ORIGINAL ARTICLE

Exploring thermal dynamics of polyaniline-modified paraffin wax phase change material with varied PANI loadings (1–4% wt.)

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Abstract

In this experimental study, we explore the potential enhancements in thermal conductivity while investigating alterations in latent heat and phase change temperature within Composite Phase Change Materials (PCMs). These composites consist of Paraffin Wax (PW) as the base material, incorporating dispersed conducting Polyaniline (PANI) powder in varying concentrations ranging from 1% wt. to 4% wt. The mass fractions of PANI added to PW include 1%, 2%, 3%, and 4%, and the composite PCMs are meticulously prepared through ultrasonication. Examining the surface morphology of Composite Phase Change Materials (PCMs) involved utilizing a Scanning Electron Microscope (SEM), while the determination of thermal conductivity employed a Heat Flow Meter. Additionally, latent heat and phase change temperatures were assessed through Differential Scanning Calorimetry (DSC). The obtained results indicate an augmentation in the thermal conductivity of the composites when compared to Paraffin Wax (PW). Specifically, thermal conductivity exhibited a 40% increase for 1% wt. of PANI, yet experienced a subsequent decline for the remaining weight percentages. Furthermore, the latent heat and phase change temperatures of the composites were observed to decrease in comparison to PW. These composite PCMs with enhanced thermal conductivity, achieved through the incorporation of Polyaniline in Paraffin Wax, are highly potential for several applications in energy storage systems, thermal regulation devices, and heat management technologies.

Nomenclature

$°C$	Degree centigrade
K	Thermal conductivity, W/(mK)
k_{comp}	Thermal conductivity of composite PCMs, W/(mK)
k_{pw}	Thermal conductivity of PW, W/(mK)
ΔH_{comp}	Latent heat of composite PCMs, kJ/kg
ΔH_{pw}	Latent heat of PW, kJ/kg
mW	Milli Watts
$\%Wt.$	Percent weight

Greek Letters

Ψ	Psi, %

Acronyms

DSC	Differential Scanning Calorimetry
PANI	Polyaniline
SEM	Scanning Electron Microscope

Extended author information available on the last page of the article

Abbreviations

PW	Paraffin Wax
PWPA	Paraffin Wax Polyaniline

1 Introduction

Storing thermal energy efficiently has become a vital requirement to meet the current global energy demand. Research on PCMs that store latent heat is generating increasing curiosity and interest in the fields of engineering and technology due to their immense potential in wide-ranging situations where they can be utilized. Phase Change Materials (PCMs) are an innovative class of materials that can store and release large amounts of energy as they undergo a phase change. This makes them highly attractive for thermal energy storage applications, as they can help to improve the energy efficiency of various systems, reduce greenhouse gas emissions, and mitigate climate change.

The PCM's low thermal conductivity value diminishes the rate at which heat gets transferred, thus causing

a limitation on their usage [1]. Exploring the technique of adding a highly conductive nanoparticle to PCMs, is emerging as a viable solution among the various techniques available to increase the thermal conductivity of PCMs further, owing to its capability and simplicity [2, 3]. Particles at the nanoscale have increased surface area to volume ratio, decreased degree of precipitation, and can influence the melting and solidification rates that assist in increasing the thermal conductivity of PCMs [3, 4]. Investigations provide evidence that PCMs embedded with nanoparticles exhibit improved phase change and heat storage capabilities and hence are suitable for efficiently storing energy [5].

