ENHANCEMENT OF THERMO-PHYSICAL PROPERTIES OF FORM-STABLE NANO-ENHANCED PHASE CHANGE MATERIALS: ADVANCING MARITIME SUSTAINABILITY

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SUMMARY

This paper explores the application of novel, form-stable eutectic mixtures in thermal energy storage systems for maritime environments. These advanced materials present a significant leap forward, addressing critical challenges faced by conventional phase change materials (PCMs) in marine environments. The stability, eliminating leakage and fluidity issues encountered with liquid PCMs are ensured by incorporating 2-hydroxypropyl ether cellulose (HPEC) as a gelling agent. Additionally, the thermal properties and heat transfer capacities were significantly enhanced, and eventually, overall system efficiency improved by including nano-graphene platelets (NGPs). Notably, NGPs effectively suppress supercooling, minimizing energy losses and guaranteeing consistent performance at elevated temperatures.

Further, the eutectic mixture demonstrates exceptional durability through an accelerated thermal reliability test, guaranteeing optimal performance over a projected seventy-year lifespan. This enhanced thermal performance, and enduring stability combination establishes the form-stable eutectic mixture with NGPs as an up-and-coming solution for diverse maritime applications requiring efficient and reliable thermal energy storage. This research provides a compelling case for implementing form-stable eutectic mixtures with NGPs in maritime thermal energy storage systems. Its superior performance and sustainability offer significant advantages for diverse shipboard and offshore applications, contributing to improved energy efficiency, environmental sustainability, and operational resilience within the maritime sector.

KEYWORDS

Nano-enhanced phase change material (NEPCM), Eutectic mixture, Nano-graphene platelets, Thermal energy storage, Maritime applications

1. INTRODUCTION

The maritime industry faces unique challenges in achieving energy efficiency due to several factors: limited space on vessels, fluctuating operational demands, and the need for reliable performance in diverse marine environments (Dedes et al., 2012). Conventional energy sources often contribute to significant fuel consumption and greenhouse gas emissions, impacting operational costs and environmental sustainability (Zhou et al., 2016). In this context, PCMs emerge as a promising technology offering efficient thermal energy storage and the potential to reduce reliance on fossil fuels. During phase transitions, PCMs store and release large amounts of energy at nearly constant temperatures; these materials can absorb and release large amounts of energy during phase transitions (Ahmed et al., 2010). This characteristic allows them to regulate temperatures, reduce energy consumption, and minimize temperature fluctuations within maritime applications. For instance, PCMs integrated into shipboard climate control systems can store excess heat during off-peak periods and release it when needed, ensuring crew comfort and efficient equipment operation. Additionally, they can be utilized to recover waste heat from engines and utilize it for onboard operations, leading to reduced fuel consumption and lower emissions.

PCMs have been used to regulate building temperatures and reduce energy consumption and temperature fluctuations (Dolado et al., 2012). PCMs can be installed in the ceiling, walls, or ground, typically covered in thin polymer films. The effectiveness of salt hydrate PCMs is lowered by cycling due to supercooling and phase separation (Hendricks and Van Sark, 2013, Kosny et al., 2013). On the contrary, fatty acids are non-toxic, have a low phase transition volume, and require little to no supercooling over long cycling times (Cao et al., 2014, Qian et al., 2015). The benefits of methyl esters include their low flammability, low combustibility, low corrosion of building materials, and high latent heat. (Jamil et al., 2019, Leong et al., 2019). The melting point and the thermal conductivity of fatty acids and methyl esters are higher than the building's thermal comfort zone. Consequently, eutectic mixtures can adjust PCM melting points to optimal ranges (Ali et al., 2021). Eutectic fatty acid mixtures have been shown in various studies to have desirable structural properties. (Jamil et al., 2019, Leong et al., 2019, Radomska et al., 2020). This was also one of the few studies to examine the eutectic mixture's thermo-physical properties like specific heat capacity, thermal conductivity, and thermal diffusivity. The references (Tiwari et al., 2023, Syam Sundar et al., 2023) provide an in-depth evaluation of appropriate PCMs for building structures. In previous eutectic mixture studies, researchers did not experimentally determine the thermal stability during cycling. Because of the characteristics of the eutectic mixture, it is necessary to determine the composition ratio with meticulous accuracy to achieve consistent performance and long-term stability. The specific heat capacity, thermal conductivity, and thermal diffusivity of liquid and solid eutectic mixtures were only occasionally reported in the studies that came before. Several studies have been done on these additional thermophysical properties (Pereira et al., 2023). Before adding PCMs to a thermal system, this data is needed to model their optimal quantity, efficiency, and placement. More heat capacity allows for storing a more significant amount of sensible heat. This is a significant consideration in situations where PCMs operate beyond their phase transition period, such as when severe weather occurs and when the discharge from the previous cycle is insufficient to reach the range.

