

Preparation and Thermal Properties of Myristic Acid/Hydrophobic Fumed Silica/Hexagonal Boron Nitride Composite Phase Change Material

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To address the issues of leakage and poor thermal conductivity in organic solid-liquid phase change materials, this study prepared a shape-stabilized composite phase change material using myristic acid (MA) as the phase change energy storage matrix, hydrophobic fumed silica (FS) as the porous carrier, and hexagonal boron nitride (BN) as the thermally conductive filler via a solvent-assisted impregnation method. The composite material was characterized by scanning electron microscopy, Fourier transform infrared spectroscopy, X-ray diffraction, and differential scanning calorimetry. The results indicate that the composite with an MA mass fraction of 65 % exhibits both satisfactory shape stability and a relatively high latent heat of phase change. The sample with 2 % BN addition demonstrates the best overall performance, with melting and solidification temperatures of 52.47 °C and 51.89 °C, and melting and solidification latent heats of 115.6 J/g and 110.8 J/g, respectively. The thermal conductivity is increased by 83.6% compared to the composite without BN, and the addition of BN also showed a preliminary effect in reducing the supercooling degree. The composite system shows application potential for low-to-medium temperature solar thermal storage, providing a theoretical basis and technical reference for the development of efficient, low-cost, and scalable phase change energy storage materials.

Keywords: composite phase change material, thermal properties, hydrophobic fumed silica, boron nitride.

1. INTRODUCTION

Energy serves as the lifeblood of the national economy, the cornerstone for safeguarding national security and driving social development. Renewable energy sources are abundant, with green power industries represented by solar and wind energy achieving leapfrog advancements [1]. However, such energy sources face challenges including intermittency and fluctuation, which limit power generation efficiency and complicated flexible regulation. Therefore, there is an urgent need to develop theories and methods for large-scale utilization of renewable energy with high efficiency and low cost [2]. Among various technological approaches, thermal energy storage technology demonstrates considerable application prospects due to its capability for efficient energy harvesting, conversion and storage, as well as its significant advantages in conversion efficiency, operational convenience and cost-effectiveness [3].

Phase change materials (PCMs) stand out in thermal energy storage technology. They are substances capable of efficiently storing and releasing latent heat through phase transitions within a specific temperature range [4]. Such materials are widely used in the field of latent heat storage. Among them, solid-liquid PCMs such as fatty acids [5], fatty alcohols [6], and paraffin waxes [7] are widely applied due to their absence of phase separation, low supercooling degree, and high thermal stability. Immiscible binary phase change materials have also drawn considerable attention. Favakeh et al. reported that the melting interface evolution and compositional distribution in multi-component PCMs significantly affect their thermal charging/discharging

behavior, providing useful insights for optimizing the thermal response of organic phase change materials [8]. Among various fatty acid PCMs, myristic acid (MA) is particularly attractive because its phase change temperature matches the operating range of low-to-medium temperature solar thermal storage systems. Compared with lauric acid [9] and stearic acid [10], its phase change temperature is closer to the common operating temperature window of such systems. Therefore, MA is selected as the phase change matrix in this study to validate the feasibility and superiority of the hydrophobic fumed silica/BN composite system. However, in practical applications, they still face challenges related to phase change leakage and poor thermal conductivity, which limit their further promotion [11]. To address the above issues, several review articles have summarized various enhancement strategies for latent heat storage systems, including porous matrix encapsulation, incorporation of highly thermally conductive fillers, introduction of fins, and the coupling of multiple enhancement approaches [12].

