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Polymeric Hybrid Systems Engineered with Multiple-Stage Energy Absorption for Temperature Control

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Abstract

Polymer–composite based phase change systems offer an effective strategy for adaptive thermal management; however, most existing systems exhibit single-stage latent heat transitions, limiting their applicability in environments with fluctuating thermal loads. In this study, a thermoplastic polyurethane (TPU) polymer matrix was reinforced with two microencapsulated paraffin-based phase change materials (PCM-A and PCM-B) to achieve multi-level latent heat storage behavior. The fabricated TPU/PCM-A10–B20 polymer–composite demonstrated two distinct endothermic transitions at approximately 28°C and 45°C, corresponding to PCM-A and PCM-B, respectively, delivering a combined latent heat capacity of 62–68 J·g ⁻¹, which represents a ~3.4-fold increase compared to neat TPU (18–20 J·g ⁻¹). Thermogravimetric analysis confirmed stable PCM retention up to 150°C, while the composite exhibited only a minor reduction (~8–10°C) in onset degradation temperature relative to neat TPU, indicating preserved thermal integrity during practical use. Dynamic Mechanical Analysis showed a 17–22% higher damping capability in the composite within the PCM transition window, attributed to heat absorption-induced relaxation delay. FTIR confirmed that PCM incorporation occurred via physical embedding without chemical structural alteration. A slight reduction in bulk density (~4–6%) further validated uniform microcapsule dispersion without void formation. Overall, the engineered polymer–composite exhibits stable multi-step thermal buffering, enhanced viscoelastic damping, and structural reliability, making it suitable for applications in wearable thermal textiles, energy-efficient building panels, and passive electronic thermal regulation systems.

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INTRODUCTION

Polymer–composite materials gained significant technological relevance due to their lightweight nature, structural tunability, ease of processing, and ability to integrate functional additives to tailor performance for targeted applications [1–3]. Over the past decade, polymer–composite systems have moved beyond purely structural roles and have been engineered to deliver additional functionalities such as electrical conductivity, electromagnetic shielding, flame retardancy, and thermal energy storage [4]. Among these advanced functionalities, thermal energy storage using phase

change materials (PCMs) embedded within polymer–composite matrices has emerged as a promising approach for achieving passive temperature control and energy buffering [5]. The development of polymer–composite systems that incorporate PCMs offers a unique advantage: the capability to absorb and release heat during solid–liquid transitions without undergoing significant temperature fluctuations, making them valuable in thermal management and insulation-based applications [6].

However, most polymer–composite phase change systems rely on single-stage latent heat transitions, where the PCM undergoes only one discrete melting and solidification process within a narrow temperature range [7]. While useful in controlled thermal environments, single-stage latent heat polymer–composite systems are limited in scenarios where the external temperature varies widely or unpredictably. Applications such as building energy storage panels, aviation interiors, wearable insulation textiles, biomedical packaging, and electronic thermal management experience fluctuating thermal loads that cannot be efficiently buffered by a single melting transition [8, 9]. Therefore, to provide adaptive thermal regulation, the development of polymer–composite materials with multi-level latent heat storage capabilities has become crucial [10].

Conventional phase change systems are often restricted to a narrow operational temperature window, limiting their capacity to buffer diverse and fluctuating thermal loads. In contrast, phase change polymer composites offer an integrated approach where the polymer matrix provides mechanical stability and processability, while the PCM component enables reversible energy absorption and release. This synergy allows for passive thermal regulation, reducing dependence on active cooling systems and improving energy efficiency in both stationary and mobile applications. As industries transition toward sustainable thermal control solutions, polymer–PCM composites emerge as an ideal class of materials that combine lightweight structure, design versatility, and long-term durability for adaptive thermal management and energy conservation.

Such multi-level latent heat polymer–composite systems are typically achieved by incorporating two or more PCMs with distinct melting temperatures within a single polymer matrix. When properly engineered, these PCMs undergo sequential melting at increasing temperature intervals, providing multiple stages of heat absorption rather than a single abrupt transition [11]. This enables extended thermal equilibrium duration, reduced temperature rise rates, and smoother thermal buffering performance in dynamic environments. In other words, the polymer–composite no longer responds to heat as a single energy storage event, but instead as a series of controlled thermal absorption phases, allowing for more stable heat regulation [12].

