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Characterization Studies on a Nano-doped Organic Phase Change Material for Improving Thermal Energy Storage

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The increasing need for effective thermal energy storage (TES) systems has prompted the investigation of phase change materials (PCMs) with improved thermal characteristics. This study examines the effect of incorporating magnesium oxide nanoparticles (nano-MgO) into paraffin wax on its thermal storage characteristics. Nano-MgO/paraffin phase change materials were synthesized with nanoparticle loadings ranging from 0 to 2.0 wt.% and characterised by differential scanning calorimetry, thermogravimetric analysis, Fourier-transform infrared spectroscopy, field emission scanning electron microscopy, and thermal conductivity evaluation. The results indicated that nano-MgO substantially enhanced thermal conductivity, attaining a 113.89% improvement at a 2.0% loading compared to unmodified paraffin. FESEM validated the homogeneous dispersion of nanoparticles, whilst FTIR examination revealed the absence of chemical interactions, indicating physical stability. DSC measurements indicated considerable decreases in latent heat with increased concentrations of nano-MgO; nevertheless, the 1.0% composite achieved a balance between greater thermal conductivity and negligible latent heat loss. TGA exhibited enhanced thermal stability, with residual mass increasing to 0.9% at 450°C for the 2.0% composite. These findings demonstrate the exciting prospects of nano-MgO/paraffin phase change materials for solar energy systems and industrial waste heat recovery, presenting a feasible approach to enhance energy storage efficiency.

Keywords: Paraffin; PCM; nano-MgO; thermal storage; characterization

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The growing worldwide need for energy, along with the pressing necessity to address climate change, have intensified the focus on finding sustainable energy solutions. Among these, thermal energy storage (TES) systems have become essential technologies for improving energy efficiency and utilising sources of clean energy like solar and geothermal energy [1, 2]. Among the different strategies for thermal energy storage, latent heat thermal energy storage (LHTES) utilising phase change materials (PCMs) has become a notable option because of its impressive energy density and capacity to store and release thermal energy at nearly constant temperatures [3]. Phase change materials (PCMs) are essential to thermal energy storage systems because they can effectively store and release significant amounts of latent heat during phase transitions, usually involving solid-liquid changes [4].

In the past decade, PCMs have been extensively utilized for TES due to their impressive properties. Because of their congruent melting behavior chemical inertness, thermal stability, recyclability and wide operating temperature ranges, organic PCMs are widely preferred over other PCMs [5, 6]. Especially, the organic PCM, paraffin wax, has remained a topic of special attention because of its high latent heat capacity, chemical stability, non-toxicity, and low cost [7]. The effectiveness of PCMs and suitability of paraffin for low to medium temperature applications, particularly building heating/cooling and solar water heating, have been documented by Pasupathi et al. [8]. Nevertheless, its general application is limited by the fact that its thermal conductivity is low, limiting heat transfer rates, as well as moderate subcooling effects. Anitha Selvasofia et al. [9] has also stressed that to make this claim strong, the low thermal conductivity of paraffin requires groundbreaking amendments for

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improving the heat transfer efficiency. In order to address this limitation, researchers have explored several strategies including the incorporation of high thermal conductivity additives like metallic nanoparticles, carbon-based materials, and metal oxides to form composite PCM [10, 11].

Over the past few decades, several studies have investigated the enhancement of thermal performance of PCMs through the incorporation of highly conductive metallic/metal oxide nanoparticles [12, 13]. Saravanakumar et al. [14] reported the preparation of paraffin-based nano-composites with ceramic oxide nanoparticles and observed a marked increase in thermal conductivity and reduced supercooling effect. Similarly, Kant et al. [15] demonstrated that the thermal conductivity of paraffin can be enhanced significantly through the addition of graphene, though at the expense of latent heat.

More recently, the use of metal oxide nanoparticles such as Al_2O_3, TiO_2, ZnO, and MgO has gained popularity due to their moderate cost, environmental safety, and decent thermal conductivity [16, 17]. Huang et al. [18] highlighted the benefits of dispersing alumina nanoparticles into PCMs and noted improved heat transfer and solidification rates. Likewise, Ramya et al. [19] synthesized a paraffin-based PCM with dispersed ZnO nanoparticles and confirmed enhancement in both thermal conductivity and heat storage efficiency.

