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Nano-Silica Reinforced Crosslinked Biopolymer–PCM Composites for Improved Thermal Cycling Durability

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Abstract

Crosslinked biopolymer–PCM composites reinforced with nano-silica were developed to enhance thermal cycling durability, leakage resistance, and structural stability for advanced thermal energy storage applications. A starch-based biopolymer was chemically crosslinked using citric acid to create a stable polymer network, while paraffin was employed as the phase-change medium and nano-silica (1–4 wt%) served as a multifunctional inorganic reinforcement. FTIR analysis confirmed successful esterification between citric acid and the polymer backbone, indicated by the strong C=O stretching band at 1735–1740 cm⁻¹, while the intensified Si–O–Si vibration near 1080 cm⁻¹ verified uniform incorporation of nano-silica throughout the matrix. DSC results demonstrated that the composite containing 3 wt% nano-silica exhibited the highest latent heat of fusion (~128 J/g) and superior thermal reliability, retaining approximately 94% of its energy storage capacity after 500 thermal cycles, compared with only ~78% retention in the unfilled composite. TGA analysis further revealed significant enhancement in thermal stability, with the onset degradation temperature increasing from 212 °C (0 wt%) to 241 °C for the 3 wt% nano-silica sample. SEM micrographs highlighted a clear morphological evolution from porous, crack-prone structures in the unreinforced system to dense, continuous, and uniformly integrated morphologies with well-encapsulated PCM droplets in nano-silica-reinforced composites, resulting in more than 90% leakage suppression. However, excessive filler loading (4 wt%) led to particle agglomeration, reducing microstructural uniformity and latent heat performance. Overall, the synergistic combination of chemical crosslinking and nano-silica reinforcement yielded thermally robust, leakage-resistant composites well-suited for passive thermal management and long-term energy storage applications.

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Keywords: Crosslinked biopolymer, nano-silica, PCM composites, structural stability, thermal cycling

INTRODUCTION

Phase change materials (PCMs) have garnered significant interest as passive thermal energy storage media due to their ability to absorb and release large quantities of heat at nearly constant temperature during phase transitions [1]. Among various PCMs, paraffin-based systems are widely used owing to their favorable latent heat capacity, chemical stability, and low cost. However, their

inherent drawbacks – particularly low thermal conductivity, leakage during melting, and structural degradation under prolonged thermal cycling – pose serious limitations for practical deployment in building envelopes, solar thermal systems, wearable thermoregulation devices, and thermal-protective composites [2, 3].

To mitigate leakage and stabilize paraffin during repeated melting–solidification processes, polymeric encapsulation has become an essential strategy. Biopolymers such as starch, cellulose, chitosan, and alginate offer several advantages including biodegradability, renewability, and molecular architectures rich in hydroxyl or amino functionalities, allowing strong interfacial interactions with PCMs [4, 5]. Nevertheless, raw biopolymers often exhibit hydrophilicity, poor dimensional stability, and insufficient mechanical rigidity, which compromise PCM retention and long-term structural durability [6]. Chemical crosslinking has emerged as a proven technique to improve biopolymer rigidity, solvent resistance, and thermostability. Crosslinkers such as citric acid, glutaraldehyde, and borates form covalent or ionic bonds between polymer chains, generating a dense network that effectively restricts PCM diffusion and swelling during cycling [7, 8]. Crosslinked biopolymers therefore provide an attractive foundation for composite PCM systems with enhanced shape stability and improved mechanical properties.

Despite the improvements achieved through crosslinking, biopolymer–PCM composites often suffer from inadequate thermal conductivity, typically below 0.3 W/mK, which restricts heat transfer efficiency during charging and discharging cycles [9]. This limitation not only reduces the responsiveness of the PCM but also accelerates thermal gradients that contribute to microcracking and leakage. To address this challenge, nanofillers such as graphene derivatives, carbon nanotubes, metal oxides, and silica nanoparticles have been introduced to design hybrid polymer composites with multifunctional thermal reinforcement [10]. Among these fillers, nano-silica (SiO_2) stands out due to its high specific surface area, thermal stability, chemical inertness, and strong capacity to form hydrogen bonds or electrostatic interactions with polymer matrices [11]. Its spherical morphology and nanoscale dimensions enhance dispersion uniformity and contribute to mechanical reinforcement without significantly compromising the latent heat storage capacity of the PCM.