To address the leakage issue, infiltrating phase change materials into porous carriers is an effective strategy. Porous materials adsorb liquid-phase PCMs through capillary force and surface tension, confining them within the pores [13]. This method features simple processing, low cost and suitability for large-scale applications. Currently, porous materials such as cellulose nanofibers [14], silica [15] and expanded perlite [16] have been widely used to prepare composite phase change materials. Among them, hydrophobic fumed silica (FS) shows promising potential due to its unique structural advantages. FS is prepared by surface modification of hydrophilic fumed silica with silane

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or silazane, whereby the surface silanol groups are replaced by non-polar groups, resulting in a high specific surface area and nanoscale pores that can confine liquid PCMs via capillary forces. Meanwhile, the particles form a three-dimensional network through hydrogen bonding or van der Waals forces, further inhibiting flow and leakage. Therefore, impregnating PCMs with FS enables the preparation of cost-effective and shape-stabilized composite phase change materials [17–19]. Nguyen et al. introduced 1-octadecanol (C₁₈OH) into FS to prepare a shape-stabilized composite phase change material with high latent heat. The results demonstrated that the maximum impregnation amount of C₁₈OH in FS reached 75 %, with a latent heat value of 160.3 J/g, and the material exhibited good thermal reliability [20]. Wei et al. employed FS to adsorb a binary eutectic mixture of lauric acid and hexadecanol (LA-HD) to prepare a shape-stabilized composite phase change material. Leakage tests showed that the loading ratio of LA-HD in FS reached 60 % [21]. These studies collectively indicate that FS offers significant advantages as a porous carrier in achieving high loading ratios and shape stability for phase change materials.

To address the issue of poor thermal conductivity, incorporating highly thermally conductive materials is a simple and effective improvement strategy. Currently, utilizing the high thermal conductivity and high-frequency Brownian motion of nanoparticles as fillers to enhance the thermal conductivity of thermal storage materials has become a research hotspot [22, 23]. Hosseinineh et al. found that the melting interface evolution and compositional distribution of multi-component PCMs significantly affect their heat absorption and release behavior, offering new insights for optimizing the thermal response of organic phase change materials [24]. Hexagonal boron nitride (h-BN) possesses a hexagonal layered structure and offers superior corrosion resistance, high thermal conductivity and electrical insulation, making it an ideal thermally conductive filler [25, 26]. Su et al. added 15 wt.% h-BN to an octadecane (ODE)-stearic acid (SA) eutectic system, achieving a solid-state thermal conductivity of 0.3272 W/(m·K), which is 9.7 % higher than that of the pure eutectic material [27]. Fang et al. found that adding 10 wt.% h-BN nanosheets increased the thermal conductivity of paraffin wax by 60 %, with almost no effect on the melting and solidification points, only a slight decrease in the latent heat of fusion [28].

Based on the above discussion, in this study, MA was selected as the phase change matrix, FS as the supporting carrier, and BN as a thermally conductive enhancement filler; the MA/FS/BN composite PCM is prepared via a solvent-assisted impregnation method. Compared with previous studies, the present work emphasizes the following aspects: first, hydrophobic FS is employed to enhance the affinity with non-polar MA, thereby improving the impregnation effect; second, the thermal conductivity enhancement at low BN loadings is highlighted to avoid excessive latent heat loss caused by high filler content; and third, the synergistic effects of BN on both the thermal conductivity and supercooling behavior of the composite are concurrently examined. Through these investigations, this study aims to provide experimental evidence and a

theoretical reference for optimizing the performance of fatty acid-based composite PCMs.

2. EXPERIMENTAL SECTION

2.1. Materials

Myristic acid (C₁₄H₂₈O₂, tetradecanoic acid, AR) was purchased from Sinopharm Chemical Reagent Co., Ltd. Hydrophobic fumed silica (purity 99.8 %) was purchased from Shanghai Macklin Biochemical Technology Co., Ltd., with a BET specific surface area of 230 m²/g and a primary particle size of 7–40 nm, belonging to a nanoporous material. Hexagonal boron nitride (purity ≥ 99.9 %) was purchased from Aladdin Chemical Reagent Co., Ltd.

2.2. Preparation of composite PCMs

First, the calculated amount of MA powder was uniformly dispersed in anhydrous ethanol. Then, FS was added slowly, and the mixture was stirred for another 2 h at the same temperature and rotation speed, followed by ultrasonic dispersion for 0.5 h to allow MA to fully infiltrate the porous network of FS. Subsequently, the mixture was heated at 80 °C to evaporate the ethanol until a white powder was obtained. Finally, the obtained SPCMs were dried in a forced-air drying oven for 24 h to completely remove residual ethanol. After determining the optimal loading ratio, BN was added to enhance the thermal conductivity of the composite materials. The composition and ratio of the samples are listed in Table 1. The experimental procedure is shown in Fig. 1.