Recently, magnesia (MgO) nanoparticles have been studied for applications in thermal conductivity enhancement of PCMs because of its unique properties among metal oxides [20]. However, MgO has good thermal conductivity, chemical inertness, thermal stability and compatibility with organic matrix such as paraffin. In addition, MgO is both easily available, environment friendly and cost effective thereby also making it a good candidate for large scale TES systems [21, 22]. Dispersed at nanoscale levels, MgO can greatly enhance thermal conductivity and structural stability of PCMs, and its negative effects on the latent heat storage capacity are minimized.

Despite the substantial progress in this domain, a comprehensive evaluation of nano-MgO dispersed paraffin PCMs across multiple concentrations using consistent preparation and characterization methods remains limited. Most studies have focused on a single concentration or lacked thorough characterization across a spectrum of thermal and morphological properties.

Among all works utilizing MgO, this work distinguishes itself in its systematic method to optimize MgO concentration and provide a road map to achieve tradeoff regarding thermal conductivity and energy density. In this study, nano MgO/paraffin PCMs with concentration of nano MgO dispersed in paraffin of 0, 0.5, 1.0, 1.5, and 2.0 wt.% are developed and characterized. Then, different methods have been used to analyze the synthesized composites. Key objectives of the study include:

- To synthesize nano-MgO/paraffin composites by disbanding 0, 0.5, 1.0, 1.5, and 2.0 mass fractions of nano-MgO in paraffin matrix.
- To investigate chemical compatibility and structural integrity through FTIR (Fourier Transform Infrared Spectroscopy) and FESEM (Field Emission Scanning Electron Microscopy) analysis.
- To analyze the thermal behavior of the nano-PCMs using DSC (Differential Scanning Calorimetry) and TGA (Thermogravimetric Analysis) and assess the impact of nano-MgO loading on phase change temperatures and latent heat values.
- To evaluate the enhancement in thermal conductivity as a function of nano-MgO concentration using TEMPOS thermal properties analyzer.

The outcomes of this study are expected to contribute valuable insights toward the development of high-performance, cost-effective, and scalable thermal energy storage materials suitable for applications in solar energy systems, building heating/ cooling, and electronic thermal management.

MATERIALS AND METHODS

Synthesizing Nano-MgO/PCM

The highly pure commercial grade paraffin wax having melting point of around 58 °C had been sourced from a certified local chemical supplier and used without any further purification. Nanoparticles of magnesium oxide (nano-MgO) having average particle size 30-50 nm was procured from Ultrananotech. The nano-MgO/paraffin composite PCMs were synthesized using a two-step method [23]. It is the method, where nanoparticles are first synthesized and then mechanically mixed into the PCM matrix, is widely adopted due to its simplicity and effectiveness in ensuring uniform dispersion. This approach allows better control over the nanoparticle concentration and reduces the risk of agglomeration, which is critical for maintaining consistent thermal performance over multiple cycles. In this method, the accurately weighed nanoparticles were distributed inside a molten paraffin carefully with the aid of a magnetic stirrer as presented in Figure 1. The stirring action was continued for 90 minutes to confirm the uniform dispersal of the nanoparticles within the PCM matrix [24]. The prepared samples were labelled as shown in Table 1.

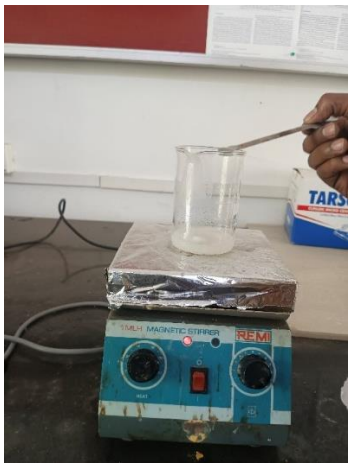

Figure 1. Synthesizing nano-MgO/paraffin PCM using magnetic stirrer.