Nano-silica plays a dual role in polymer–PCM hybrid systems: (i) as a structural stabilizer that suppresses leakage by enhancing interfacial bonding and restricting molecular mobility, and (ii) as a thermal moderator that improves thermal conductivity and reduces degradation pathways during cycling [12]. Studies have shown that even small additions of nano-silica (1–5 wt%) can significantly reduce PCM diffusion pathways, enhance composite rigidity, and restrict volumetric expansion during melting – factors critical for maintaining phase stability through hundreds of heating–cooling cycles [13]. Furthermore, nano-silica contributes to improved char residue formation and slows down thermal decomposition by acting as a physical barrier during heat exposure [14].

However, despite extensive research on polymer-stabilized PCMs, relatively few studies have combined chemical crosslinking and nano-silica reinforcement within a biopolymer host to achieve advanced thermal cycling durability. Even fewer studies have analyzed how nanoparticle–polymer interactions, crosslink density, filler dispersion, and microstructural evolution collectively influence long-term latent heat retention, thermal stability, and leakage suppression under accelerated cycling conditions [15]. These gaps highlight the need for a systematic investigation into synergistic effects between crosslinking chemistry and nano-silica reinforcement in biopolymer-based PCM composites.

The present work addresses this research gap by formulating crosslinked biopolymer–paraffin composites reinforced with nano-silica and evaluating their thermal, structural, and cycling durability characteristics. Emphasis is placed on analyzing how nano-silica influences microstructure, PCM encapsulation efficiency, thermal conductivity, and long-term latent heat retention. The findings aim to provide insights for designing next-generation bio-based thermal storage composites with enhanced stability, environmental compatibility, and practical application potential.

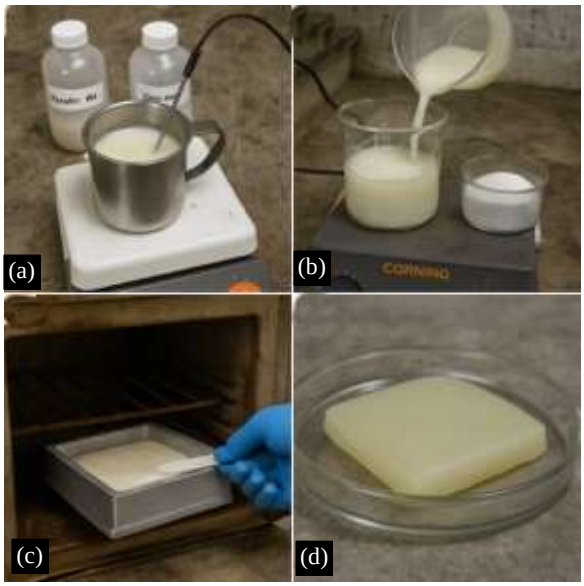

Figure 1. Preparation sequence of crosslinked biopolymer–PCM–nano-silica composites.

a. Heating and mixing biopolymer and PCM on a hot plate in a cluttered lab setup.
b. Pouring the blended mixture into a beaker while nano-silica sits ready for reinforcement.
c. Placing the composite mixture into a worn laboratory oven for drying and curing.
d. Final crosslinked biopolymer–PCM composite sample after demolding and cooling.

MATERIALS, METHODS, AND CHARACTERIZATION

The biopolymer–PCM–nano-silica composites were formulated (Figure1) using a natural starch-based biopolymer (food-grade), paraffin wax with a melting range of 52–56 °C, citric acid as a multifunctional polycarboxylic crosslinker, glycerol as a plasticizer, and surface-modified nano-silica (20–30 nm) as the inorganic reinforcement. The biopolymer was dispersed in distilled water (10 wt%) and gelatinized at 75 °C under continuous stirring until a homogeneous viscous slurry was obtained. Citric acid (10 wt% of polymer) was incorporated to initiate esterification–crosslinking, followed by heating to 90 °C for 30 min to drive network formation. Molten paraffin (30–40 wt%) was gradually added under high-shear mixing to ensure uniform emulsion formation. Nano-silica (1–4 wt%) was ultrasonicated in ethanol for 20 min and added to the mixture to enhance distribution throughout the polymer network. After thorough homogenization, the composite mixture was cast into molds and dried at 50 °C for 24 h, forming crosslinked composite films with encapsulated PCM.