Table 1. The composition and ratio of samples

Samples	MA, %	FS, %	BN, %
SPCM1	75	25	–
SPCM2	70	30	–
SPCM3	65	35	–
CPCM0.5	64.5	35	0.5
CPCM1	64	35	1
CPCM2	63	35	2

2.3. Characterization

The materials were characterized using the following methods. Microstructure was observed using scanning electron microscopy (SEM, ZEISS/Sigma560, Germany). Functional groups were analyzed by Fourier transform infrared spectroscopy (FTIR, Nicolet iS50, USA) via the KBr pellet method, with spectra recorded in the wavenumber range of 4000 cm⁻¹ to 400 cm⁻¹ to evaluate the chemical compatibility. Crystal structure was determined by X-ray diffraction (XRD, D8ADVANCE, Germany) over a 2θ range of 5–70°. Phase change properties were characterized using differential scanning calorimetry (DSC, DSC8000, USA). All samples were heated and cooled between 0 °C and 100 °C at a rate of 5 °C/min under a nitrogen atmosphere (flow rate of 10 mL/min). Each sample was tested once; the calorimetric accuracy of the instrument is ±0.2 % and the temperature control precision is ±0.008 °C. Thermal stability was analyzed using thermogravimetric analysis (TGA, TGA4000, PerkinElmer, USA) by heating the samples from 30 °C to 600 °C at a rate of 10 °C/min under a nitrogen atmosphere.

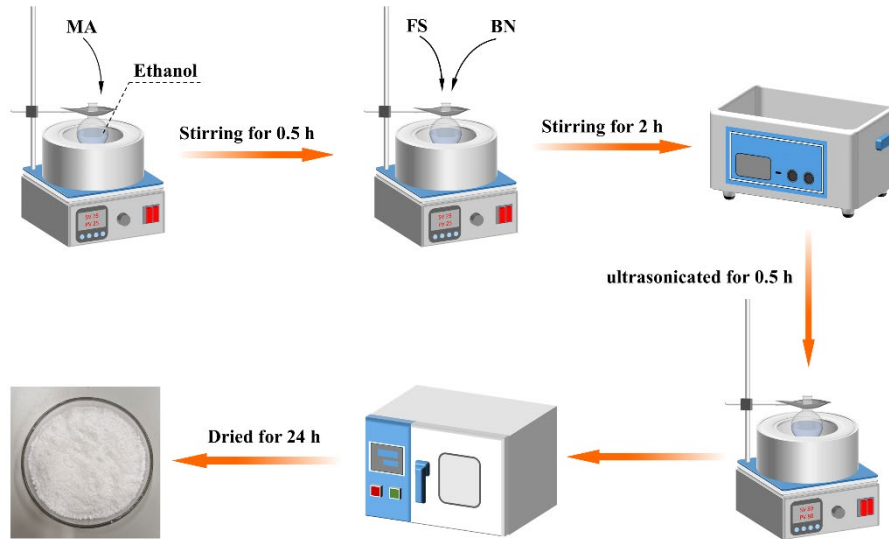


Fig. 1. Preparation process of composite PCM

Each sample was tested once, with a balance sensitivity of 0.1 μ g. Thermal conductivity was measured using a Hot Disk thermal constants analyzer (TPS 2500s). Prior to measurement, the powder sample was pressed into two cylindrical pellets with a diameter of 35 mm and a thickness of approximately 4 mm, with smooth surfaces. The polyimide-coated sensor was placed between the two pellets for measurement. Each sample was measured three times, and the results are expressed as mean $\pm$ standard deviation.

3. RESULTS AND DISCUSSION

3.1. Anti-leakage ability

To evaluate the leakage resistance, pure MA and SPCMs were placed on filter paper and heated in an oven at 65 $^{\circ}$ C for 1 hour, after which leakage traces were observed, as shown in Fig. 2. The corresponding mass loss and loss percentages are listed in Table 2.