Table 1. Nano-PCM sample compositions.

Specimen No.	% of nano-MgO	% of paraffin PCM
1	0	100
2	0.5	99.5
3	1.0	99
4	1.5	98.5
5	2.0	98

Characterization of nano-MgO/PCM

Characterization of nano-MgO/PCMs is essential to understanding the impact of nanoparticles on thermal properties. FESEM helps in visualizing the dispersion of nanoparticles and evaluating the microstructure, while thermal conductivity testing quantifies the improvement in heat transfer performance using Transient Plane Source method.

DSC analysis provides insights into the phase transition temperatures and latent heat values. Approximately 5 mg of each sample was sealed in an aluminum pan and scanned under a nitrogen atmosphere at a heating and cooling rate of 5 °C/min over a temperature range. TGA reveals thermal degradation patterns and thermal stability. TGA was conducted under a nitrogen atmosphere. The samples of around 10 mg were heated from room temperature to 450 °C at a rate of 10 °C/min. TGA curves were used to identify degradation onset temperatures and determine the suitability of composites for long-term thermal cycling.

FTIR is employed to confirm the absence of chemical reactions between the nano-MgO and the base PCM, ensuring that the thermal performance is purely physical. The FTIR spectra were recorded in the range of 4000 to 400 cm^{-1} using the ATR method.

RESULTS AND DISCUSSION

The prepared nano-doped PCM samples had been characterized using diverse techniques to assess their characteristics, thermal stability, and chemical stability under the dispersion of nano-MgO particles at varying concentrations. Each measurement was accompanied with three samples to check the consistency and accuracy of the results. Figure 2 displays the FESEM images of the unaltered paraffin and the nano-MgO doped paraffin under 10 KX and 5 KX magnifications, respectively. The unaltered PCM seems to be have the uninterrupted surface layer with uniform granules (Figure 2a). However, the dispersion of nano-MgO caused the diffusion of the particles within the surface layers (Figure 2b). The nanoparticles are looking to be distributed as a uniform network across the entire surface of the paraffin matrix without extensive agglomeration when mixed with paraffin at 1.0% mass fraction. The results assured the provision of appropriate blending action through the magnetic stirrer.

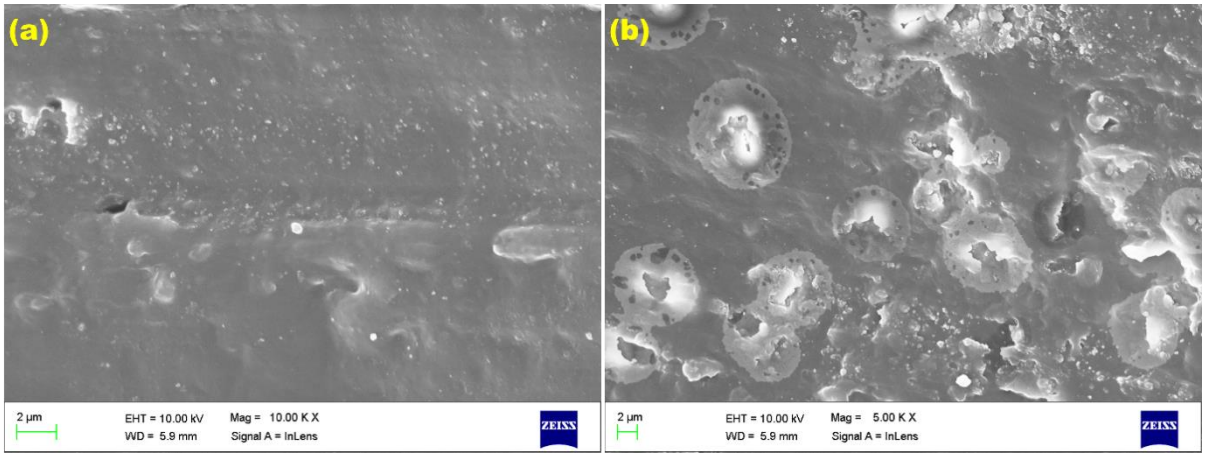

Figure 2. FESEM image of the (a) Pure PCM (b) 1.0% MgO + Paraffin PCM.