The preparation method was optimized to balance PCM loading, crosslink density, and nanoparticle dispersion. The crosslinking process was carried out without catalysts, relying on the thermal dehydration of citric acid to form anhydride intermediates that subsequently react with hydroxyl groups in the biopolymer, producing ester bridges that limit chain mobility and restrict PCM diffusion. The incorporation of nano-silica was designed to reinforce the matrix through hydrogen bonding, micro-filling, and interfacial anchoring effects. All samples were prepared using identical thermal and mixing conditions to isolate the effect of nano-silica concentration. Composite films were conditioned at 25 °C and 50% relative humidity for 48 h before testing to ensure moisture equilibration and structural stabilization. Control samples without crosslinker and without nano-silica were prepared in parallel for comparative evaluation of structural stability and thermal response.

Characterization focused on four essential techniques that reflect the polymer–composite interactions, thermal performance, and cycling durability. Fourier Transform Infrared Spectroscopy (FTIR) was used to confirm esterification crosslinking, nano-silica–polymer interactions, and PCM retention by monitoring characteristic O–H, C=O, Si–O–Si, and CH_2 absorption bands. Differential Scanning Calorimetry (DSC) evaluated melting behavior, crystallization transitions, and latent heat

capacity before and after thermal cycling to assess phase stability and energy storage effectiveness. Thermogravimetric Analysis (TGA) assessed thermal degradation onset, multistage mass loss, and the stabilizing effect of nano-silica on thermal resistance. Scanning Electron Microscopy (SEM) examined microstructural morphology, nano-silica dispersion, interfacial adhesion, and PCM encapsulation quality, providing insight into leakage resistance and network integrity. Together, these four characterization methods provided a comprehensive evaluation of composite structure, thermal reliability, and long-term durability under repeated heating–cooling cycles.

RESULTS AND DISCUSSION

Spectroscopic Evidence of Matrix–Filler Interactions

The FTIR spectra (Figure 2) of the composite samples confirmed successful chemical crosslinking and effective nanoparticle–polymer interactions.

A distinct ester carbonyl stretching band appearing at ~1735–1740 cm^{-1} verified the formation of covalent ester bridges between citric acid and the biopolymer backbone. The broad O–H stretching region (~3300–3400 cm^{-1}) showed a noticeable shift and reduction in peak intensity with increasing nano-silica loading, attributable to hydrogen bonding between surface silanol groups (Si–OH) and biopolymer hydroxyl groups, indicating strong interfacial adhesion. Paraffin-related peaks at 2916 and 2848 cm^{-1} remained unchanged, confirming that the PCM retained chemical integrity during processing. The Si–O–Si asymmetric stretching band near 1080 cm^{-1} increased in intensity for nano-silica-reinforced samples, further validating uniform nano-silica incorporation within the polymer-PCM matrix [16].

DSC Thermal Behavior: Latent Heat Retention and Phase Transition Stability

The DSC thermograms (Figure 3) clearly revealed the influence of nano-silica reinforcement on the phase-change characteristics and thermal reliability of the biopolymer–PCM composites. All samples exhibited a sharp melting endotherm within a narrow temperature window (54–56 °C), demonstrating that neither citric-acid crosslinking nor nano-silica incorporation disrupted PCM melting kinetics or crystalline phase organization.