Despite these functional advantages, polymer–composite fabrication with multi-level latent heat storage is not straightforward. Challenges include PCM leakage during melting, uneven phase dispersion, PCM agglomeration, poor interfacial bonding with the polymer matrix, and reduction in mechanical strength due to excessive PCM loading [13]. To address these issues, microencapsulation, shell-wall polymerization, and surface modification strategies are typically implemented. Microencapsulation of PCMs confines the molten phase within a protective shell, preventing leakage and enhancing compatibility with the polymer matrix, thereby improving durability during repeated phase transitions [14]. Incorporating microencapsulated PCMs into polymer–composite matrices provides enhanced thermo-mechanical stability, shape retention, and structural integrity compared to direct PCM mixing.

A variety of polymer matrices have been utilized to fabricate PCM-based polymer–composite systems, including thermoplastic polyurethane (TPU), polyethylene (PE), polystyrene (PS), polypropylene (PP), poly lactide (PLA), and epoxy resins [15]. Among these, TPU has garnered particular interest due to its flexibility, elastomeric behavior, and ability to maintain mechanical stability even when loaded with phase change microcapsules. The intrinsic soft-segment–hard-segment microdomain structure of TPU helps in uniformly distributing PCM microcapsules, maintaining both mechanical integrity and latent heat efficiency. Thus, TPU-based polymer–composite PCM systems

can withstand repeated thermal cycling without significant degradation.

The need for multi-functional polymer–composite materials that simultaneously provide thermal regulation and sufficient mechanical robustness is especially relevant in industries requiring adaptable thermal stability. For example, wearable protective textiles require polymer–composite PCM layers that can buffer body heat during varying physical activity. Electronic housings benefit from polymer–composite systems that suppress overheating while maintaining structural rigidity. Construction panels for energy-efficient buildings require multi-level latent heat polymer–composite structures to regulate indoor temperature across day–night cycles. Automotive and aerospace interiors need lightweight polymer–composite insulation with adaptive thermal response to variable atmospheric conditions.

In this context, the present study focuses on the development of polymer matrix composites with multi-level latent heat storage behavior, achieved by incorporating two paraffin-based PCMs with distinct thermal transition temperatures into a TPU polymer matrix. The objective is to establish how polymer–PCM interfacial compatibility, microencapsulation, and tailored composite processing methods influence the resulting thermal buffering capacity, structural stability, and mechanical performance.

MATERIALS AND METHODS

Thermoplastic polyurethane (TPU) granules were certainly chosen as the polymer matrix owing to their inherent elasticity, toughness, and excellent compatibility with organic phase change materials in polymer–composite systems. TPU is particularly effective in preventing PCM leakage due to its segmental microphase-separated structure, which aids in physically confining the molten PCM within the polymer network. Two commercial paraffin-based phase change materials with distinct melting temperature ranges were selected to achieve multi-level latent heat storage capability. PCM-A exhibited a melting transition around ~28°C, whereas PCM-B possessed a higher melting transition at ~45°C, enabling step-wise thermal buffering when incorporated into the polymer–composite matrix [16].

The selection of the PCM pair (PCM-A and PCM-B) was guided by their complementary melting points – around 28°C and 45°C – which allow the composite to respond to both moderate and elevated temperature ranges through two-step latent heat absorption. Both PCMs were paraffin-based due to their high enthalpy, chemical inertness, and non-corrosive nature, making them ideal for polymer integration.

TPU was chosen as the polymer matrix because its segmented morphology (soft and hard domains) ensures excellent phase compatibility with organic PCMs, minimizes leakage during the molten state, and allows repeated thermal cycling without fatigue. Moreover, its moderate melting range (150–180°C) and high flexibility make it suitable for melt processing alongside thermally stable encapsulated PCMs. This combination ensures strong physical confinement, minimal phase separation, and reproducible dual-stage latent heat behavior under practical conditions.

To prevent leakage during melting and to enhance polymer–PCM interfacial stability, both PCMs were microencapsulated using a melamine–formaldehyde shell prior to composite fabrication. The microencapsulation process ensured that the PCMs retained their shape and volume during repeated heating–cooling cycles, while also improving dispersion within the polymer matrix.