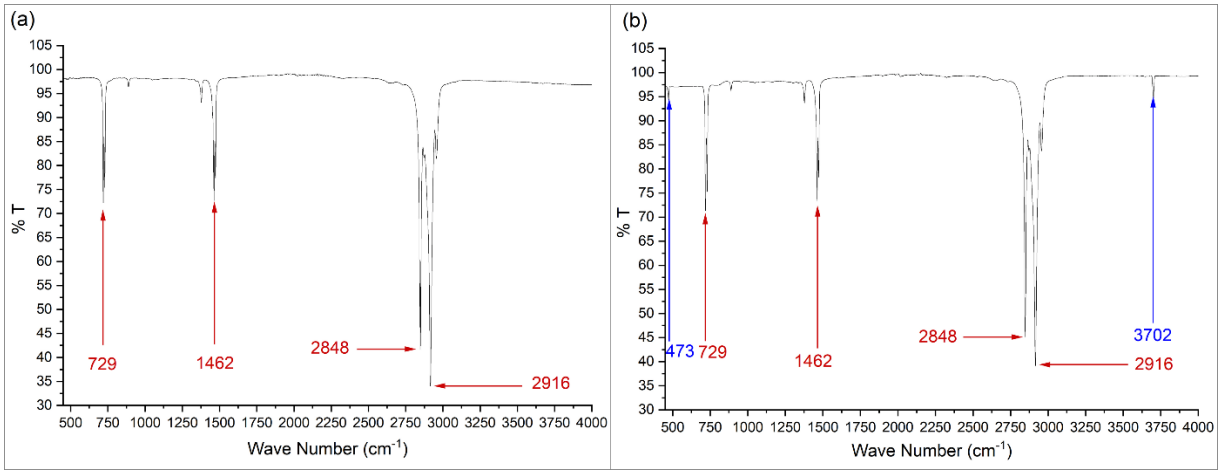

Figure 3. FTIR of the (a) Pure PCM (b) 1.0% MgO + Paraffin PCM.

Figure 3 illustrates the FTIR spectra of the unaltered PCM and the 1.0% mass fraction nano-MgO doped PCM. The unaltered paraffin has illustrated its authentic peaks at the natural wave numbers. Figure 3a shows two sturdy peaks at 2848 cm^{-1} and 2916 cm^{-1}. They can be accredited to the moderately balanced and the sturdily balanced elongating vibrations of carbon-hydrogen molecules, respectively [7, 23]. Further, at 729 cm^{-1} and 1462 cm^{-1}, a couple of shark peaks have been occurred. They highlight mild rocking and mild scissor vibrating nature of the C-C and C-H molecules, respectively [8, 23]. The similar pattern had been recognized in the FTIR spectra of the nano-MgO/PCM sample. In addition, two more weak peaks at 473 cm^{-1} and 3702 cm^{-1} wavenumbers have been identified as displayed in Figure 3b. They signal the existence of magnesium-oxygen molecular vibration and hydroxyl group, respectively [25]. The FTIR results ensured the physical existence and chemical stability of the nano-MgO particles within PCM matrix without disturbing the chemical nature of the paraffin.