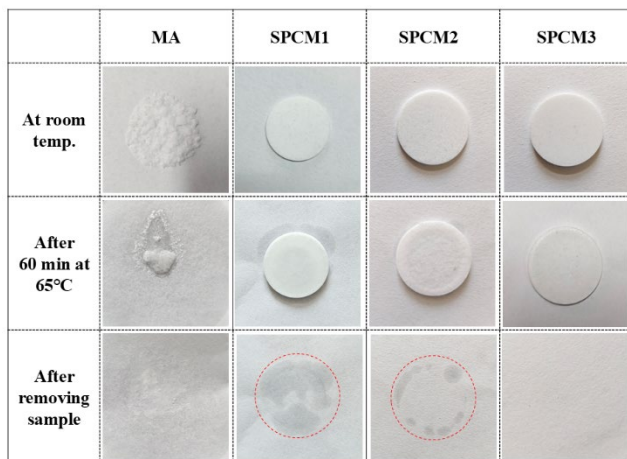


Fig. 2. Images of SPCMs before and after leakage testing

As the FS mass fraction increases, the leakage rate of the composites gradually decreases. SPCM1 exhibits significant mass loss, indicating that the MA content exceeds the loading capacity of FS. With further increase in

FS content, the leakage rate of SPCM2 decreases markedly; SPCM3 shows only a minor mass loss and no obvious leakage trace is observed on the filter paper, confirming good shape stability at this ratio and verifying the feasibility of using FS as a support for encapsulating MA. Based on this, the composite with 35 wt% FS is selected as the matrix, and CPCMs are prepared by adding BN at various mass fractions. Under the same heating conditions, no leakage is observed for all CPCMs, demonstrating stable shape-stabilized performance.

Table 2. The mass loss and loss percentages of SPCMs

Samples	At room temp., g	After 60 min at 65 $^{\circ}$ C, g	Loss in mass, g	Percentage of loss, %
SPCM1	0.5026	0.4105	0.0921	18.32
SPCM2	0.5094	0.4633	0.0461	9.05
SPCM3	0.5013	0.4815	0.0198	3.95

3.2. Morphology and microstructure

Fig. 3 shows the SEM images of FS, BN and the composite PCMs. As shown in Fig. 3 a, FS exhibits a nanoscale agglomerated structure, where particles aggregate through weak interactions to form agglomerates.

FS possesses a high specific surface area and nanoscale pores, which provide abundant capillary adsorption sites. [29]. Combined with the leakage test results, it is considered that the porous structure of FS effectively confines liquid MA through the synergistic effect of capillary force and surface tension. In Fig. 3 b, BN displays a layered agglomerate morphology. When uniformly dispersed in the composite matrix, BN sheets tend to distribute in the gaps between FS aggregates. Such morphology is generally considered beneficial for enhancing thermal transport performance [30]. From Fig. 3 c and d, it can be observed that MA is successfully adsorbed into the porous network of FS. After composite formation, the pores originally formed by the stacking of spherical lica particles are filled with MA, resulting in a dense microstructure. This indicates that, under the synergistic effects of hydrogen bonding, capillary forces and surface tension, MA is uniformly adsorbed and

filled into the pores of FS, effectively suppressing phase change leakage.

