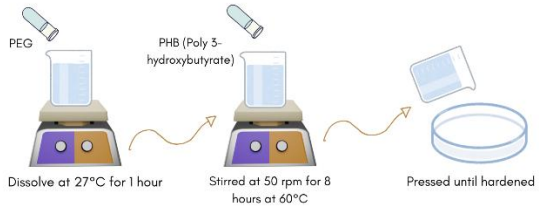

Fig. 2. Solvent Casting Method PEG 4000 composites with graphene/rGO. The process stages include PEG dissolution, filler dispersion, mixing, and solvent removal until a composite is formed. Source: Author's illustration adapted from Ref. [21], [29]

Solution-based processes can help improve dispersion by first dispersing the graphene additive before mixing it with PEG or a PEG-containing polymer matrix. This method can be beneficial at low filler concentrations, but requires additional steps such as drying, solvent recovery, and consideration of process scaling. Solid-solid PEG/GO systems and PEG/hybrid carbon composites demonstrate that solution-based or polymer matrix processes can improve the dispersion and thermal performance of the materials. However, differences in PEG type and polymer architecture must be considered, as they can affect crystallization and heat transfer. Therefore, data from these systems are used to elucidate the process mechanisms and are not directly compared with the performance of PEG 4000–rGO [21], [29].

2.6 Impregnation Methods

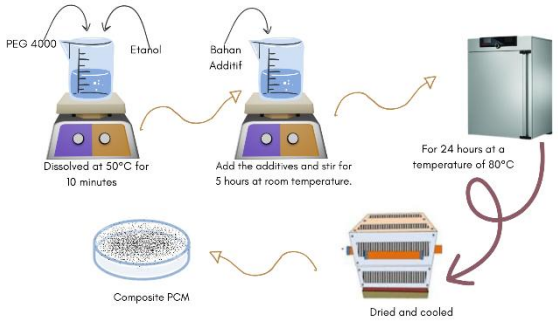

Fig. 3. PEG impregnation method for graphene/rGO-based porous scaffolds via capillary infiltration with or without vacuum assistance. Source: Author's illustration adapted from Ref. [17], [19], [20], [22], [25], [26].

Impregnation of porous frameworks is performed by introducing liquid PEG or PEG in solution into the porous structure via capillary action, vacuum-assisted methods, or other infiltration techniques. This method is widely used in PEG research because a continuous matrix material can simultaneously prevent leakage and form heat transfer pathways. PEG/rGO and graphene aerogel systems exhibit this dual function directly or nearly directly [17], [19], [20], [22], [25], [26].

Quantitative data show that impregnation of porous structures can yield a favorable combination of PCM content, latent heat, and heat transfer, although the results are strongly influenced by the matrix composition. [17] reported a PEG@MF/rGO/Cu-C composite with melting and crystallization enthalpies of 148.3 and $143.9\ J/g^{-1}$, respectively, and a thermal conductivity of $0.4621\ Wm^{-1}/K^{-1}$ [17]. [25] reported a PEG composite with a BNNS/rGO aerogel matrix that exhibits a thermal conductivity of up to $1.12\ Wm^{-1}/K^{-1}$ and a latent heat of $187.2\ J/g^{-1}$ [25]. [19] reported a PEG content of 96.0 wt% and a latent heat of $223.2\ J/g^{-1}$n a composite with a graphene aerogel scaffold [19]. Differences in the composition and architecture of the scaffolds in these three systems mean that their values cannot be directly compared based on a single parameter alone.

Studies on non-rGO porous materials have shown that capillary forces and interfacial interactions play a role in PEG retention and its crystallization process. Materials that have been used include SiO_2-modified expanded graphite, fumed silica, carbon/carbon-fiber felt, expanded vermiculite, carbon from cotton fabric waste, sunflower carbon, cornstalk biochar, hemp stalk biochar, and modified rice husk ash supports [9], [12]–[15], [28], [30]–[34].

2.7 Encapsulation Methods



Fig. 4. Encapsulation method in a PEG system with a core–shell structure, where PEG serves as the core phase and rGO/polymer as the shell material. Source: Author's illustration adapted from Ref. [35]

Encapsulation involves placing PEG inside a shell, fiber, tube, polymer network, or other supporting structure. This approach can reduce leakage and facilitate the integration of the material into devices, but the addition of a shell or structural matrix reduces the mass fraction of active PCM and can increase fabrication complexity. Das et al. used PEG as the core in electrospun core-shell fibers with rGO/polycarbonate as the shell [23]. PEG–magnetite concrete systems with macroencapsulation, as well as PEG-containing mortar, were used to complement the discussion of PCM retention at the device and building material scales [35]–[37].

In cement-based systems, PEG that is distributed or absorbed within the matrix is more accurately categorized as PCM placement within the matrix or macro-scale retention. The term "core-shell encapsulation" is used when the core and shell structures can be clearly distinguished in the material.

3. Result and Discussion

3.1 Thermal Performance Comparison

Table 1. Comparison of the thermal performance of a PEG 4000 based PCM composite with rGO additive

Ref.	Komposit PCM	Metode	$\Delta H\ (J/g)$	K (W/mK)
[22]	PEG 4000 /rGO aerogel	Impregnation	143,5	-
[10]	PEG 4000 / GNP	Solution assited blending	170,6	0,776
[38]	PEG / rGO core-shell fiber	Encapsulation	-	-

Based on Table 1, the addition of graphene/rGO-based materials to PEG results in improved heat transfer capabilities and dimensional stability through the formtion of conductive networks and porous structures. The impregnation method using a graphene or rGO scaffold is able to maintain a high PEG content due to the retention effect within the material's

pores. Meanwhile, the blending method enhances thermal conductivity through the formation of heat transfer pathways; however, the addition of carbon filler must be controlled as it can reduce the fraction of active energy-storage material. Therefore, the selection of a synthesis method must consider the balance between thermal conductivity, latent heat, and the shape stability of the composite.