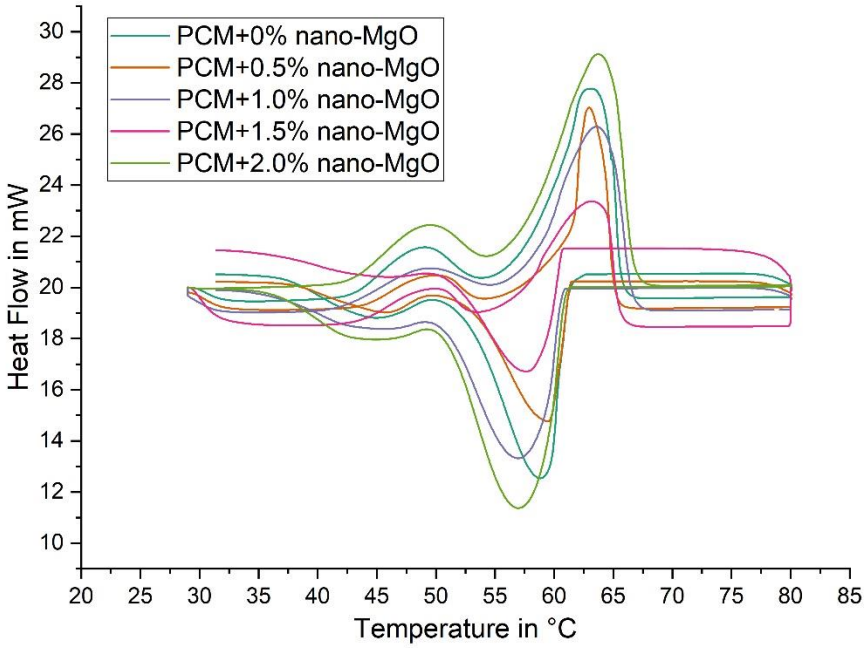

Figure 4. DSC of the nano-MgO/PCM samples.

Figure 4 shows the DSC of the nano-MgO/PCM samples. The peaks and crests have been formed during melting and solidification of the PCM/nano-PCM samples, respectively. During both heating and cooling of the samples, two peaks/crests were formed, among them one seemed to be strong and another one was weak. The weak peaks during the melting process are the evidence of breaking of crystallization structure of the PCM/nano-PCMs, whereas, the larger peaks are evidence of phase transition from solid to liquid with high latent heat of fusion. Similarly, the weak crests and the strong crests, which were formed during the crystallization of PCM/nano-PCM are the indications of crystalline changes and the crystallization processes during the solidification process. The enormous of quantity of thermal energy has to be stored/released during such phase transitions. The fusion and crystallization points, latent heat of fusion and crystallization can be measured with the aid of DSC, and they are presented in Table 2. It can be vividly seen that the fusion temperature of the PCM was gradually decreased with the increment in nano-MgO concentrations till adding 1.0% in paraffin. However, further addition of nano-MgO couldn't support for such reduction in fusion point. Similarly, the crystallization temperature of the PCM was slowly increased with addition of nano-MgO until adding 1.0% in paraffin, then declined. The analogous pattern was found for the latent heat of fusion as well as latent heat of crystallization with the increment in nano-MgO particles in PCM matrix. It can be ascertained that the addition of nano-MgO after 1.0% failed to support the enhancement in thermal characteristics of the PCM, rather it has given the negative impact. The reduction in gap between melting and solidification temperatures of the PCM would efficiently suppress the degree supercooling of the PCM.

Table 2. Properties of nano-PCMs measured through DSC.

Samples	Thermal characteristics			
	Fusion point (°C)	Fusion latent heat (kJ/kg)	Crystallization Point (°C)	Crystallization latent heat (kJ/kg)
PCM + 0% nano-MgO	60	150.5	56.5	143.8
PCM + 0.5% nano-MgO	59.2	147.4	56.9	139.3
PCM + 1.0% nano-MgO	58.4	145.2	57.6	138.4
PCM + 1.5% nano-MgO	58.5	131.6	57.5	128.7
PCM + 2.0% nano-MgO	59.1	112.3	57.0	107.6