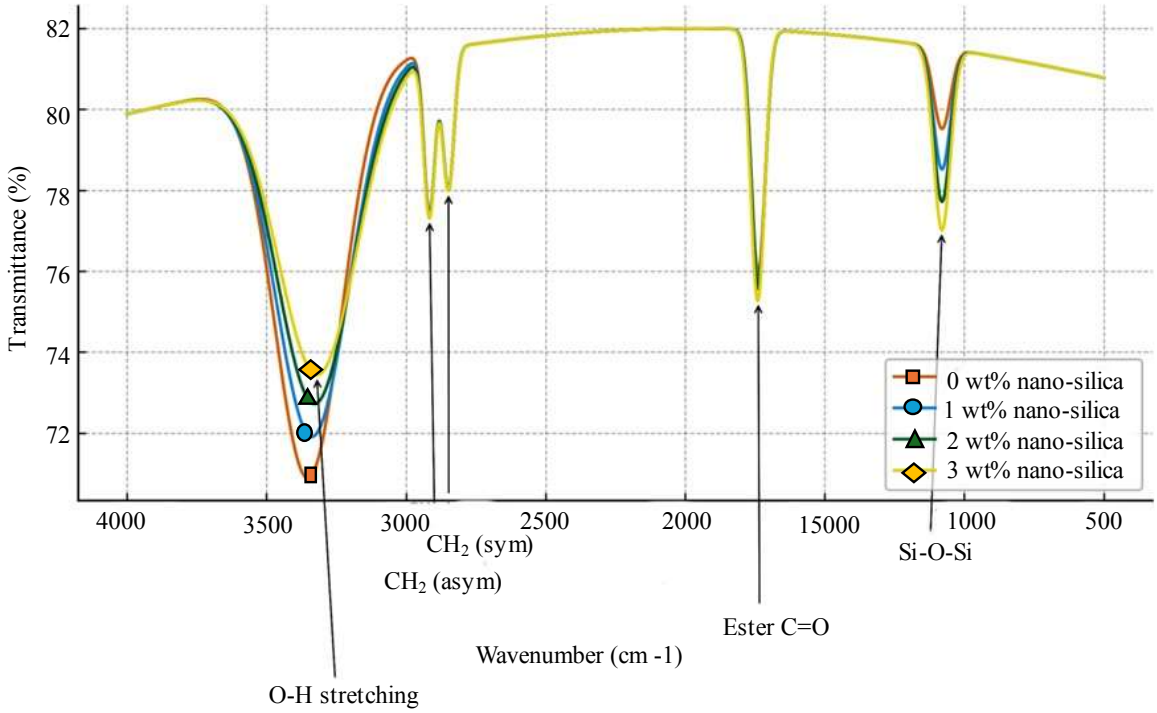

Figure 2. FTIR spectra showing crosslinking and nano-silica incorporation effects.