Paraffin Wax (PW), an organic PCM is chosen for this study because of its remarkable traits such as broad melting temperature range, thermal stability, chemical balance, easy availability, and low price. Its broad melting temperature range means it can store and release energy over a wide range of temperatures, making it highly versatile and suitable for a variety of applications. Its high thermal stability means it can withstand high temperatures without degrading or decomposing, making it highly durable and long-lasting. Therefore, it is widely employed for practical applications of thermal energy storage [6]. Ho and Gao [7] found that thermal conductivity improved by 2% and 6% respectively when paraffin was embedded with 5% and 10% mass fractions of alumina nanoparticles. Wang et al. [8] experimentally proved that the thermal properties of PW (dispersed with TiO_2 nanoparticles of 1%, 3%, 5%, and 7% weight percentages) can be improved. Copper nanoparticles with mass fractions 0.5, 1.0, and 1.5 were added to PW by Owolabi et al. [9]. Their conclusions on the composite PCMs indicate that thermal conductivity enhanced by 20.23%, 20.04%, and 20.6%, respectively. Kumar et al. [2] dispersed 0.5%, 1.0%, and 2.0% weight of Zinc oxide in PW and observed an increase in thermal conductivity by about 41.67% with the sample containing 2% wt. of ZnO nanoparticles.

The study and exploration of synthesized nanoparticles gain significance in the application and development of new Composite PCMs [10]. Synthesized particles are preferred because of their simplicity of synthesis, low cost, and easy availability. Polyaniline (PANI), a synthesized conducting polymer has excellent properties and attributes like high electrical conductivity, good thermal and chemical stability, biocompatibility, ease and simplicity of synthesis, broader applicability, and cost-effective. Therefore, PANI has caught the attention of the scientific community and has indeed become one of the most widely studied polymers in recent times [11, 12]. The applications of PANI range from electronics to biomedical engineering. It can be blended with synthetic and natural polymers and can also be used in energy storage devices [13].

Novel nanocomposites with PW and PANI have been prepared by George et al. [6] with 0.1, 0.5, 1, and 5 weight percentages of PANI. It was noted from their experiments that thermal conductivity was enhanced by 46.8% and latent heat by 8.2% at 1% wt. of PANI. A more recent study was conducted with Polyaniline@cobalt-Paraffin wax nanocomposites where the varied wt % of Polyaniline was in between 0.1 to 5. with 0.1%, 0.5%, 1%, and 5% weight, Cobalt with 1% wt. (say PC1) and 2% wt. (say PC2) was added to PW. The nanocomposite PCM (containing 0.5 wt.%) exhibited a notable 20.4% increase in thermal conductivity, while the PCM with 0.1 wt.% demonstrated a 15.9% enhancement in latent heat. However, the latent heat of fusion exhibited both ascending and descending trends in both samples with 1 and 2 wt. % [14].

A popular recommendation for the addition of nanoparticles is in the range between 0.2% and 4.0% of weight ratios [15]. George et al. [6] studied till 1% wt. and jumped to 5% wt. Addition of PANI to PW, leaving a wide gap in studies of composite PCMs in the intervening range. This leads to observations related to the thermal behaviour of PCMs in this range potentially remaining unexplored. Therefore, this has inspired us to study the PCMs with 1%, 2%, 3%, and 4% weights of PANI.

Investigating PCMs in the weight percentage range mentioned above can provide crucial insights to researchers seeking to optimize the performance of composite PCMs for energy storage applications. Breakthroughs achieved in improving the PCMs' ability to store and release thermal energy effectively and efficiently will allow researchers to productively develop more sustainable and energy-efficient storage solutions. Hence, weight percentages of 1%, 2%, 3%, and 4% of PANI were added to PW and investigated for thermal conductivity, latent heat, and phase change temperature variations.

2 Materials and procedure

2.1 Materials

PW in pellet form was acquired from Pioneer Chemicals Ltd., Hyderabad, India. The melting point of this material ranges between 54 °C to 56 °C. Conductive Polyaniline powder in emeraldine salt form, doped with p-toluene sulfonic acid with an average size of around 400 nm was procured from Progressive Polymers Ltd., Pune, India.