A design challenge is incorporating and containing liquid phase PCM without compromising system dependability or performance. Form-stable nanoscale eutectic mixtures will be developed to improve thermal efficiency. These additives can produce potential PCMs (Kalidasan et al., 2023). When HPEC is used as a thickening or gelling agent, the PCM becomes less fluid, forms more solidly, and does not leak any liquid. Additionally, the use of NGPs improves heat transfer. With the creation of new PCMs with a higher energy storage capacity and a suitable phase transition temperature, the objective is to improve upon the existing PCMs. The gelling agent makes the shape more stable to withstand repeated freezing and thawing. It is possible to accomplish this objective by preventing the formation of the eutectic mixture phase. The homogeneity of the mixture is preserved by a cross-linked gel, which also prevents phase separation and segregation. This is accomplished by preventing the migration of nanostructures (Hosseininaveh et al., 2022). The movement of nanostructures is prevented by a cross-linked gel, which helps maintain the mixture's homogeneity and prevents phase separation or segregation. Another advantage of gelling agents is that they have a higher heat capacity than other substances because of their crystallinity; even though they are inexpensive, gelling, thickening agents might not be compatible with PCM. In order to thicken or gel particular PCMs, a particular gelling agent is required to produce the desired effect. In order to determine whether or not the gelling agent is viable, two PCM eutectic mixtures need to undergo experimental verification. Increasing the ignitability and thermal conductivity of PCM is accomplished through using NGPs.

This research delves into developing a novel form-stable eutectic mixture designed for maritime applications. This paper proposes a form-stable nanoscale eutectic mixture incorporating HPEC and NGPs as gelling agents to improve thermal conductivity. Through rigorous testing and analysis, this research explores the proposed eutectic mixture's thermophysical properties, thermal stability, and energy storage capacity. The ultimate goal is to demonstrate its feasibility and effectiveness for various maritime applications, contributing to improved energy efficiency, operational resilience, and environmental sustainability within the maritime sector.

2. MATERIALS AND METHODS

The binary eutectic mixture of MPLA was created by combining methyl palmitate (MP) and lauric acid (LA), which were acquired from Sarna Chemicals. Table 1 provides a comprehensive listing of the physical and chemical properties of PCMs. Powdered HPEC from Jigchem Universal has been purchased for use in thickening gels. Light Powder from Shilpa Enterprises containing chosen NGPs has a density of at least 2 g/cm^3, a specific surface area of 15 m^2/g, a through-plane average thickness of 10 nm, and a lateral thickness of 5 µm.

The mixed phase change medium in eutectic point proportioning is stable because of its consistent performance, defined latent heat of fusion, and single melting point. Schroeder's formula (Elston, 1994) can be used to determine the optimal ratio for combining the mixture and the theoretical eutectic point. The liquidus equation for a binary mixed phase change medium can be determined through the application of phase equilibrium theory and the second law of thermodynamics:

$$T_m = \frac{H_i}{\frac{H_i}{T_i} - R\ln X_i} \tag{1}$$

In this case, i = A, B, where T_i represents the Kelvin phase transition temperature and H_i represents the J/mol latent heat of fusion for PCM components A and B, respectively. X_i denotes the molar ratio of A to B in this case. T_m is

Table 1. Data regarding the PCMs' theoretical thermal characteristics and chemical composition