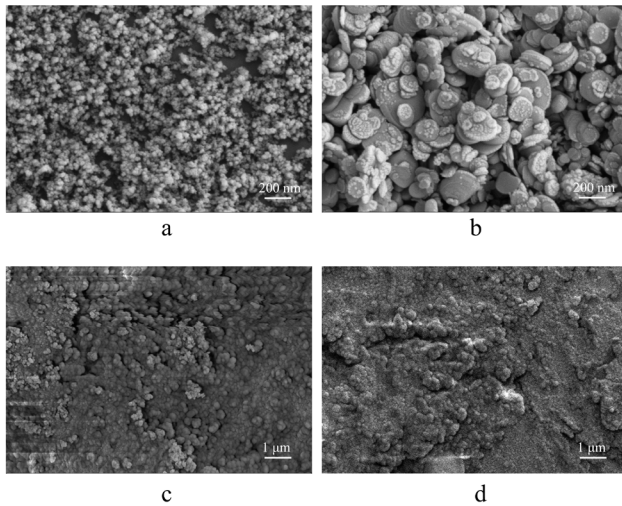


Fig. 3. SEM images: a – FS; b – BN; c – SPCM3; d – CPCM2

3.3. FTIR analysis

Fig. 4 presents the Fourier transform infrared spectra of SPCM3 and CPCM2, along with those of MA, FS and BN for comparison. In the spectrum of FS, the peaks at 471 cm^{-1} , 809 cm^{-1} and 1104 cm^{-1} correspond to the bending, symmetric stretching, and asymmetric stretching vibrations of siloxane groups (Si–O–Si), respectively.

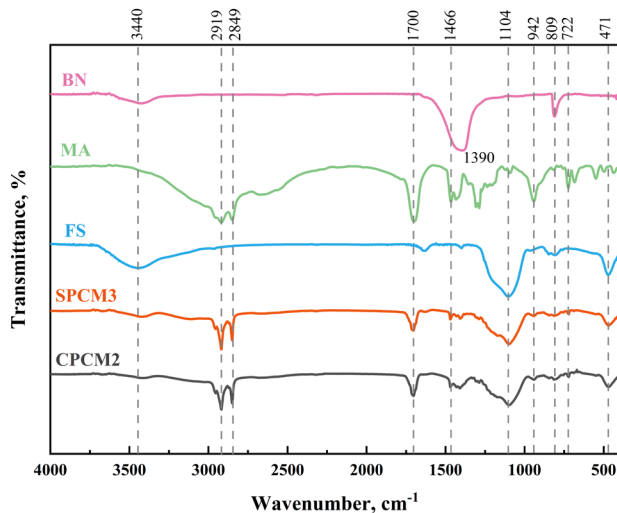


Fig. 4. FTIR spectra of samples

The broad peak at 3440 cm^{-1} is attributed to the mixed stretching vibrations of silanol groups (–Si–OH) and surface-adsorbed water [31]. In the spectrum of MA, the absorption bands at 2919 cm^{-1} and 2849 cm^{-1} arise from the asymmetric and symmetric stretching vibrations of $-\text{CH}_2$ groups; the peak at 1700 cm^{-1} is due to the stretching vibration of C=O groups; the band at 1466 cm^{-1} corresponds to the scissor bending vibration of $-\text{CH}_2$ groups; and the peaks at 942 cm^{-1} and 722 cm^{-1} originate from the out-of-plane rocking vibration of –OH groups and the rocking vibration of $-\text{CH}_2$ groups [32]. In the spectrum of BN, the absorption band near 1390 cm^{-1} is assigned to the transverse optical stretching vibration of sp^2 -hybridized B–N bonds,

while the peak near 809 cm^{-1} corresponds to the transverse optical bending vibration [33]. It is noteworthy that the C=O stretching vibration peak of MA shows no obvious shift after compositing, indicating that no strong chemical bonds form between the carboxyl groups of MA and the FS surface. This further supports the physical adsorption of MA within the FS pores. By comparison, no new characteristic absorption peaks appear in the spectra of the composites; only slight changes in the intensity of the original peaks are observed. It can be concluded that the components exhibit good compatibility with each other, involving only physical interactions without any chemical reaction.

3.4. XRD analysis

Fig. 5 shows the X-ray diffraction patterns of MA, FS, SPCM3 and CPCM2. FS exhibits no obvious characteristic diffraction peaks within the measured range, indicating that it is a typical amorphous structure [34], consistent with the intrinsic nature of fumed silica. MA displays strong characteristic diffraction peaks at $2\theta = 5.6^\circ$, 8.4° , 14° , 21.5° and 24° , corresponding to the interlayer stacking and lateral packing planes of its molecular chains [35], reflecting the well-defined crystalline structure of MA.