2.2 Fabrication of nanocomposite PCMs

Composite PCMs comprising PW and PANI were prepared by ultrasonication, and the method followed is described here. The required quantity of PW was measured in a microbalance and transferred to a mould where it was heated to 70 °C. Synthesized PANI powder of the required quantity was measured and added to the melted PW along with surfactant Triton X-100. The mixture was then placed in an electrically heated

Table 1 Labels of composite PCMs

Sample	%Wt. PANI	Labels of Composite PCMs
1	1%	PWPA-1
2	2%	PWPA-2
3	3%	PWPA-3
4	4%	PWPA-4

ultra sonication device (40 kHz, 120 W) where it was subjected to heating at 70 °C and vibration at the same time and ultra sonicated for 4 to 5 h. This technique helps prevent the agglomeration of nanoparticles and provides stability. Finally, the mixture was allowed to slowly cool down to room temperature. Composite PCMs thus prepared were used for further testing. Composite PCMs were labelled based on the weight percentage of PANI added as presented in Table 1.

Figure 1 shows the image of composite PCM, PWPA-2 prepared by the method described above.

2.2.1 Scanning Electron Microscope (SEM)

Surface morphology and microstructures of PW, PANI, and Composite PCMs were captured with HITACHI S-3700N SEM. The magnification of this instrument ranges from 5× to 300,000×.

2.2.2 Thermal conductivity

The instrument used to determine thermal conductivity was the FOX 50 Heat Flow Meter from TA Instruments. The

Fig. 1 Composite PCM PWPA-2

sample was placed between the two temperature-controlled plates that are parallel to each other. The temperature of the two plates is maintained different and constant. The measurements were carried out under steady-state one-dimension heat transfer conditions and thermal conductivity was evaluated based on Fourier's law of conduction heat transfer. The thickness of the sample was 25mm. The instrument has a temperature range of -10 °C to 110 °C, accuracy ± 3%, and a range of testing 0.1 to 10 W/(mK).

2.2.3 Differential Scanning Calorimetry (DSC)

Latent heat values and temperatures at Phase change of composite PCMs under study have been computed using Shimadzu Differential Scanning Calorimeter (Model: DSC-60 A). The sample weighing about 2 mg was placed in an Aluminium pan for the test. The samples were evaluated under an atmosphere of Nitrogen between 30 °C and 120 °C with rate of heating maintained at 10 °C/min.

To ensure that the results are more accurate, each test has been repeated three times and the average values have been presented. Table 2 shows the equipment used for experimentation and their details.

3 Results and discussion

3.1 Morphology

Surface morphological images were captured by SEM. Figure 2(a) presents the SEM image of PW at 500× magnification. The microstructure of PW reveals a layered structure. The imaging was done in low vacuum mode. Figure 2(b) shows the SEM image of Polyaniline at 500× magnification. PANI tubes appear to be porous with irregular granular distribution. The mean size of the particles was found to be around 400 nm.

The SEM images of PWPA-1, 2, 3 and 4 at 500× magnification can be seen in Figs. 2(c–f) respectively. The visual data obtained from these figures clearly depicts a seamless integration of the dark green PANI particles into the layered architecture of PW. The microscopic analysis reveals that the PANI particles are precisely incorporated within the base matrix, creating a homogenous distribution throughout the material. PW and the composites were not subjected to higher magnification because the high intensity of the electron beam would melt the material before capturing the image of the scan [6, 15].

3.2 Thermal conductivity

Thermal conductivity is a material property that determines the rate of heat transfer through a material. It is a measure of

Table 2 Equipment details

Instrument	Make	Details	
Digital Ultrasonic Cleaner	SISCO 20A	Unit Size	$270 \times 170 \times 210$ mm
		Ultrasonic Power	120W
		Frequency	40 kHz
Analytical Micro Balance	VIRGO - 10	Accuracy	0.01 g (for 200 g)
		Operating Temperature	5 °C–35 °C
SEM	HITACHI S-3700N	Magnification	$5\times$ - $300,000\times$
DSC	SHIMADZU - 60A	Temperature Range	-140 °C to +600 °C
		Atmosphere	Oxidative or inert gas
Heat Flow Meter	TA Instruments – FOX 50	Range of Testing	0.1 to 10 W/mK
		Accuracy	$\pm 3\%$
		Temperature Range	-10 °C to +110 °C
		Sample Thickness	25 mm (1 inch)