	Methyl Palmitate (MP)	**Lauric Acid (LA)**
Scientific Name	Methyl hex decanoate	Dodecanoic acid
CAS Number	112-39-0	143-07-7
Purity	99%	99%
Molecular Formula	$C_{17}H_{34}O_2$	$C_{12}H_{24}O_2$
Molecular Weight [g/mol]	270.4507	200.3178
Density [g/ml] 25°C	0.852	0.883
Specific heat [J/gK] 25°C	1.75	2.01
Latent heat [J/g]	229.5	188.7
Melting point [°C]	29	44

the expected melting point of the A-B binary mixture in Kelvin, and R is the universal gas constant, with a value of approximately 8.314 J/mol/K. The production of NGPs from graphene powder required a two-step process. Initially, graphene powder was heated. Graphene powder underwent conventional pre-treatment by mixing it with a solution of 98% H_2SO_4 and 30% H_2O_2 in a 1:1.5 (v/v) ratio and exposing the solution to a temperature of 20°C for 2 hours. The solid was filtered and rinsed with deionized water to achieve a pH of 7. The solid was dried at 80°C for 24 hours. The solid was subjected to a thermal shock using an 800W microwave for 60 seconds. The solid sample was subjected to pre-treatment and thermal shock following the methodology previously described to obtain an HPEC sample. The HPEC was ground with NaCl salt in the second stage following the methodology outlined in (Alinejad and Mahmoodi, 2017).

A mixture containing 5 grams of HPEC and 1 gram of NaCl salt was prepared in a molar ratio 3:1. Pulverisette 5 planetary ball mill is used for homogenizing, grinding, and combining samples. The Pulverisette 5 consists of two main lines: classic and premium. The Pulverisette 5/2 model has two grinding stations, whereas the Pulverisette 5/4 model has four. There are two different models available for these lines. The mixture was ground in a Pulverisette 5 for two hours at 350 rpm with an argon inert gas medium and a ball-to-powder weight ratio of 20:1. The NaCl salt was removed by treating the powder with deionized water in an ultrasonic bath. The final sample was obtained through centrifugation and subsequent vacuum drying at 80°C. The binary eutectic mixture of MPLA, with a 60/40 molar ratio, was created by melt-blending. The MPLA was fully melted by a 20-minute vacuum drying process in an oven at 50°C. NGP/HPEC was then incorporated into the molten MPLA at mass ratios of 4%, 6%, 8%, and 10% by weight compared to the MPLA. The control group did not receive NGP and was supplemented with 4% HPEC by weight. The mixtures were all submerged at 50°C for twenty-four hours in a vacuum-drying oven.

The prepared samples were marked as follows:

- MPLAH04 = MP-LA/HPEC-4,
- MPLAH04N = MP-LA/HPEC-4/NGP,
- MPLAH06N = MP-LA/HPEC-6/NGP,
- MPLAH08N = MP-LA/HPEC-8/NGP, and
- MPLAH10N = MP-LA/HPEC-10/NGP.

3. CHARACTERIZATION OF NEPCM

3.1 DIFFERENTIAL SCANNING CALORIMETER (DSC)

A Diffraction Scanning Calorimeter (DSC) was used to analyze the phase transition temperature, latent heat of phase change, and other thermal properties of composites. Each sample showed two distinct peaks during the solid-liquid phase transition, representing the solidification and melting phases of the transition. Each sample displays the same behavior during phase transitions, as shown in Figure 1. Various percentage decreases in PCM melting and solidification temperatures were noted when HPEC and NGP/HPEC were added at different weight percentages (4%, 6%, 8%, and 10%).

3.2 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning electron microscopy (SEM) was used to analyze the morphological characteristics of composite phase change materials (PCMs) made of HPEC, MPLAH04, and MPLAH10N at different magnifications. Figure 2 (a) shows the distinctive worm-like configuration of the generated HPEC, where graphene flakes intertwine to

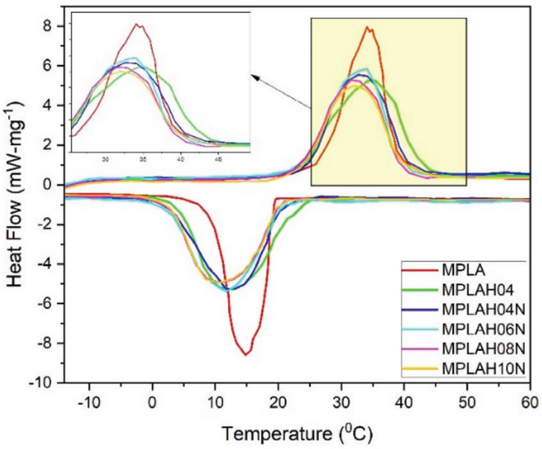

Figure 1. DSC curve for MPLA, MPLAH04, MPLAH04N, MPLAH06N, MPLAH08N, and MPLAH10N