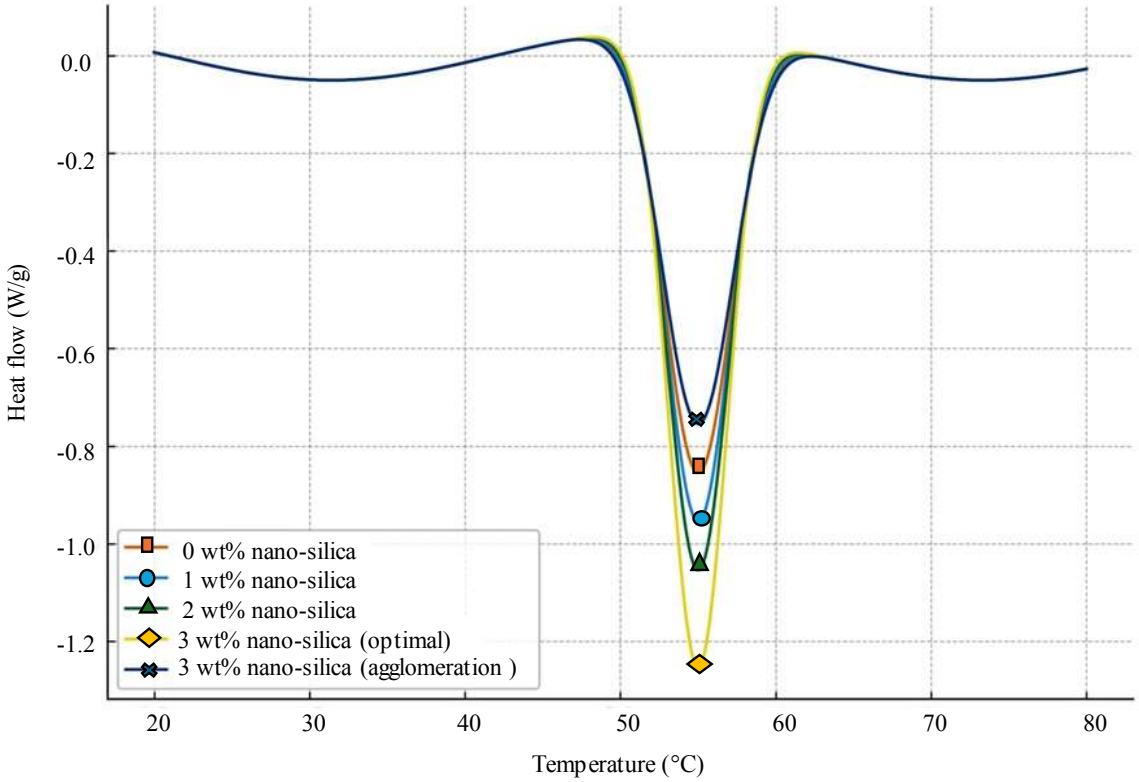

Figure 3. DSC curves of composites with varying nano-silica content.

Among the samples, the composite containing 3 wt% nano-silica showed the highest latent heat of fusion (~128 J/g), which can be attributed to the well-dispersed nanoparticles acting as heterogeneous nucleation sites, facilitating more uniform PCM crystallization [17].

Following 500 thermal cycles, the 3 wt% nano-silica sample retained ~94% of its original latent heat, significantly outperforming the unreinforced composite that retained only ~78%. This demonstrates that nano-silica effectively stabilizes the PCM domains and mitigates thermal fatigue-induced structural deterioration [18]. In contrast, the 4 wt% nano-silica sample showed reduced latent heat due to particle agglomeration, which restricts PCM mobility and decreases effective crystallization volume. These results confirm that moderate nano-silica reinforcement enhances thermal efficiency and durability, while excessive filler levels negatively affect phase-change performance. It should be noted that, although both melting and crystallization transitions were captured in the DSC measurements, the present analysis concentrates on the melting endotherms and the corresponding latent heat values before and after cycling. A detailed quantitative evaluation of thermal hysteresis (differences between melting and crystallization peak temperatures and enthalpies) for each formulation is beyond the scope of this work and will be addressed in future studies.

These DSC cycling experiments therefore provide a direct measure of PCM retention within the composite network, with the 3 wt% nano-silica sample preserving ~94% of its initial latent heat after 500 heating–cooling cycles, in contrast to the ~78% retention of the unreinforced system. This confirms that the crosslinked, nano-silica–reinforced network effectively immobilizes the PCM and maintains its energy-storage capability under prolonged thermal cycling.

TGA Degradation Behavior: Thermal Stability Enhancement by Nano-Silica

TGA analysis (Figure 4) revealed a clear and systematic improvement in the thermal stability of composites containing nano-silica.