Fig. 2 **a** Microstructure of PW, **b** Microstructure of PANI, **c** Microstructure of PWPA-1, **d** Microstructure of PWPA-2, **e** Microstructure of PWPA-3, **f** Microstructure of PWPA-4
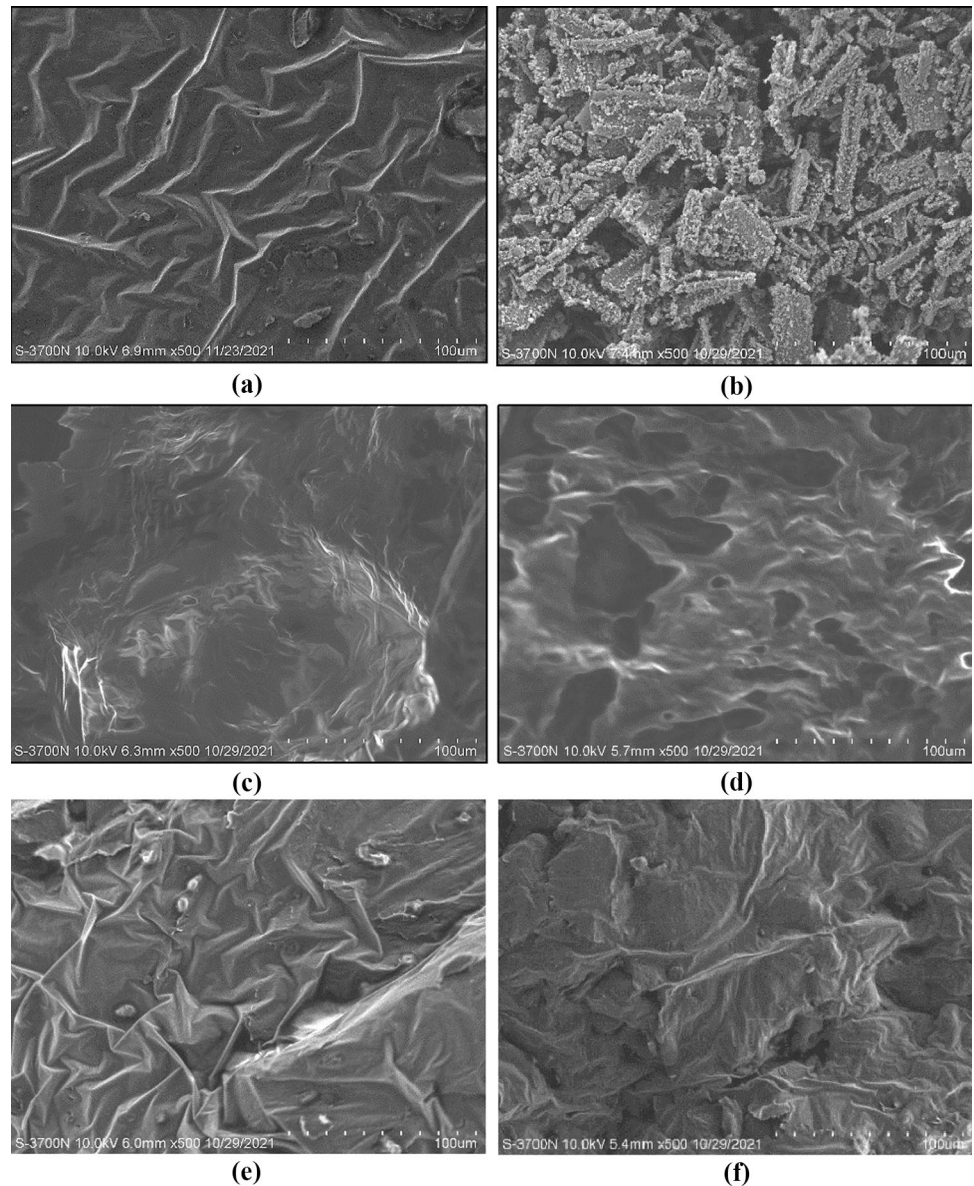
a material's ability to conduct heat. This is an important property to consider when evaluating the effectiveness of phase change materials (PCMs) in thermal energy storage applications. PCMs are designed to store and release large amounts of energy as they undergo a phase change between solid and liquid states. By measuring the thermal conductivity of a PCM, researchers can assess how efficiently it can transfer heat and store thermal energy. This information is critical in determining the suitability of a PCM for specific thermal energy storage applications. A higher thermal conductivity indicates that the PCM can transfer heat more efficiently, which makes it more effective in storing and releasing thermal energy.