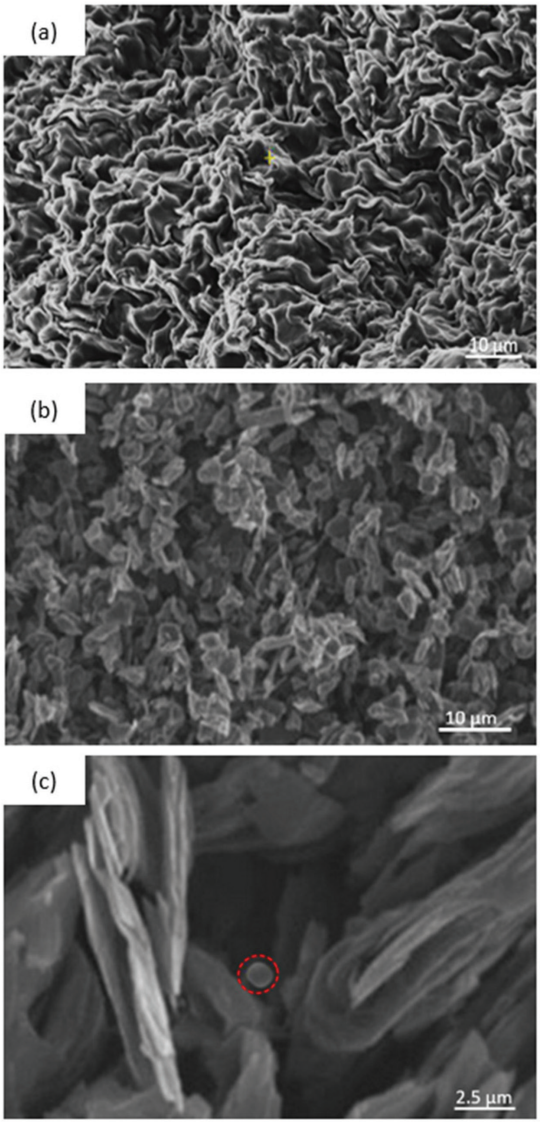

Figure 2. SEM image of (a) HPEC, (b) MPLAH04, and (c) MPLAH10N

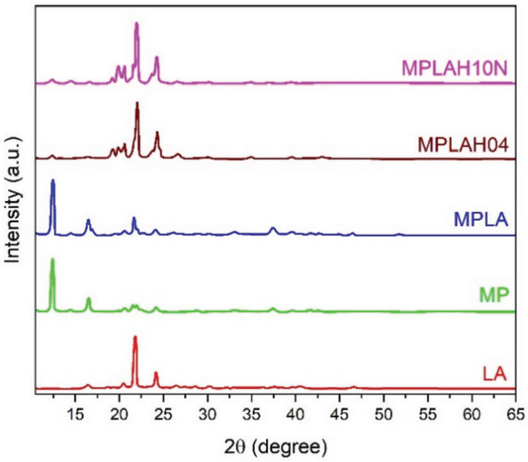

Figure 1. XRD spectrum of MP, LA, MPLA, MPLAH04, and MPLAH10N

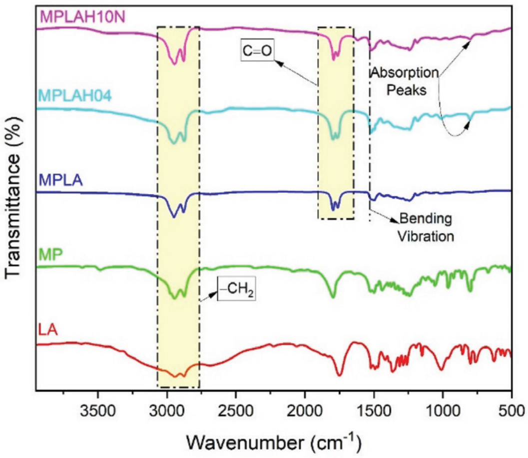

Figure 2. FTIR spectrum of MP, LA, MPLA, MPLAH04, and MPLAH10N

create a porous network. NGP has the potential to act as a supportive medium in the adsorption of PCM. The worm-like structure formed by the interconnections of multiple microcells was maintained in NGP Figure 2 (b). Segregation is notably missing, and many disk-shaped HPEC nanoparticles are evenly spread out. Figure 2 (c) illustrates the surface topology of the MPLAH10N composite PCMs. When the PCMs are added, the network structure of the NGP disappears and transforms into a dense, spherical shape.