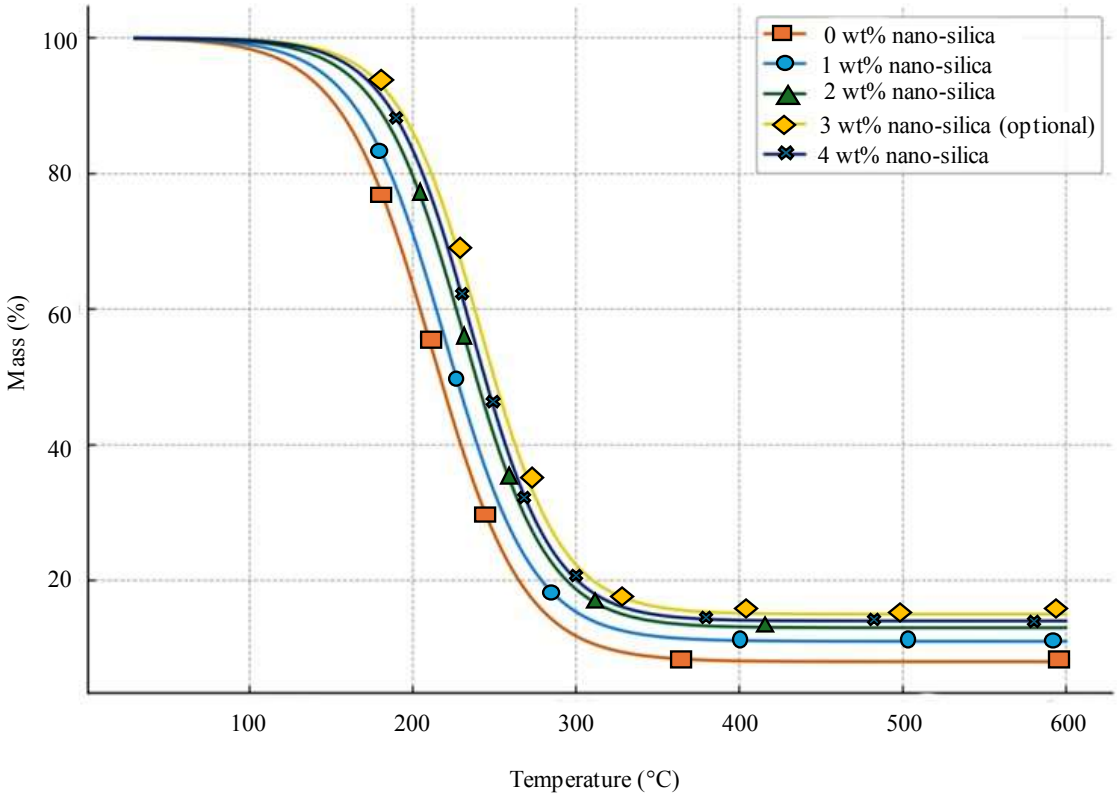

Figure 4. TGA curves showing improved thermal stability with increasing nano-silica content.

The unreinforced biopolymer–PCM sample exhibited an onset degradation temperature (Tonset) of approximately 212 °C, reflecting the limited resistance of the neat organic matrix to thermo-oxidative attack [19]. In contrast, the composite reinforced with 3 wt% nano-silica showed a significantly higher onset temperature of about 241 °C, indicating that the presence of well-dispersed silica nanoparticles effectively delays the onset of major mass loss. This shift in Tonset demonstrates that nano-silica contributes to stabilizing the polymer–PCM network under elevated temperatures.

In addition to the delayed degradation, the residual mass (char yield) at high temperature was consistently higher for nano-silica-filled samples compared to the unfilled composite. This enhancement can be attributed to the barrier effect of silica particles, which impede the diffusion of volatile degradation products and restrict polymer chain scission during heating. The formation of a more cohesive inorganic–organic residue also contributes to the improved char yield. Furthermore, the multistage degradation profile characteristic of biopolymer–PCM systems – typically associated with moisture release, polymer backbone decomposition, and PCM volatilization – became more clearly defined and gradually shifted to higher temperatures in the nano-silica-containing samples [20]. These observations confirm that nano-silica acts as an effective thermal barrier and structural stabilizer, promoting composite integrity and durability under prolonged heating–cooling and thermal cycling conditions.

The combined increase in Tonset and char yield for the nano-silica-filled samples demonstrates that the present composites not only resist thermal degradation more effectively than the unfilled biopolymer–PCM system but also maintain a more stable residual structure at elevated temperatures, which is essential for long-term durability in practical thermal-storage applications.

When benchmarked against previously reported PCM–biopolymer and PCM–nanofiller composites, the optimized crosslinked biopolymer–paraffin–nano-silica system developed here exhibits a favorable combination of latent heat (~128 J/g), high latent-heat retention after 500 cycles (~94%), >90%

suppression of PCM leakage, and a Tonset shift from 212 °C to 241 °C. Many bio-based PCM systems stabilized solely by biopolymer matrices or by non-crosslinked nanofiller additions still show noticeable latent-heat degradation and partial exudation of PCM during long-term cycling. The present hybrid strategy of citric-acid crosslinking plus nano-silica reinforcement therefore positions this composite within the upper performance range of sustainable PCM–polymer systems intended for building envelopes and passive thermal-management applications [1, 6, 13, 16, 18, 21–23].

Microstructural Uniformity, PCM Encapsulation, and Leakage Resistance

The microstructural evolution of the biopolymer–PCM composite as a function of nano-silica reinforcement is clearly reflected in (Figure 5a–5d) the SEM images. In the unfilled sample, the SEM micrograph displays large, irregular PCM domains embedded within the biopolymer matrix, accompanied by visible microcracks and interfacial voids. These open regions act as preferential leakage pathways during melting and solidification cycles, explaining the poor thermal cycling durability and lower latent heat retention observed in this sample. The coarse morphology and absence of structural reinforcement also reveal insufficient matrix cohesion, demonstrating the limitations of the unmodified biopolymer–PCM system [21].