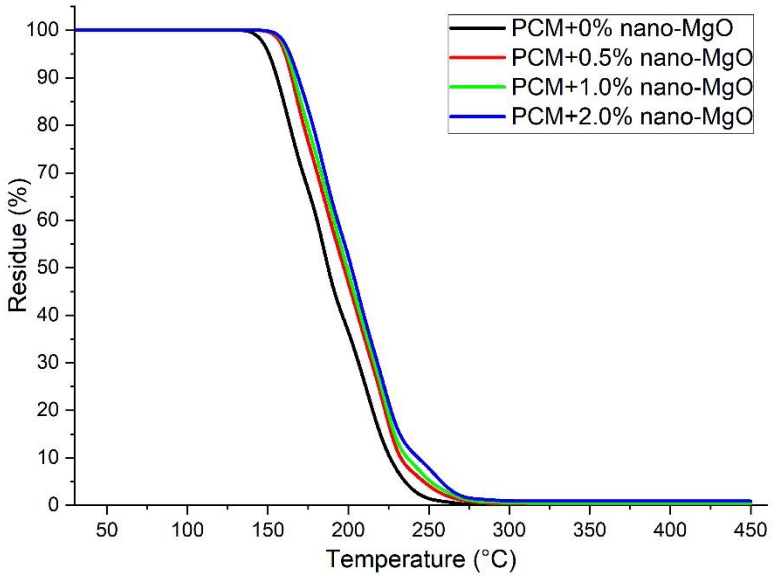

Figure 5. TGA of the nano-MgO/PCM samples.

Figure 5 shows the TGA of the PCM/nano-PCM samples. The TGA curves demonstrate the residual mass of PCM composites with different nano-MgO concentrations throughout a temperature spectrum of 50–450°C. A single-stage breakdown profile was reported for the unmodified PCM (0% nano-MgO). The initial little weight loss below 170°C is attributed to the vaporization of volatile compounds and residual moisture. The principal breakdown transpires between 170°C and 230°C, during which paraffin experiences fast volatilization, resulting in no leftover at 280°C.

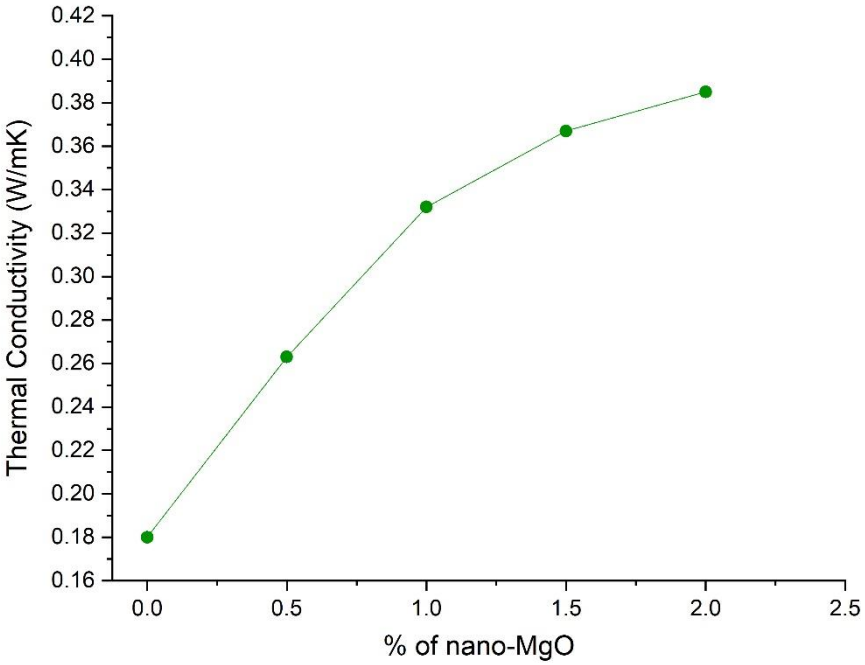

Figure 6. Thermal conductivity of the nano-PCM at increasing mass fraction of nano-MgO.

Conversely, nano-MgO-doped phase change materials demonstrated improved thermal stability. With the increase of nano-MgO content from 0.5% to 2.0%, the residual mass at 450°C consistently increased, attaining 0.9% for the nano-PCM containing 2.0% nano-MgO. This pattern highlights the function of nano-MgO as a thermally stable additive that inhibits paraffin breakdown. The nanoparticles presumably functioned as physical barriers, obstructing the release of volatile breakdown products. Moreover, the uniform distribution of nano-MgO, as verified by FESEM, established a fortified matrix that enhanced heat transfer by mitigating localized thermal deterioration.