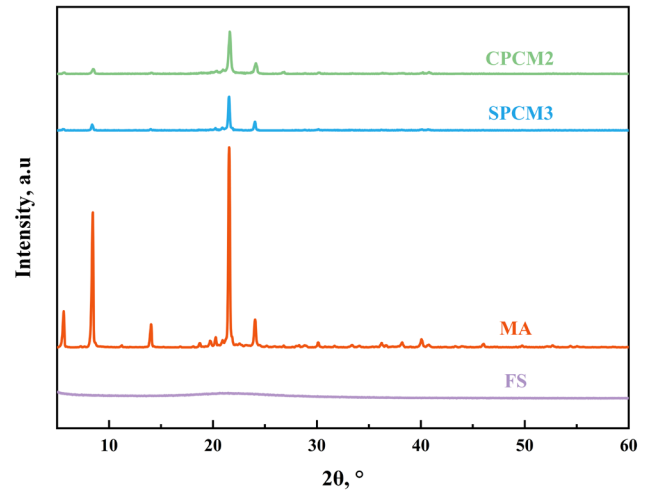


Fig. 5. XRD patterns of samples

The diffraction peak positions of SPCM3 and CPCM2 are essentially identical to those of pure MA, with no new diffraction peaks appearing, but the peak intensities are significantly reduced. This indicates that MA retains its inherent crystalline structure within the FS matrix; however, the introduction of FS partially disrupts the crystalline integrity of MA, causing MA to exist as dispersed microcrystals within the porous network of FS. However, the intensities of the diffraction peaks decrease, which can generally be attributed to reduced crystallite size or decreased crystalline order, possibly due to the spatial confinement effect of the porous FS scaffold. The XRD results further confirm that the composite preparation involves only physical mixing, with no new phases formed.

3.5. Thermal properties analysis

The phase change temperature and latent heat of MA, SPCM3 and CPCMs were measured using DSC. Fig. 6 presents the melting and solidification curves of the samples, respectively. Table 2 lists the melting temperature

(T_m), melting latent heat (ΔH_m), solidification temperature (T_s) and solidification latent heat (ΔH_s) of the samples.

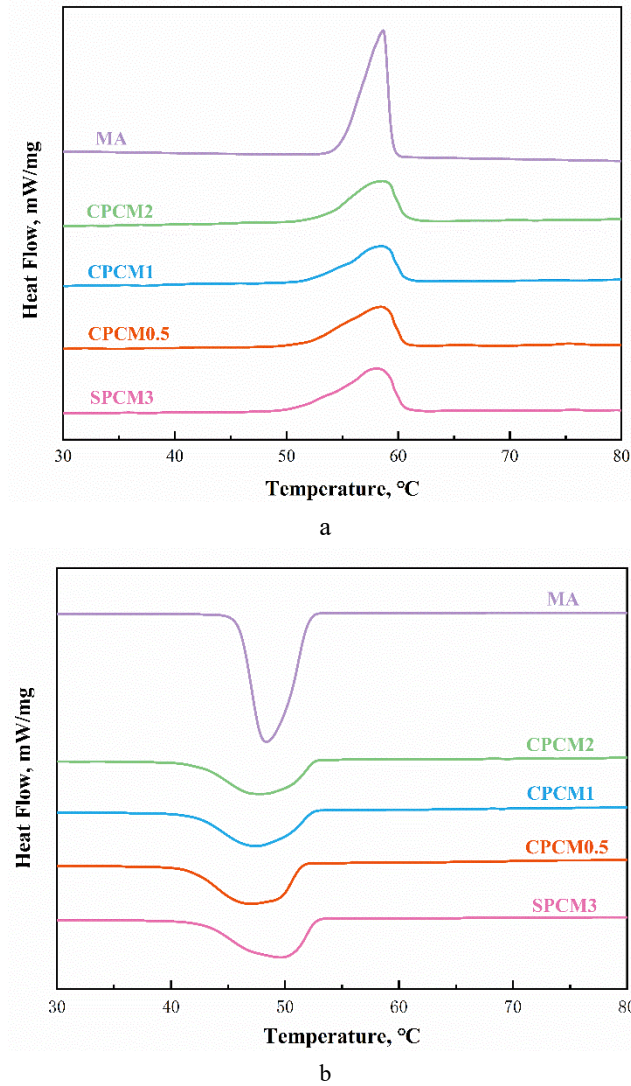


Fig. 6. a–heating DSC curves; b–cooling DSC curves

The melting peak of SPCM3 shifts slightly to a lower temperature and broadens, indicating that the confinement effect of the FS porous skeleton causes a slight decrease in the melting point. The solidification temperature also decreases slightly, and the supercooling degree is marginally reduced. After the introduction of BN, the melting peaks of the CPCMs further shift to lower temperatures, and the peak area decreases with increasing BN content. This is attributed to BN acting as an inert filler that dilutes the effective mass fraction of MA, leading to a reduction in latent heat. The solidification temperature of