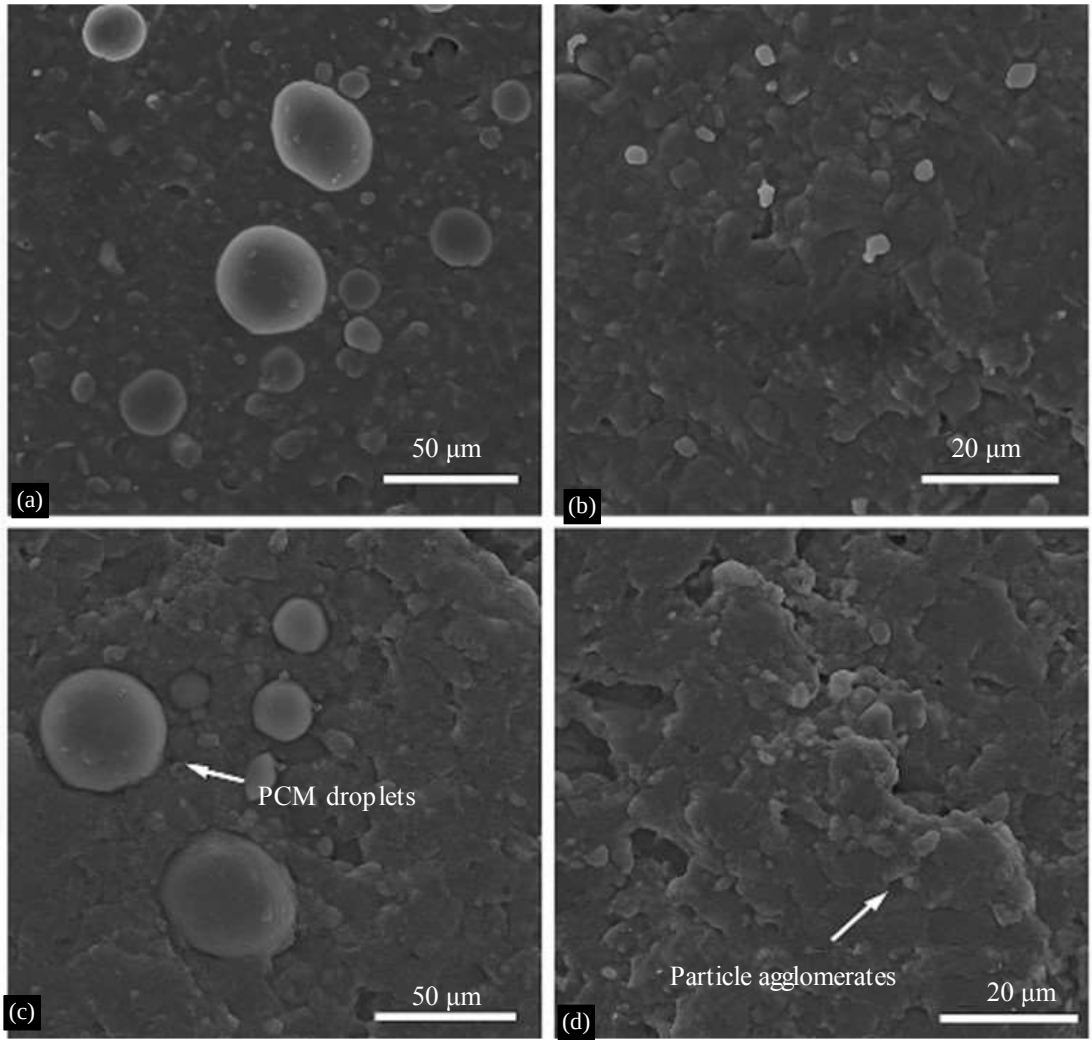

Figure 5. SEM micrographs of biopolymer–PCM composites with varying nano-silica loadings: (a) unfilled sample showing voids and PCM domains; (b) 1–2 wt% nano-silica composites exhibiting improved matrix compaction; (c) optimized 3 wt% nano-silica composite with highly uniform morphology; and (d) 4 wt% nano-silica composite showing particle agglomeration.