The beginning disintegration temperature increased slightly with greater nano-MgO loading, indicating delayed degradation mechanisms. The residual mass at 450°C is closely linked to the nano-MgO concentration, as the inert oxide particles persist during paraffin disintegration. It verifies the effective incorporation of nano-MgO into the PCM without chemical modification, as indicated by FTIR spectra that reveal no new functional groups.

The thermal conductivity of nano-MgO/PCMs was methodically assessed to determine the influence of nanoparticle distribution on heat transfer capability. Figure 6 and Table 3 depict the correlation between nano-MgO quantity and the thermal conductivity of the nano-MgO/PCMs. The unaltered paraffin (0% nano-MgO) demonstrated a standard thermal conductivity of 0.18 W/mK, aligning with the intrinsic property of organic phase change materials (PCMs). The addition of nano-MgO particles markedly improved this characteristic, with thermal conductivity increasing to 0.385 W/mK at a 2.0% loading, representing a notable 113.89% enhancement.

The improvement exhibited an unconventional pattern, with the most significant improvements noted at reduced nanoparticle concentrations. The incorporation of 0.5% nano-MgO enhanced thermal conductivity by 46.11% (0.263 W/mK), whereas a 1.0% loading led to an 84.44% increase (0.332 W/mK). The observed pattern indicates that nano-MgO particles efficiently establish conductive routes inside the paraffin matrix, enhancing phonon transfer and diminishing heat resistance. At elevated loadings (1.5–2.0%), the enhancement rate diminished, with thermal conductivity increasing by 103.89% (0.367 W/mK) and 113.89% (0.385 W/mK), correspondingly. This declining return may arise from the initial clumping of nanoparticles at high concentrations, leading to interfacial gaps and disrupting heat transmission pathways.

The percentage improvements correspond with research on analogous nano-PCMs; however, the findings here exceed the standard values documented for paraffin-based systems. A 113.89% boost at 2.0% loading significantly surpasses the improvements observed in paraffin composites with alternative oxides at similar dosages, underscoring the effectiveness of nano-MgO and the used dispersion method. FTIR research conclusively demonstrated that the increase resulted only from physical interactions, with no evidence of chemical bonding between paraffin and nano-MgO identified.

The results highlighted that the inclusion of nano-MgO particles in the paraffin-based PCM would significantly enhance the thermal storage characteristics of the PCM to a greater extent. However, the inclusion of nanoparticles above 1.0% was not recommended owing to the agglomeration issues at higher mass fractions.

Table 3. Thermal conductivity of nano-MgO/PCMs.

Sample	Thermal conductivity (W/mK)	% enhancement of thermal conductivity (%)
PCM + 0% nano-MgO	0.18	-
PCM + 0.5% nano-MgO	0.263	46.11
PCM + 1.0% nano-MgO	0.332	84.44
PCM + 1.5% nano-MgO	0.367	103.89
PCM + 2.0% nano-MgO	0.385	113.89

CONCLUSION

This research comprehensively evaluated the influence of nano-MgO dispersion on the thermal storage characteristics of paraffin-based phase change materials (PCMs). The key findings are as follows:

- The integration of nano-MgO particles using a two-step process markedly improved thermal conductivity, achieving a 113.89% enhancement at a 2.0% loading, while preserving and chemical stability.
- The uniform dispersion, validated by FESEM, enhanced phonon transport, overcoming paraffin's intrinsic heat transfer barriers.
- Despite higher nanoparticle concentrations slightly diminishing latent heat storage capacity, the 1.0% composite proved to be the optimum composition, with an 84.44% enhancement in thermal conductivity with negligible reductions in energy density.
- The improved thermal stability, demonstrated by TGA, further validates the appropriateness of these composites for use at elevated temperatures.
- Challenges like nanoparticle accumulation at loadings over 2.0% indicate the necessity for future optimization to enhance sustainability.

In summary, nano-MgO/paraffin phase change materials have significant potential for enhancing thermal energy storage technologies, especially in solar thermal systems, where efficient heat transfer and durability are essential.

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