the CPCMs increases with higher BN content, while the supercooling degree gradually decreases. CPCM2 exhibits the highest solidification temperature, approximately 51.89 °C, and the supercooling degree decreases to about 0.78 °C. This trend preliminarily suggests that BN sheets may act as heterogeneous nucleation sites in the matrix, contributing to improved solidification performance [36]. All composite samples retain the characteristic peaks of MA, demonstrating that BN does not destroy the thermal storage structure, effectively suppresses supercooling, but reduces the melting latent heat. Among them, CPCM2 achieves a favorable balance between thermal storage capacity and crystallization regulation ability.

To quantitatively evaluate the effects of FS and BN on the thermal storage performance of MA, the energy storage efficiency E and encapsulation efficiency R of the composites were calculated according to Eq. 1 [37] and Eq. 2, [38]

$$E = \frac{\Delta H_{m,c} + \Delta H_{s,c}}{(\Delta H_{m,p} + \Delta H_{s,p})\omega} \times 100\%; \quad (1)$$

$$R = \frac{\Delta H_{m,c}}{\Delta H_{m,p}} \times 100\%, \quad (2)$$

where $\Delta H_{m,c}$ and $\Delta H_{s,c}$ are the melting enthalpy and solidification enthalpy of the composite, $\Delta H_{m,p}$ and $\Delta H_{s,p}$ are those of pure MA, and ω is the mass fraction of MA in the composite. The E values of SPCM3, CPCMO.5, CPCM1, and CPCM2 are 100 %, 99.1 %, 97.7 % and 96.8 %, respectively, and the corresponding R values are 66.5 %, 65.6 %, 64.2 % and 62.4 %. The E values being close to 100 % indicate that MA maintains high crystalline integrity within the FS pores, and the agreement between the R values and the actual MA mass fractions further confirms that the reduction in latent heat is mainly attributed to the dilution effect of the inert fillers.

3.7. Thermal stability

Fig. 7 shows the TGA curves of MA, FS, SPCM3 and CPCMs. Pure MA exhibits a sharp mass loss between 200 and 300 °C, corresponding to its thermal decomposition. Pure FS shows no significant mass loss over the tested temperature range, indicating excellent thermal stability. The thermal decomposition curves of SPCM3 and the CPCMs are similar to that of MA, but the final residue remains stable at approximately 33 %, suggesting that the FS skeleton structure is preserved. The introduction of BN does not alter the thermal decomposition characteristics of MA; it only increases the residue through physical mixing. The designed operating temperature of this composite material is below 100 °C. In the range of 30 to 100 °C, all composites exhibit low mass loss and good thermal stability.

Table 2. Thermal performance parameters of the samples phase transition process

Samples	Heating process		Cooling process	
	T_m , °C	ΔH_m , J/g	T_s , °C	ΔH_s , J/g
MA	54.71	185.3	52.58	185.9
SPCM3	53.56	123.2	52.37	118.5
CPCMO.5	53.08	121.6	51.20	115.6
CPCM1	52.78	118.9	51.64	113.2
CPCM2	52.67	115.6	51.89	110.8