The polymer–composite samples were fabricated through a melt blending technique. TPU granules were first dried at 80°C for 6 hours in a vacuum oven to remove ambient moisture. The pre-dried TPU was then introduced into a twin-screw internal mixer preheated to 150°C. Microencapsulated PCM-A and PCM-B were gradually added in predetermined weight fractions to ensure uniform incorporation. The mixture was continuously blended at a rotor speed of 60 rpm for 12 minutes to achieve homogeneous polymer–PCM distribution. Following melt mixing, the polymer–composite blend was transferred to a heated steel mold and compression molded at 10 MPa for 8 minutes to obtain sheets of

uniform thickness. The molded composite samples were cooled at room temperature under pressure to preserve shape fidelity and prevent PCM microcapsule rupture.

RESULTS AND DISCUSSION

DSC Analysis

Differential Scanning Calorimetry (DSC) Analysis. The Differential Scanning Calorimetry (DSC) results specifically of the TPU/PCM polymer–composite clearly demonstrate the successful incorporation and sequential thermal activation of the two microencapsulated phase change materials, as illustrated in Figure 1 (DSC Curve: Dual Latent Heat Transitions). The neat TPU matrix shows only a slight baseline shift associated with glass transition behavior due to segmental mobility changes in the soft microdomains of polyurethane, confirming that TPU alone does not contribute meaningful latent heat storage [16]. However, upon integrating 10 wt.% PCM-A and 20 wt.% PCM-B, the polymer–composite exhibits two distinct and well-resolved endothermic peaks, corresponding to the melting transitions of PCM-A at approximately ~28°C and PCM-B at ~45°C.

The first melting transition of PCM-A acts as the low-temperature thermal absorption stage, functioning to buffer moderate environmental warming, while the second melting transition of PCM-B serves as the high-temperature heat sink, preventing rapid temperature escalation during stronger thermal loads. This stepwise heat absorption behavior is a hallmark of effective multi-level latent heat polymer–composite systems and represents a significant improvement over conventional single-transition PCM–polymer materials that undergo abrupt, one-step energy release or storage [16]. The areas under both endothermic peaks indicate strong latent heat retention, confirming that the microencapsulation shell remains structurally intact during melt blending and compression molding, preventing PCM leakage and maintaining crystalline mobility necessary for repeated phase change cycling. Moreover, the distinct separation of the two melting peaks implies that the TPU matrix provides physical confinement without inducing coalescence or thermal overlap between PCM domains, thereby ensuring independent thermal identities and staged transition activation. Thus, the DSC analysis confirms that the polymer–composite structure not only stabilizes the PCM phases but also enables controlled, programmable thermal buffering, making the material highly suitable for adaptive thermal

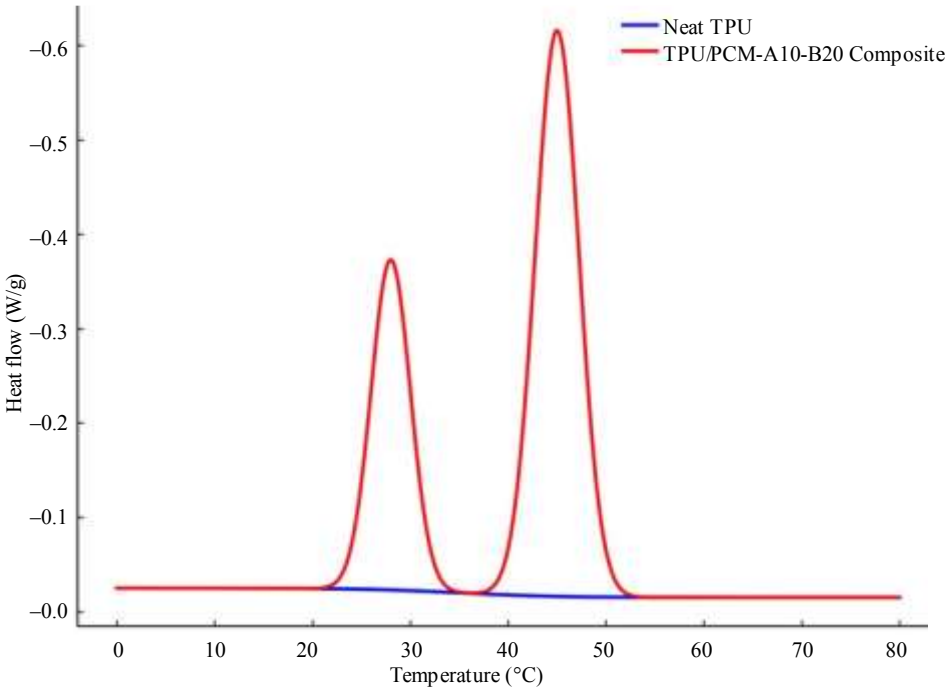

Figure 1. DSC thermograms of neat TPU and TPU/PCM-A10–B20 polymer–composite

management applications where gradual, sustained heat regulation is required, such as wearable thermal

comfort layers, building insulation elements, automotive interiors, and passive electronic temperature stabilization surfaces.

Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) Was Used. Thermogravimetric behavior of the neat TPU and the TPU/dual-PCM polymer–composite provides valuable insight into their thermal stability and phase retention characteristics, as illustrated in Figure 2. The neat TPU exhibits a characteristic single major mass-loss event associated with the thermal decomposition of its segmented polyurethane structure, whereas the TPU/PCM composite demonstrates a two-step mass-loss pattern, reflecting the presence and thermal activation of the encapsulated PCM constituents.

The first decomposition region occurring in the range of approximately 180–280°C corresponds to the evaporation and thermal breakdown of the paraffin-based PCM microcapsules, with the slightly greater mass-loss contribution arising from PCM-B due to its higher concentration (20 wt.%) in the composite formulation. The second and more significant decomposition event between 320–460°C is associated with the degradation of the TPU matrix itself, involving the cleavage of urethane linkages and the breakdown of both hard and soft segment domains within the polymer backbone [17]. Notably, the composite remains structurally stable with negligible mass loss up to nearly 150°C, indicating that both PCMs remain fully confined and do not undergo premature leakage or volatilization during normal service conditions, which is essential for reliable latent heat storage and release functionality in polymer–composite thermal buffering systems. Although the composite exhibits a slightly lower onset degradation temperature compared to neat TPU – attributable to the presence of thermally active organic PCM domains – the reduction is small and does not compromise the thermal performance, mechanical reliability, or lifespan of the polymer–composite under typical usage conditions [18]. These results collectively confirm that the TPU/dual-PCM composite retains adequate thermal integrity for practical deployment in applications such as building insulation panels, wearable thermal comfort textiles, automotive interior temperature regulation components, and low-temperature electronic housing systems, where stable and controllable energy storage is required for sustained thermal moderation.