3.3 X-RAY DIFFRACTION (XRD)

Figure 3 shows an X-ray diffraction (XRD) spectrum of various composite PCMs. Three primary diffraction peaks in MP, LA, MPLA, and MPLAH10N composites have melting points that range from 12°C to 21°C. These melting points are found in the composites. At temperatures ranging from 21°C to 24°C, the MPLA, MPLAH04, and MPLAH10N composite PCMs display diffraction peaks related to LA. Additionally, the composite PCMs MPLAH10N and MPLAH04 exhibited a distinct diffraction peak at a temperature of 25.2°C, corresponding with HPEC. At a temperature of 10°C, composite PCMs made of MPLAH10N show a diffraction peak. It is difficult to detect the diffraction peak of NGP in the composites' spectra because of the extremely low concentration of NGP present in the composites. It is clear from the results that the procedure for manufacturing the MPLAH10N composite PCMs was successfully implemented.

3.4 FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The FTIR spectra of the multi-phase composite PCMs made of MP, LA, MPLA, MPLAH04, and MPLAH10N can be seen in Figure 4. Absorption peaks at 2944 cm^{-1}

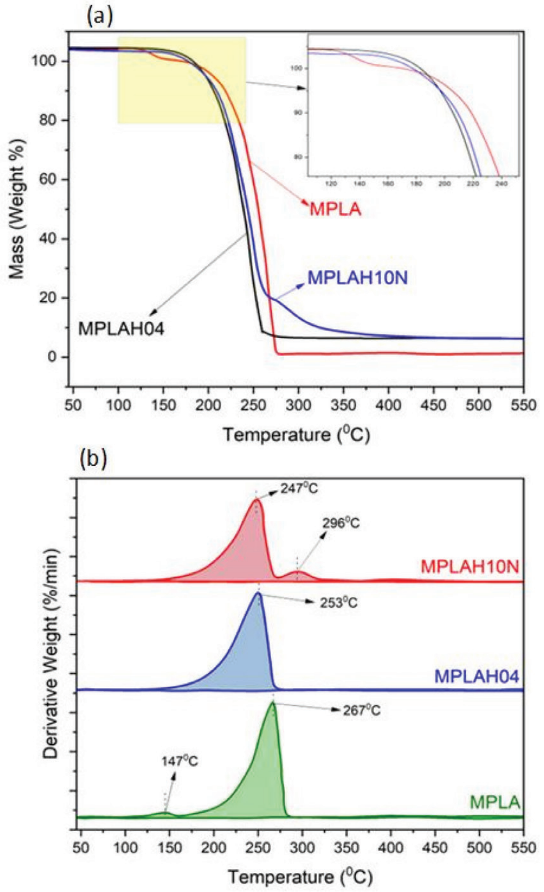

Figure 3. (a) TGA and (b) DTG curves of MPLA, MPLAH04, and MPLAH10N

represent the symmetric stretching vibrations of —CH$_2$, while 2874 cm^{-1} represent the asymmetric stretching vibrations of —CH$_2$. The stretching vibration of the C=O functional group in saturated aliphatic ester MP occurs at 1793 cm^{-1}, whereas the vibration of the fatty acid LA occurs at 1751 cm^{-1} with the same frequency. Two characteristics of the fatty chain are present in the MPLA mixture. The first is an absorption peak at 799 cm^{-1}, corresponding to the in-plane rocking vibration of C—H. The second is the C—H bending vibration in the —CH$_2$ group, which occurs at 1522 cm^{-1}. There is no discernible change when looking at the peak spectra of the composites and the MPLA.