3.2 Comparison of Synthesis Route Characteristics

Blending involves a relatively simple process and does not require the separate fabrication of a support structure, but the homogeneity of the filler dispersion must be controlled [10], [11], [28]. Solvent-assisted processes can help produce a more uniform dispersion, but they require drying steps and solvent handling [21], [29]. Impregnation into a porous framework is suitable for systems requiring high PCM content and good dimensional stability [9], [12]–[15], [17]–[21], [26], [30], [31], [33], [34], [39], [40]. Meanwhile, encapsulation provides stronger retention and facilitates integration into devices, but the mass of the shell or matrix reduces the fraction of active PCM relative to the total mass of the composite [35]–[38].

Based on the available data, impregnation shows advantages, particularly in porous systems with high PCM content and good dimensional stability. However, differences in composition and testing methods across studies have not yet allowed for the conclusion that impregnation is always a superior approach compared to blending, solution-assisted processes, or encapsulation.

3.3 Commercial rGO and Biomass-Based Carbon Materials

A number of studies on PEG composites have utilized carbon from cotton fabric waste, sunflower carbon, corn stover biochar, hemp stalk biochar, cellulose foam containing biochar, rice husk matrix, and cellulose from paper waste [30], [31], [33], [34], [39], [41]–[43]The results of these studies show that porous support materials derived from renewable sources or waste can retain a sufficiently high PCM content, reduce leakage, and still provide good thermal response. These materials should be named according to their reported form—for example, biochar, porous carbon, or biomass-based supports—and should not be equated with rGO unless the graphene oxide reduction process has been verified.

To obtain a valid comparison, commercial rGO and biomass-based rGO must have comparable characteristics, particularly the degree of reduction, oxygen content, surface area, defect density, and pore structure. Both materials must then be tested using PEG 4000, the same filler fraction, process route, DSC procedure, thermal conductivity measurement method, leakage test, and number of thermal cycles. This approach will isolate the influence of the rGO source from the effects of differences in composition and testing methods.

Direct comparisons between commercial graphite-based rGO and biomass-based rGO in a PEG 4000 system remain limited. Most biomass-based studies use biochar, porous carbon, silica/carbon derived from rice husks, or other carbon frameworks that differ in composition and structure from rGO. These differences make it impossible to directly compare thermal conductivity and energy storage capacity to determine which material is superior. Therefore, a comparison between commercial rGO and biomass-based rGO still requires testing under equivalent systems and conditions.

3.4 Challenges and Directions for Research

The development of PCM composites based on PEG 4000 and graphene/rGO still faces several challenges, particularly regarding the consistency of data reporting, thermal cycling stability, and the balance between improved thermal conductivity and latent heat storage capacity. Differences in the types of graphene derivatives, filler composition, PEG molecular weight, and synthesis conditions mean that results across the literature cannot yet be directly compared. Therefore, future research should employ more uniform testing parameters while simultaneously evaluating thermal conductivity, latent heat, dimensional stability, leakage, and changes in properties following thermal cycling [1], [2], [5]–[7].

In addition to thermal performance, future development efforts should consider process efficiency, solvent usage, production costs, and the potential for larger-scale application. A direct comparison between commercial rGO and biomass-based rGO in a PEG 4000 composition and under the same testing conditions is also necessary to gain a clearer understanding of how the carbon source affects the structure and performance of the composite. Thus, material

development should focus not only on improving heat transfer but also on material stability, energy storage capacity, and the sustainability of the production process [1], [2], [16], [41], [42].

4. Conclusions

PEG 4000 has potential as a latent heat storage material, but its low thermal conductivity and leakage in the liquid phase remain major limitations. The addition of graphene/rGO can enhance heat transfer through the formation of a conductive network, while the porous structure can help retain PEG within the composite matrix. The use of a matrix material also affects the fraction of active PEG and crystallinity; thus, an increase in thermal conductivity may be accompanied by a decrease in latent heat. The performance of the composite must therefore be evaluated based on several thermal and structural parameters simultaneously.

Each synthesis route imparts different characteristics to the structure and performance of the composite. Blending involves relatively simple process steps, whereas solvent-assisted processes can help improve filler dispersion but require a solvent removal step. Impregnation into a porous matrix can yield high PCM content and good dimensional stability if the pore structure and interfacial interactions are appropriate. Encapsulation provides stronger retention of liquid PEG but increases the fraction of non-PCM material and process complexity. Given these conditions, the choice of synthesis method must be tailored to the application's requirements, particularly regarding energy storage capacity, heat transfer rate, dimensional stability, and process ease.

Comparisons across the literature are still limited by differences in PEG molecular weight, types of graphene derivatives, filler composition, composite architecture, and thermal testing procedures. Future research should employ more uniform synthesis conditions and characterization methods to enable a clearer comparison of the relationship between material structure and thermal performance. Testing of commercial rGO and biomass-based rGO in a PEG 4000 system with identical compositions and processing conditions is also necessary to evaluate the influence of rGO source on performance and its potential for application as a thermal energy storage material.

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