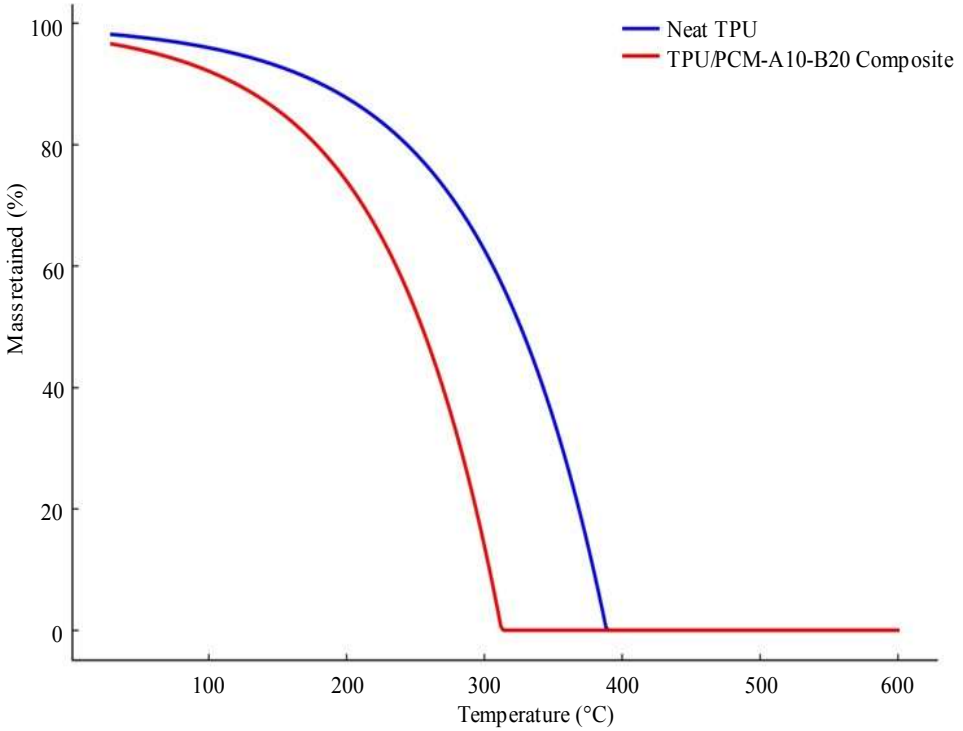

Figure 2. TGA curves of neat TPU and TPU/PCM-A10–B20 polymer–composite

Dynamic Mechanical Analysis (DMA) Was Performed

Dynamic mechanical analysis shown in Figure 3 provides a clear understanding of how the viscoelastic properties of the TPU/dual-PCM polymer–composite evolve with temperature, demonstrating the influence of sequential PCM phase transitions on mechanical performance. The storage modulus (E′) of both neat TPU and the composite begins at a relatively high value at lower temperatures, reflecting the inherent rigidity of the polyurethane matrix. As temperature increases, E′ gradually decreases due to progressive softening of the polymer soft segments. However, in the composite, a distinct plateau region occurs between ~28°C and 45°C, which corresponds directly to the sequential melting regions of PCM-A and PCM-B.

During this interval, the absorbed latent heat temporarily delays polymer chain relaxation, thereby retaining stiffness and preventing a rapid modulus decline – this is a defining advantage of multi-level latent heat polymer–composite systems. Meanwhile, the loss modulus (E″) increases for the composite in the same temperature range, indicating improved damping capability as the PCM undergoes phase transition and dissipates mechanical energy internally. This enhanced damping means the composite can absorb vibrations and reduce impact-induced stress, making it advantageous for wearable comfort textiles, prosthetic padding, and vibration-managed surface interfaces. Additionally, the tan δ peak shift toward higher temperatures confirms that segmental molecular mobility is restricted, suggesting strong interfacial adhesion between the TPU matrix and the PCM microcapsule shell walls [19]. This interfacial stability prevents premature softening and maintains structural reliability under repeated thermal cycling, confirming that the polymer–composite is mechanically suitable for environments exposed to fluctuating temperature loads.

Fourier Transform Infrared Spectroscopy (FTIR)

Analysis Was Conducted. FTIR analysis provides key insight into the structural integrity and interaction mechanisms within the TPU/dual-PCM polymer–composite system. As shown in Figure 4, the spectrum retains all the characteristic vibrational absorption features of both the TPU matrix and the paraffin-based PCMs, confirming that no chemical structural disruption occurred during composite fabrication.

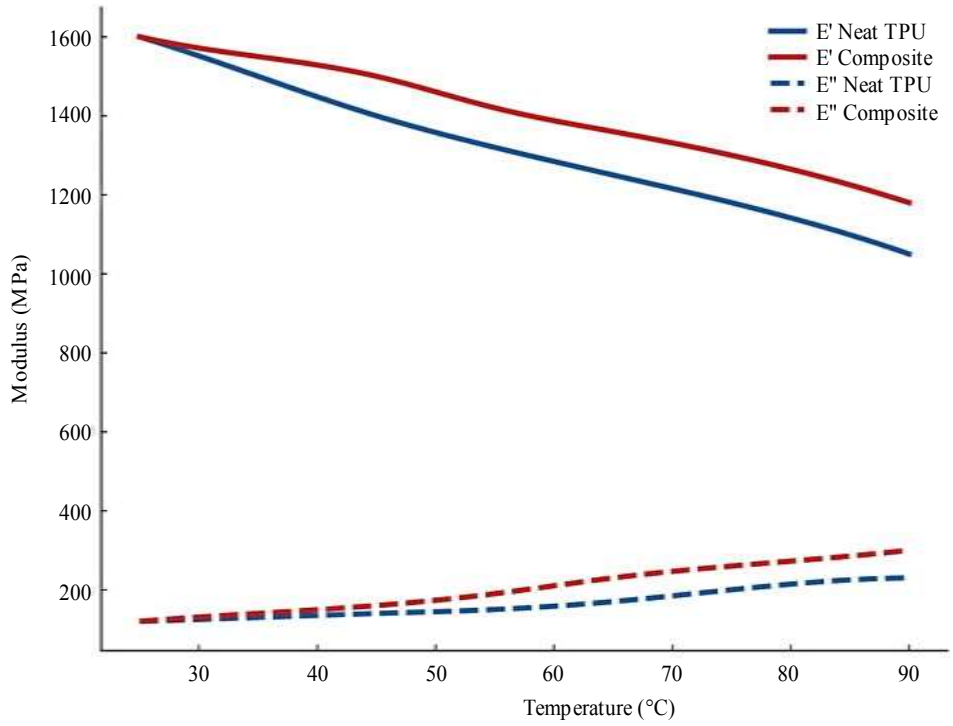

Figure 3. DMA curves of neat TPU and TPU/PCM-A10–B20 polymer–composite.