3.5 THERMOGRAVIMETRIC ANALYSIS (TGA) AND DERIVATIVE THERMOGRAVIMETRIC ANALYSIS (DTG)

The Thermogravimetric Analysis (TGA) curves of two different compounds, MPLAH04 and MPLAH10N, can be seen in Figure 5 (a). The MPLA weight loss procedure consists of two phases. Weight loss began at an initial temperature of 102°C. MPLA experienced almost complete decomposition at around 285°C, leading to a weight loss rate of 97.96%. MPLAH04 showed a single weight loss peak over a prolonged duration with a total rate of 92.63% at 147°C. The measured mass ratio matched the residual mass fraction of the MPLAH04 composite PCM, which was 6.02%. The onset weight loss temperature of the MPLAH10N composite was around 153.6°C, similar to that of the MPLAH04 composite. Based on these findings, it is feasible to use composite phase change materials produced at temperatures under 98°C. The thermogravimetric analysis (TGA) of the composite PCMs containing MPLAH10N showed a quick weight loss at lower temperatures, which then decreased in speed. The introduction of NGP particles into the lamellar nanostructure led to surface roughening of the NGP/HPEC, resulting in a deceleration of the degradation of the MPLA, which constituted around 10% of the material. The final decomposition temperature of 517°C for MPLAH10N shows improved heat stability compared to MPLA (302°C) and MPLAH04 (291°C). The thermal decomposition process near the MPLA, MPLAH04, and MPLAH10N interfaces is better defined by examining the DTG curves shown in Figure 5 (b). The area under the curve represents the weight loss proportion for each sample.

4. RESULTS AND DISCUSSION

The developed PCM exhibited desirable phase transition behavior with a latent heat of 205 J/gK and melting and freezing temperatures of 36°C and 20.4°C, respectively. Before adding the form-stable PCM to the MPLA mixture, DSC was used to compare the two to ensure no interference from the phase transition. Gelling agents reduce the potential energy of PCM mixtures. In order to create a PCM composite that is both form-stable and leak-proof, this study set out to determine the ideal and minimum concentrations of HPEC gelling agent. Samples gelled were cooled to 30°C for 24 hours after preparing PCM mixtures. The amount of mass that was lost as a result of leakage was calculated. There is a direct correlation between the ratio of HPEC gelling agent and the changes in mixture texture, from liquid to thick gel, and from 100% leakage ratio of PCM to 0%. As a reference, a combination containing 3.5% HPEC gelling agent results in a fragile gel texture and a leakage ratio of 42.50%.

In contrast, a mixture containing 16% HPEC gelling agent yields a thick gel texture and a leakage ratio of 0%. This study compares thermal conductivity measurements of thermally enhanced form-stable PCMs with those of pure and gelled PCMs and PCMs with varying ratios of NGPs. This study compares the thermal conductivity of the PCM before and after the addition of NGPs, as measured in K%, to determine the relative increase in thermal conductivity. Optimal NGP concentration prior to phase separation or incongruent melting was determined by studying the effect of NGPs on the melting phase transition. The melting point, weight fractions of PCMs, and full width at half maximum (FWHM) of pure (MPLA), gelled (MPLAH04),

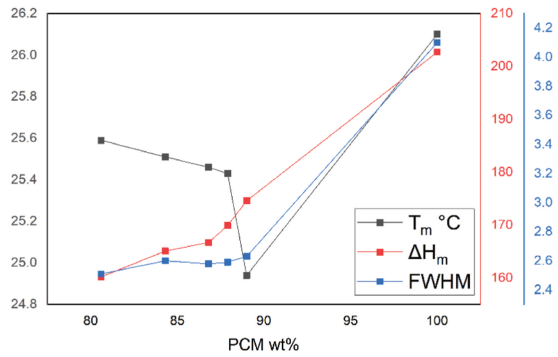

Figure 4. Comparison of the melting peak, PCM weight fractions, and FWHM for MPLA, MPLAH04, MPLAH04N, MPLAH06N, MPLAH08N and MPLAH10N

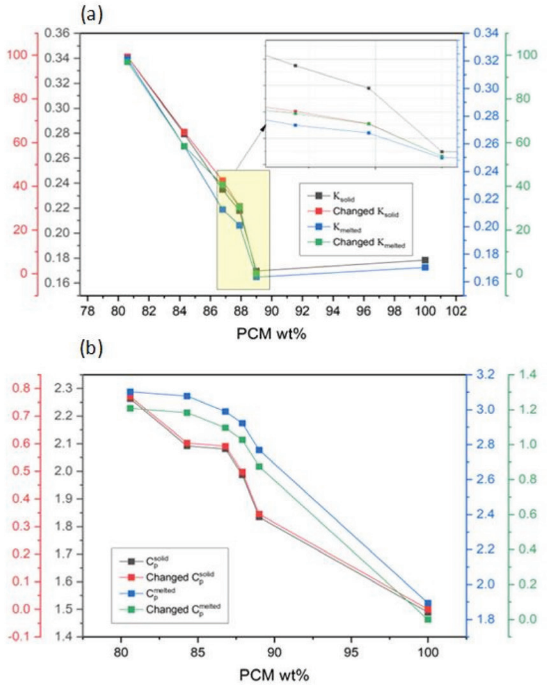

Figure 5. Comparison of (a) thermal conductivity and (b) specific heat capacity before and after adding NGPs to PCMs