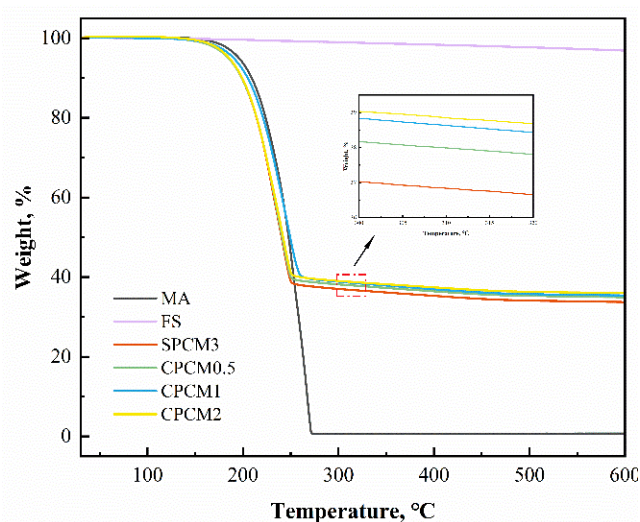


Fig. 7. TGA curves for samples

3.7. Thermal conductivity

Fig. 8 presents the thermal conductivity of MA, SPCM3, and CPCMs. Each data point represents the mean of three repeated measurements, and the error bars indicate the standard deviation. The thermal conductivity of MA is 0.275 W/(m·K).

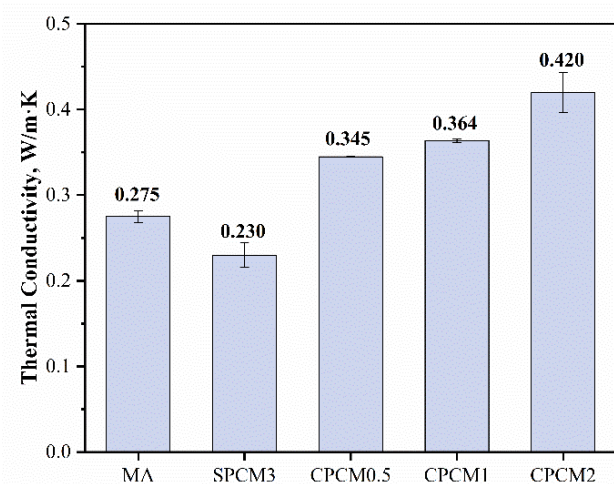


Fig. 8. The thermal conductivity of samples

The thermal conductivity of SPCM3 decreases, which is attributed to the introduction of air gaps within the porous structure of FS, increasing thermal resistance. After the incorporation of BN, the thermal conductivity of the composite system gradually increases with rising BN content. Compared with SPCM3, the thermal conductivity of CPCM2 has increased by 83.6 %. Although the thermal conductivity of the composite remains lower than that of common metals, in practical solar thermal storage systems, the PCM is typically used in conjunction with highly thermally conductive metal heat exchange structures; therefore, the relative enhancement in thermal conductivity achieved at low BN loadings still holds certain engineering reference value. In this study, the BN content was controlled within the range of 0.5 % to 2 % to avoid excessive dilution of the latent heat by high filler loadings. The experimental results indicate that, within the investigated range, the

composite with 2 % BN exhibits the best overall performance.

4. CONCLUSIONS

In this study, a shape-stabilized composite phase change material of MA/FS/BN was prepared via a solvent-assisted impregnation method. Leakage tests showed that the composite with 65 % MA loading possessed good shape stability while retaining a relatively high latent heat. After the addition of BN, CPCM2 exhibited the optimal performance, with melting and solidification temperatures of 52.47 °C and 51.89 °C, melting and solidification latent heats of 115.6 J/g and 110.8 J/g, respectively. Compared with the composite PCM without BN (CPCM3), the thermal conductivity of CPCM2 increased by 83.6 %, and the addition of BN also preliminarily indicated a reduction in supercooling. FTIR and XRD analyses indicated that the components were physically blended with good chemical compatibility. The composite system shows application potential for low-to-medium temperature solar thermal storage, providing a theoretical basis and technical reference for the development of efficient, low-cost, and scalable phase change energy storage materials. In addition, this study examined BN loadings in the range of 0–2 wt.%; the balance between thermal conductivity enhancement and latent heat retention at higher BN contents remains to be further clarified in future work. The quantitative effects of the FS confinement on MA crystal size and crystallinity, as well as the long-term thermal cycling reliability of the composite, will be systematically investigated in subsequent studies through BET analysis, Rietveld refinement of XRD data, and accelerated aging tests.

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