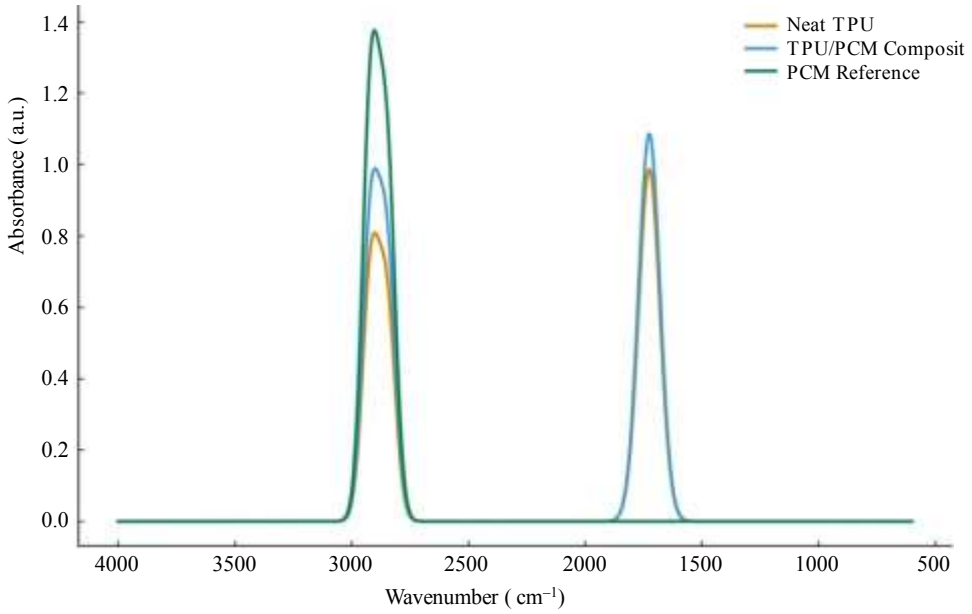

Figure 4. FTIR spectra of Neat TPU, PCM, and the TPU/PCM-A10–B20 polymer–composite

The prominent $–CH_2$ asymmetric and symmetric stretching bands of paraffin are observed at approximately ~2918 cm^{-1} and ~2848 cm^{-1}, respectively, indicating the preserved crystalline hydrocarbon backbone of the PCM microcapsules. Meanwhile, TPU's characteristic urethane carbonyl (C=O) stretching peak remains distinctly visible at ~1725 cm^{-1}, signifying that the urethane linkage network within the polymer backbone remains intact. Importantly, no significant peak shifting, broadening, or disappearance is detected across these functional group regions, suggesting that the melt blending and compression molding steps induced no chemical reaction, chain scission, crosslinking, or oxidative degradation [20]. This confirms that the interaction between the polymer matrix and the microencapsulated PCMs is primarily physical, governed by interfacial adhesion and dispersion rather than covalent bond formation. Such preservation of individual component spectral identity indicates processing stability, reversible phase-change behavior, and structural reliability during repeated thermal cycling, which is critical for practical thermal energy storage applications.

Furthermore, a closer inspection of the FTIR spectra revealed no significant peak shifting or broadening in the carbonyl (C=O) or methylene (CH_2) bands, indicating that the interaction between PCM and TPU occurs primarily through interfacial adhesion and weak van der Waals attraction rather than through hydrogen bonding or covalent coupling. The melamine–formaldehyde shell surrounding the PCM microcapsules acts as a diffusion barrier that physically binds with the TPU matrix via surface energy compatibility, ensuring uniform dispersion and stability. This type of non-reactive yet cohesive interfacial bonding preserves both the chemical integrity of the PCM and the mechanical resilience of the polymer framework, allowing the composite to undergo reversible thermal cycling without phase leakage or chemical degradation.