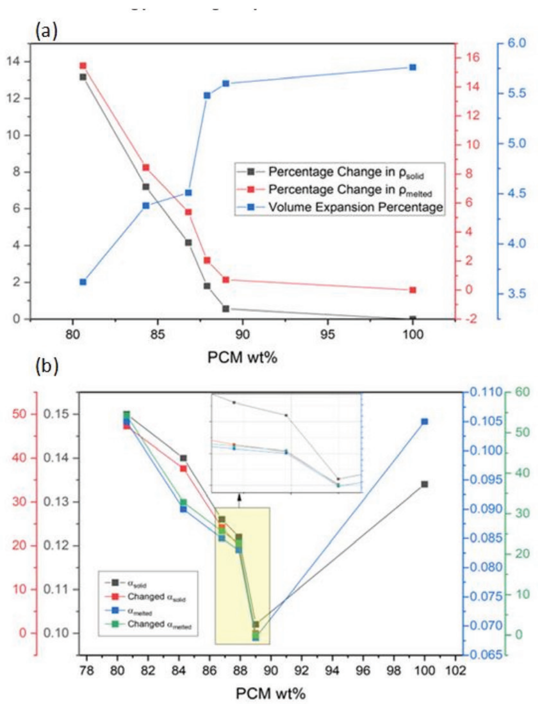

Figure 6. Comparison of (a) percentage change in density and resultant volume expansion (b) thermal diffusivity before and after the addition of NGPs to PCMs in solid state and melted state

and nano-enhanced PCMs (with different concentrations of NGPs) are displayed in Figure 6.

DSC thermal analysis showed that no phase changes or incongruent melting were detected when NGPs were added. In every mixture containing NGP, there was one distinct melting point. The ability to fully store latent heat of fusion in a smaller temperature range was responsible for the narrowest FWHM of 2.62°C, which was achieved by adding 10% NGPs.

After adding NGPs to the mixture, the PCM's melting point rose marginally. Although adding 10% NGPs raised the temperature by only 0.21°C, it did not affect the melting point. The precision of DSC thermocouples, which are used to measure melting temperatures, is quite close to the 0.04°C increase. Hence, it is logical to assume that the melting temperature was unaffected by adding NGPs. Results comparing thermal conductivity measurements on thermally enhanced form-stable PCMs, pure PCMs, and gelled PCMs are shown in Figure 7 (a) for various NGP ratios. To determine the increase in thermal conductivity (Change in K) the PCM's thermal conductivity is compared before and after the addition of NGPs. With a margin of error of 0.004 W/mK, the given values represent the average of 10 measurements taken for each sample. An interval of five times the duration of the experiment was set to allow PCMs sufficient time to thermally relax and return to their steady state between each set of measurements. The temperature at which it melted was 36°C, while it froze at 10°C.

Several different enhanced PCM mixtures' experimental specific heat capacities (C_p) in the solid and liquid states are shown in Figure 7 (b) over a 20°C temperature range close to the melting point. The form-stable PCM had a much higher specific heat capacity before adding NGPs (45.3% for the melted state and 23.1% for the solid state). Another critical factor is the PCM eutectic mixture's growing crystallinity. The addition of 10% NGPs increased the specific heat capacity of solid PCM by 51.4%, the specific heat capacity of molten PCM by 64.8%, and the total energy storage by a notable 1.3%.