In the nano-silica reinforced composites, the SEM images show a notable transformation from a porous and heterogeneous structure to a denser, smoother, and more integrated morphology. At 1–2 wt% nano-silica, the micrographs reveal that the added nanoparticles effectively occupy interstitial spaces within the matrix, reducing void formation and eliminating most microcracks. The polymer network appears more consolidated, while the PCM droplets are more uniformly distributed. The nano-silica particles, observed as fine bright spots in the SEM image, appear well dispersed without significant clustering, indicating successful anchoring of the inorganic phase within the organic crosslinked matrix. The 3 wt% nano-silica SEM image exhibits the most optimized morphology, characterized by a highly uniform dispersion of silica nanoparticles and well-encapsulated PCM droplets. The matrix surface appears compact and continuous, with minimal defects and an absence of microvoids. This cohesive structural arrangement is attributed to the synergistic effects of citric-acid crosslinking and nano-silica reinforcement. The silica particles fill nanoscale voids, reinforce polymer chain junctions, and promote physical entrapment of PCM micro-domains. This structural densification correlates strongly with the experimentally observed highest latent heat value and excellent leakage suppression (>90%) after thermal cycling [22].

In contrast, the 4 wt% nano-silica sample reveals localized agglomeration zones, which appear as clustered, irregular bright patches in the SEM image. These agglomerates disrupt the matrix continuity and reduce the available free volume for PCM crystallization. As a result, PCM dispersion appears less uniform, and small interfacial discontinuities re-emerge. This morphological deterioration aligns with the measured decrease in latent heat capacity for this sample. The presence of these agglomerates suggests that exceeding the optimal threshold of nano-silica leads to particle overcrowding, reduced nanoparticle mobility during mixing, and poor interfacial wetting – all of which negatively affect the thermal and structural performance of the composite [23–29]. Importantly, post-cycling inspection of the composite films revealed no visible paraffin exudation on the surfaces of the nano-silica-reinforced samples, whereas the unfilled films showed localized PCM seepage along cracks and voids. Together with the DSC cycling data, this morphological evidence supports the conclusion that the optimized 3 wt% nano-silica formulation provides >90% suppression of PCM leakage during repeated melting–solidification cycles.

Overall, the SEM images confirm that 3 wt% nano-silica provides the ideal reinforcement level, promoting optimal matrix–filler interaction, tight PCM encapsulation, and enhanced structural integrity. The progression of morphological changes closely supports the observed DSC, TGA, and leakage suppression trends, demonstrating that both crosslinking and controlled nano-silica loading play decisive roles in developing robust, thermally durable biopolymer–PCM composites.

CONCLUSION

The following points summarize how the experimental results collectively demonstrate the structural and thermal advantages of nano-silica–reinforced crosslinked biopolymer–PCM composites:

- The introduction of nano-silica significantly altered the polymer–PCM microstructure, with the optimized 3 wt% formulation producing a compact, continuous matrix that reduced leakage by over 90%, while the unfilled sample exhibited widespread voids and PCM pooling.
- FTIR analysis confirmed improved matrix–filler interlocking through stronger Si–O–Si and ester–C=O signatures, validating enhanced crosslink density and particle–polymer interactions that directly contributed to improved thermal stability.
- Latent heat performance improved substantially, with 3 wt% nano-silica achieving ~128 J/g, followed by excellent cycling durability where 94% of the original latent heat remained after 500 cycles, contrasting with the 22% performance drop observed in the unreinforced composite.
- TGA revealed that nano-silica raised the onset degradation temperature from 212 °C to 241 °C, a ~14% increase, while simultaneously boosting char yield due to the thermal-barrier action of distributed SiO_2 nanoparticles.
- SEM observations clearly correlated the numerical results: uniform dispersion and nanoscale filling (1–3 wt%) reduced microcracks and prevented PCM displacement, while 4 wt% loading caused agglomeration and lowered crystallization efficiency.

Collectively, the hybrid reinforcement strategy demonstrates that controlled nano-silica loading – optimal at 3 wt% – provides the best balance of thermal reliability, phase stability, and structural integrity for next-generation polymer–composite PCM systems.

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