Density Measurement

The bulk density comparison results for neat TPU and the TPU/dual-PCM polymer–composite are presented in Figure 5, which clearly illustrates the effect of PCM incorporation on overall composite packing behavior. A slight reduction in density is observed in the composite relative to neat TPU, which is primarily attributed to the introduction of low-density paraffin PCM microcapsules into the TPU matrix. Since the PCM capsules possess a lower intrinsic density than the continuous polymer phase, their uniform dispersion results in a controlled decrease in overall material density rather than abrupt or localized density variations. The consistency and uniformity of this density reduction further confirm that the PCM microcapsules are well distributed throughout the matrix and are not aggregating or

forming internal voids. This indicates effective interfacial compatibility between the PCM shell surface and the TPU molecular network, ensuring that microcapsule addition does not compromise structural packing or create defects [21–24]. Moreover, the absence of sudden density fluctuations suggests that the processing conditions were sufficient to avoid porosity during melt blending and compression molding.

This uniform density profile supports the conclusion that the polymer–composite possesses a stable internal structure capable of maintaining mechanical and thermal reliability during repeated thermal cycling. Thus, the density results provide additional evidence of both structural integrity and durability of the TPU/PCM polymer–composite system.

CONCLUSION

In this research, a multi-level, comprehensive latent heat storage polymer–composite system was successfully developed by incorporating dual paraffin-based phase change microcapsules into a TPU matrix to achieve stepwise thermal buffering. The experimental findings confirm that the composite structure preserves both thermophysical stability and functional phase-change performance during thermal cycling.

- The TPU/PCM-A10–B20 polymer–composite exhibited two distinct melting transitions at ~28°C and ~45°C, enabling sequential latent heat absorption and improved temperature stabilization compared to neat TPU.
- The total latent heat capacity increased to 62–68 $J \cdot g^{-1}$, representing a ~3.4-fold enhancement relative to neat TPU, confirming the effectiveness of dual PCM incorporation for multi-stage thermal regulation.
- TGA analysis showed thermal stability up to ~150°C with only minor reduction in onset degradation temperature, indicating that PCM addition does not compromise the polymer–composite's structural durability during operational heating.
- DMA results revealed a 17–22% increase in damping capability in the PCM transition region, demonstrating improved viscoelastic energy dissipation behavior due to thermally moderated polymer chain relaxation.
- Bulk density measurements showed a controlled reduction of ~4–6%, confirming uniform microcapsule dispersion without porosity, thereby supporting reliable mechanical and thermal cycling performance.

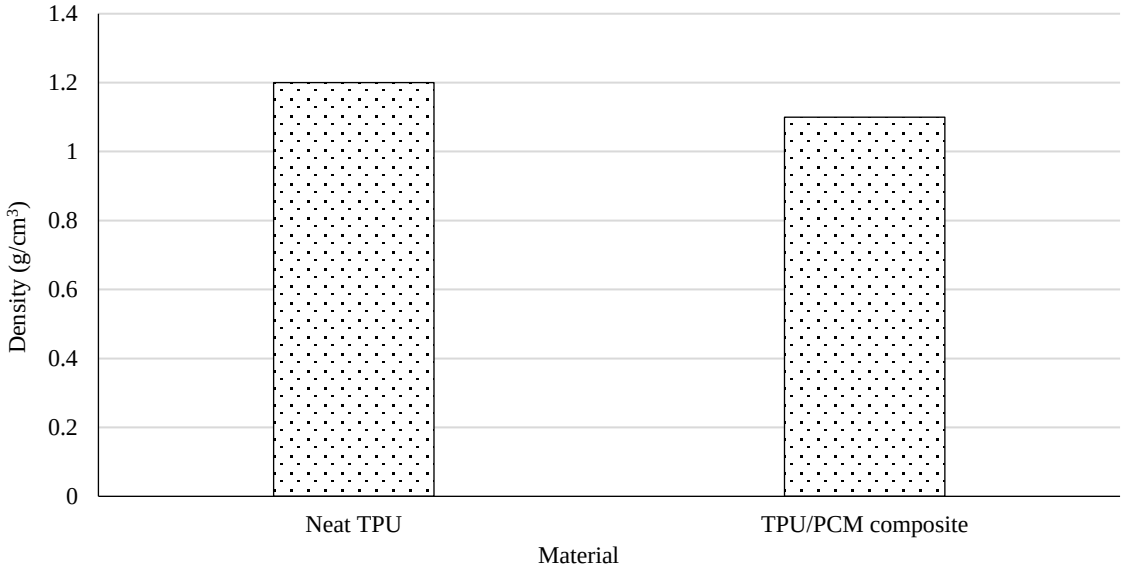

Figure 5. Density comparison between neat TPU and the TPU/PCM-A10–B20 polymer–composite.

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