In this study, thermal conductivity was measured by Heat Flow Meter for PW and composite PCMs. Thermal conductivity values of PW and composites for various PANI weight percentages including Enhancement ratios of composites have been presented in Table 3.

Enhancement ratio ψ is calculated from Eq. (1) as shown below [16]. The various parameters in this equation have been explained in the Nomenclature section, provided towards the end of this article.

$$\psi = [(k_{comp} - k_{pw})/k_{pw}] \times 100 \tag{1}$$

**Table 3** Thermal conductivity values of PW and composite PCMs

Material	Thermal Conductivity k W/(mK)	Enhancement Ratio ψ (%)
PW	0.2	0
PWPA-1	0.28 ± 0.02	40
PWPA-2	0.26 ± 0.02	30
PWPA-3	0.247 ± 0.02	23.5
PWPA-4	0.232 ± 0.02	16

Increment in thermal conductivity of composites compared to PW can be appreciated from Fig. 3.

The composite PCMs displayed enhanced thermal conductivity compared to PW. The highest thermal conductivity value of 0.28 ± 0.02 W/(mK) is obtained for 1% wt. of PANI with an enhancement ratio of 40%. In the context of materials, understanding thermal heat transport is important to get an insight into how materials transfer and store thermal energy. The two main mechanisms of thermal heat transport in materials are electron transport and phonon transport. Electron transport is predominant in metals and conducting polymers, while phonon transport is the primary mode in insulators and semiconductors. Understanding the mechanisms of thermal heat transport is important for evaluating the thermal conductivity and effectiveness of materials in thermal energy storage applications.

In PW, electron thermal transport gets enhanced when the particles added form a connected thermal network, which does not disturb its phonon thermal transport. The elevation of thermal conductivity in composite PCMs when compared with PW can be attributed to this phenomenon. However, in this study, as the weight percentage of PANI increased, thermal conductivity were found to be in a decreasing trend [17]. This may be because of the agglomeration of the particles which caused a collapse of the thermal network and thereby reduced the electron thermal transport [18]. The change in thermal conductivity values could be influenced by the material's structural and compositional characteristics. Initially, as the concentration of polyaniline increases, there might be improved alignment of polymer chains or enhanced intermolecular interactions, leading to higher thermal conductivity. However, at higher concentrations, factors such as increased disorder, presence of defects, or changes in the material's morphology may start to dominate, causing a subsequent decrease in thermal conductivity [17, 19]. Additionally, it