The thermal diffusivity of solid-state PCM was measured after its physical density was established. Figure 8 (a) shows the final PCM compound mixture densities. Comparative analysis is performed on the relative densities of pure PCM, form-stable PCM, and enhanced PCM compositions. Thermal diffusivity is a crucial parameter to characterize heat diffusion in practical contexts. In a thermal energy storage application, the latent heat capacity and phase transition temperature are not high enough to accurately predict the overall improvement in heat transfer or to model the actual behavior of PCMs. Figure 8 (b) shows how NGPs change the thermal diffusivity of the PCM mixtures. The incorporation of NGPs enhanced the form-stable PCM's thermal diffusivity. When compared to the form-stable PCM, the solid-state thermal diffusivity increased by 48.49% and the liquid-state thermal diffusivity by 55.13% when NGPs were added at mass fractions of 4%, 6%, 8%, and 10%, respectively. Thermodynamic diffusivity is comparable to pure PCM in form-stabilized enhanced PCM with 10% NGPs. The material's specific heat capacity, thermal conductivity, and thermal diffusivity were all improved by adding NGP at a concentration of 10%.

5. FUTURE SCOPE

The research findings indicate that the improved form-stable eutectic mixture has significant potential for maritime engineering applications due to its exceptional thermal stability, high latent heat, and superior thermal conductivity. Energy efficiency, operational resilience, and environmental sustainability are all areas in the maritime sector that stand to benefit from these advantages. A nano-enhanced PCM was found. In the eutectic mixture of fatty acids and methyl esters, MP and LA serve as PCMs. Adding an HPEC gelling agent reduced the PCM's fluidity and improved its form stability, eliminating the problem of liquid leakage. By incorporating 10% NGPs into a stable PCM, the thermal conductivity was enhanced, as evidenced by a 99.27% increase in the solid state and a 96.9% increase in the liquid state compared to the initial value. The specific heat capacity in the solid state increased by 51.98%, in the liquid state by 63.8%, and in the thermal diffusivity by 47.27% and 54.28%, respectively. The latent heat values for melting and freezing the enhanced form-stable PCM are 175.8 J/g and 173.9 J/g, respectively, higher than those of other PCMs with improved form stability. The MPLAH10N demonstrated outstanding freezing behavior, as confirmed by DSC and experimental apparatus, with a temperature difference of less than 0.1°C between melting and freezing. Over 35,000 thermal cycles, the phase transition temperatures and latent heat values of the improved form-stable eutectic mixture remained unchanged, proving its thermal stability. There is great hope that the improved form-stable eutectic mixture will transform thermal energy management for various marine uses to get the most out of it and help make the marine industry more sustainable and efficient; research and development must continue.

6. CONCLUSION

This research demonstrates the promising potential of novel, form-stable eutectic mixtures for maritime thermal energy storage applications. By addressing critical limitations of conventional PCMs, such as limited thermal conductivity, supercooling, and stability concerns, these enhanced materials offer significant advantages for the maritime sector. The developed eutectic mixture exhibits superior thermal performance, including increased thermal conductivity, improved specific heat capacity, and enhanced thermal diffusivity. These characteristics enable efficient heat transfer and storage, which is critical for maritime applications. Incorporating nano-graphene platelets further reduces supercooling, ensuring consistent performance and minimizing energy losses. The material demonstrates exceptional thermal stability over extended cycles, guaranteeing reliable operation in demanding maritime environments. Using PCMs for thermal energy storage is an innovative strategy for reducing energy consumption, greenhouse gas emissions, and fossil fuel consumption. Even though PCMs are increasingly used for thermal energy storage, they must be improved to meet the needs of the vast majority of these applications. A more scientific approach to designing eutectic PCMs was adopted after several performance-affecting factors were verified and enhanced. Enhanced thermal performance in PCMs and new (Solid to Gel) form-stable eutectic PCMs with reduced supercooling are discussed. Thermal conductivity, specific heat, thermal diffusivity, as a function of temperature, and density are examples of thermo-physical properties that have been compiled into a valuable database for use in simulation and modeling tools to determine the optimal placement and loading of PCMs in the real world based on climate zones. Despite its promising thermal and physical properties, the enhanced form stable PCM's compatibility with building materials needs further investigation. It is concluded that the improved form-stable PCM shows promise as a thermal energy storage and thermal comfort material. Despite these promising prospects, further research is necessary to explore the compatibility of the PCM with specific maritime building materials and optimize its integration into relevant systems. Overall, this work paves the way for transforming thermal energy management within the maritime sector. Leveraging the enhanced performance and stability of the developed eutectic mixture can contribute to a more sustainable, efficient, and environmentally friendly maritime industry.

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