Fig. 3 Thermal conductivity variations of PW and Composites at various PANI weight percentages
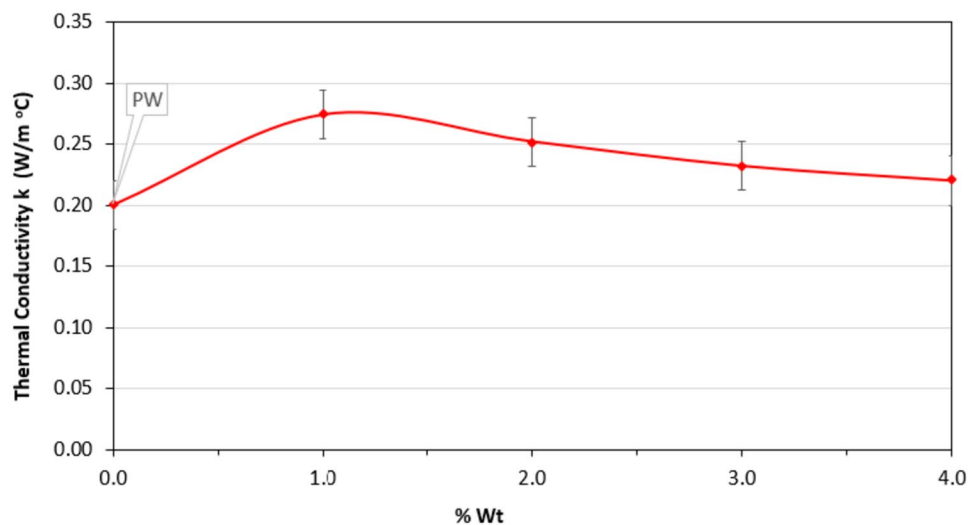
is likely possible that at certain concentrations, the thermal conductivity is affected by a balance between various competing factors. Furthermore, a higher concentration of particles also increases impurities which might have reduced the phonon mean free path, leading to a reduction in phonon thermal transport and thereby, lowered thermal conductivity [14, 20–23].

3.3 Latent heat of pristine PW and PW mixed with PANI

DSC investigation is the basis for evaluating the ability of the composite PCMs to store or release energy. The latent heat of materials under study was determined with DSC and values have been presented in Table 4. Figure 4 displays the changes in latent heat for different PANI weight percentages.

The significance of the downward peak in Fig. 4. is the transition of phase of the sample from solid to liquid. Latent heat is computed by considering the area below the phase change peak. Equation (2) is used to calculate the latent heat of composite PCMs [24, 25].

$$\text{Latent Heat} = (\Delta H_{comp} / \Delta H_{pw}) \times 100 \qquad (2)$$

The various parameters in this equation have been explained in the Nomenclature section, provided toward the end of this article.

The transition for PW and Composite PCMs starts around 55 °C to 63 °C and terminates between 70 °C and 77 °C. The range of temperature of Phase change depends on the physical and chemical properties of PW used.

The composite PCM, PWPA-1, containing 1% wt. of PANI exhibits the highest latent heat value compared to other composite PCMs, indicating its potential for efficient energy storage and release. This could be due to the particles' behavior as nucleating agents, which facilitates crystal formation during the paraffinic phase. However, it is noticeable that the latent heat decreases with an increase in particle percentage. This trend can be explained by the fact that at higher percentages, the particles hinder molecular movement in the paraffinic phase, resulting in difficulty in crystal formation [25]. The DSC curve of PW is narrow while the curves of composite PCMs appear to be wider. The reason
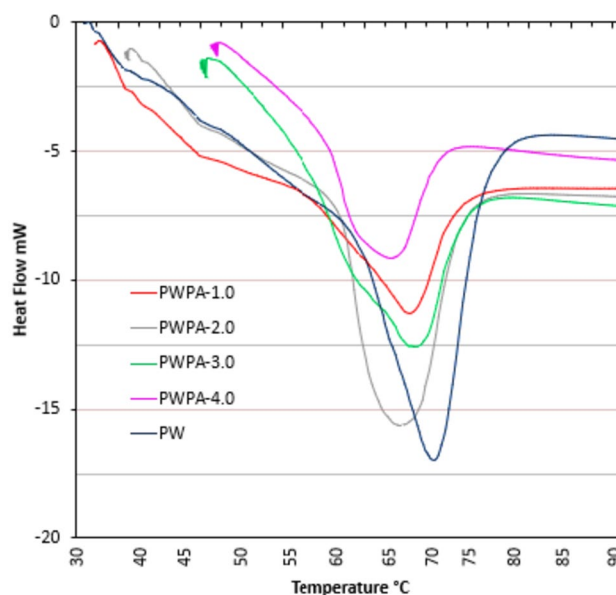

Fig. 4 DSC curves for PW and Composite PCMs

for this may be attributed to the impurities in the particles which cause phonon scattering at the boundary, leading to an impediment in phonon thermal transport and thereby reducing heat transfer from the surface to the center [26].

The latent heat of all composite PCMs decreased when compared to PW. This can be attributed to many factors. Firstly, it depends on the specific heat of the particles added to the base PCMs. Lower values of specific heat of nanoparticles than base PCMs will reduce the thermal absorbing capacity of composite PCMs, leading to the reduction in its latent heat of fusion. Secondly, the thermal conductivity of nanoparticles and their percentage of addition also reduces the latent heat of the composite PCMs. The volume or Weight percent of nanoparticles influences latent heat. Thirdly, the stability of nanoparticles inside the base PCMs also plays an important role in increasing or decreasing the latent heat of PCMs. The increase in latent heat at PANI 4 Wt% could be attributed to potential structural changes, altered intermolecular interactions, variations in crystallinity, or differences in the degree of polymerization compared to lower concentrations. These factors, influenced by the higher concentration of polyaniline, may contribute to the increased energy required for the phase change, showcasing the complex interplay of material properties in determining latent heat values [27–29]. Additional experimental analyses would be necessary to pinpoint the specific mechanisms underlying this observed trend.

Particles within the base matrix must be well dispersed and homogenously distributed throughout the matrix. Nanoparticles tend to agglomerate and form clusters. The size, shape, and cluster distribution perform an important part in

Table 4 Latent heat values of PW and composite PCMs

Material	Latent Heat (kJ/kg)
PW	184.56
PWPA-1	103.54
PWPA-2	85.07
PWPA-3	78.54
PWPA-4	90.44

the stability of PCMs [30, 31]. Longer sonication time can help to increase stability and thereby improve the Latent Heat of PCMs [5]. Functionalized graphene with amine (NH_2) group integration is used because it has been demonstrated that functionalization improves thermal properties over base materials, reduces nanoparticle agglomeration, and increases dispersibility in organic solvent along with stability [32, 33].

3.4 Phase change temperature

Table 5 depicts temperatures at a change of phase for PW and Composites.

The temperature variations at a change of phase for PW and composites for different weight percentages of PANI can be noticed in Fig. 5.

The melting temperature of all composites declined in comparison with PW. This could be because of the feeble interaction between the matrix and the fluid [34]. It is generally observed from other studies, that in most of the PCMs, melting temperatures decreased when nanoparticles were added.

The phase change temperature of polymers is predominantly affected by their degree of crystallinity, which is the measure of the ordered arrangement of their constituent molecules. Additionally, the phase change temperature of polymers is also influenced by their symmetry, which plays a crucial role in determining the formation of crystalline structures. Moreover, the intermolecular forces between the polymer chains also contribute significantly to the determination of the phase change temperature of polymers. Thus, understanding the complex interplay of these factors is essential for the precise prediction and manipulation of the phase change behavior of polymers.

In addition to the factors mentioned earlier, the phase change temperature of a material is also affected by several other external parameters, such as the rate of heating, mass of the sample, and heat transfer conditions. The rate of heating plays a crucial role in determining the phase transition behavior of a material, as it affects the time taken for the material to reach its melting or solidification temperature. The mass of the sample is another important factor that influences the phase change temperature, as a larger sample size requires a greater amount of energy to induce phase change. Finally, the heat transfer conditions for each sample, including the type of container used, the presence of any thermal insulation, and the ambient temperature, can significantly impact the measured phase change temperature. Therefore, it is imperative to consider all these external factors while analyzing the phase change behavior of a material. The percentage decrease in melting temperature in this study ranges from 1.11% to 7.25% which is not far from the stated value. More studies are required to appreciate the effect of nanoparticle addition on phase change temperature [5].

Table 5 Phase change temperature of Paraffin Wax and composite PCMs

Material	Phase Change Temperature (ºC)
PW	70.2
PWPA-1	67.29
PWPA-2	66.2
PWPA-3	67.81
PWPA-4	65.11

Fig. 5 Melting Temperature variation with the addition of PANI
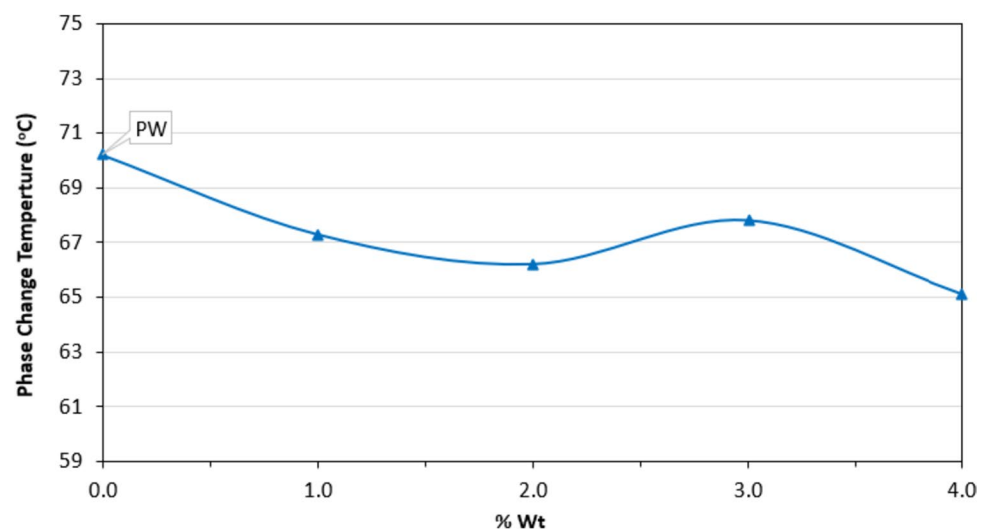
4 Conclusions

The above-mentioned experiments are performed to investigate the impact on thermal conductivity, latent heat, and melting temperatures when PANI particles in weight percentages of 1%, 2%, 3%, and 4% are added to PW.

The following are the findings from the study:

1. Thermal conductivity of all Composite PCMs increased compared to PW. The highest thermal conductivity of 0.28 ± 0.02 W/(mK) is achieved for 1% wt. of PANI.
2. The thermal conductivity Enhancement ratio for PWPA-1 Composite PCM is around 40%. The latent heat of all Composite PCMs decreased compared to PW.
3. The percentage decrease in latent heat ranges from 43.89% to 57.4%. The highest value of latent heat was obtained for 1% wt. of PANI.
4. The Melting Temperature of all Composite PCMs decreased compared to PW. The percentage decrease in melting temperature ranges from 1.11% to 7.25%.
5. The findings of this research align with the existing similar works where the composite PCMs demonstrated the highest thermal conductivity and latent heat at a PANI concentration of 1% wt.
6. The study suggests that future experiments should prioritize PW-PANI phase change materials with 1% PANI concentration, as opposed to higher concentrations, to achieve optimal benefits.

5 Future scope

Researchers can consider adding metal and metal oxide nanoparticles with high thermal conductivity along with PANI to PW to further enhance the thermal conductivity of PCMs. The advantages of adding a synthesized Polymer (PANI), which is easily available and economical, in combination with metal and metal oxide nanoparticles of high thermal conductivity make it worthwhile to pay attention to, in the quest to further enhance the thermal conductivity of PCMs. Studies on arriving at an optimal weight percent of nanoparticle addition can also be undertaken for enhancing thermal conductivity and latent heat of PCMs. Techniques like longer sonication times could aid in the improvement of the stability of nanoparticles added and this may result in fine-tuning the process of phase change. Further studies on melting temperatures and Thermal stability of PCMs may be taken up to enable their employability for thermal energy storage applications.

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Declarations

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