



Article Comparative Investigation of Thermal Properties Improvement of Nano-Enhanced Organic Phase Change Materials

Aravindh Madhavankutty Ambika ¹, Gopi Kannan Kalimuthu ² and Veerakumar Chinnasamy ³,*

- ¹ Ministry of New and Renewable Energy, New Delhi 110003, India
- ² Department of Mechanical Engineering, Indian Institute of Technology (Indian School of Mines), Dhanbad 826004, India
- ³ Department of Mechanical Engineering, Chosun University, 309 Pilmundaero, Dong-gu, Gwangju 61452, Republic of Korea
- Correspondence: veerakumar@chosun.ac.kr

Abstract: Thermal energy storage (TES) using phase change materials (PCMs) is one of the potential solutions for stockpiling thermal energy and utilizing it for different applications, which results in effective energy usage. The main drawback of organic PCMs in practical applications is poor heat transfer due to low thermal conductivity (TC). Therefore, investigations into nano-enhanced PCMs are being explored to improve their thermophysical properties. In this work, the various thermophysical characteristics of nano-enhanced lauryl alcohol as a PCM were investigated using carbon-based and metallic nanoparticles. The results indicated that the addition of nanoparticles improved its thermal properties and affected other physical properties, such as viscosity. The latent heat was degraded with the addition of nanoparticles. The results revealed that by adding MWCNTs and CuO nanoparticles, a maximum of 82.6% and 49.6% improvement in TC was achieved, respectively. The maximum drop in latent heat during melting and freezing for the PCM with MWCNTs was about 10.1% and 9.3%, respectively, whereas for the PCM with CuO, they were about 11% and 10.3%, respectively. The lowest supercooling for the PCM with MWCNTs and CuO nanoparticles was 8.6 and 8.3 °C, respectively. The present work confirms that nano-enhanced PCMs can be a potential material for storing thermal energy for various applications.

Keywords: phase change material; nano-enhanced; thermal energy storage; thermal conductivity; heat transfer

1. Introduction

Reducing energy consumption through more responsible and effective use of energy resources is known as energy conservation. It entails cutting down on energy waste and using practices and technology that reduce energy use while preserving the required comfort or service levels. In addition to being an essential part of sustainable development, energy saving also helps to mitigate climate change, lower greenhouse gas emissions, and encourage environmental stewardship. Various technologies are being practiced for efficient energy usage, and thermal energy storage (TES) is crucial [1]. TES systems have a wide range of industrial applications, offering benefits such as increased energy efficiency, reduced energy costs, and improved process flexibility. TES is used in district heating and cooling networks to store surplus thermal energy during off-peak hours for later use during peak demand periods. It helps balance supply and demand, reduces energy costs, and improves system reliability and stability [2,3]. TES systems are integrated into building HVAC (heating, ventilation, and air conditioning) systems to store thermal energy for space heating or cooling. They help reduce energy consumption, shift energy demand away from peak hours, and improve building comfort and temperature control. TES systems can be classified based on the storage medium, operation principle, and temperature range. Sensible heat storage systems store energy by changing the temperature



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). of a material without changing its phase. Latent heat thermal energy storage (LHTES) systems store energy by changing the phase of a material (solid to liquid or vice versa) while keeping the temperature constant. Thermochemical storage systems store energy through reversible chemical reactions that absorb or release heat. These types of thermal energy storage offer flexibility in terms of storage capacity, operating temperature range, and application suitability, allowing for tailored solutions to meet diverse energy storage needs. Among the different strategies of TES, phase change material (PCM)-based LHTES is more advantageous due to its ability to accumulate more thermal energy and maintain constant temperature storage and retrieval [4]. The PCM undergoes physical phase transformation during the stockpile and retrieval of thermal energy. PCMs can be classified based on several criteria, including their chemical composition, phase change mechanism, temperature range of operation, and application. Generally, PCMs are categorized as organic, inorganic, and eutectic. Organic PCMs are composed of organic compounds, such as paraffins, fatty acids, and esters. Organic PCMs often have high latent heat storage capacity and are typically used in moderate-temperature applications. Even though the PCM-based TES has several advantages, there are certain practical difficulties in applying it for applications, such as poor thermal conductivity (TC), compatibility with the container materials, volume of expansion and stability, etc. The heat transfer ability is a crucial property of a PCM for fast heat storage and retrieval. Various techniques are available for heat transfer enhancement of PCMs, and employing nanoparticles is a novel strategy that is widely used [5,6]. Techniques such as fins, encapsulation [7], shape stabilization [8], and nanoparticle inclusion are adopted to improve the thermal properties. Researchers have shown interest in different PCMs and nano-inclusions to enhance PCM thermophysical properties, which results in an efficient TES system. Using nanoparticles to increase PCMs' TC is a viable way to improve the material's overall efficiency and heat transfer capabilities in thermal energy storage applications. Significant increases in TC can be attained by adding nanoparticles to PCM matrices and fine-tuning their dispersion and interaction. This will improve heat transfer performance and efficiency in thermal energy storage applications.

Rashid et al. [9] presented a critical review on recent advances in PCM-based cold TES. This review investigated the experimental and numerical investigations of the heat transfer properties using PCM, factors influencing the TC, nanoparticle inclusion, and encapsulation of PCMs. The same authors reviewed the recent developments and challenges of using PCMs in concrete for TES and utilization [10]. Pereira et al. [11] presented an overview of thermal energy harvesting using NEPCMs. This critical survey provides information on energy harvesting and conversion using PCMs enhanced with nanostructures. Anand et al. [12] prepared a capric acid nano-enhanced (NE) PCM using manganese dioxide nanoparticles at different concentrations and reported that the LH was in the $145-164 \text{ kJkg}^{-1}$ range. Metallic nanoparticle nano-inclusions are beneficial since they have less of an impact on the PCM's ability to store energy. Ouikhalfan et al. [13] used four distinct nanoparticles (TiO₂, CuO, Al₂O₃, and ZnO) at varying concentrations to create a nanocomposite PCM of myristic acid. The PCM nanocomposites were studied using several characterization methods. The findings showed that myristic acid containing 2 wt% Al₂O₃ and 2 wt% ZnO is appropriate for use in solar TES applications. Han et al. [14] described a eutectic mixture of KNO₃ and NaNO₃ with 1 wt% of Al₂O₃ nanoparticles at various sizes, including 80, 135, 200, 300, and 1000 nm. Their findings demonstrated that the ideal Al_2O_3 thickness for improving liquid-specific heat at temperatures between 235 and 300 °C was 300 nm. Santosh et al. [15] examined the thermal performance of an Al₂O₃ nano-dispersed PCM and a surface-roughened PCM and reported his findings. Arshad et al. [16,17] researched the thermophysical properties of nanocomposites containing mono and hybrid nanoparticles. According to their findings, the hybrid nanocomposite PCMs produced have better thermal properties and can be applied to electronic device thermal management. Muzhanje et al. [18] prepared an NE-PCM and studied the physical and thermal characterization. The melting rate was improved threefold with 5 wt% of metallic nanoparticles. Karthikeyan et al. [19] developed a binary eutectic PCM using hybrid nanoparticles, and its thermal properties were investigated using various techniques. The NE-PCM showed a maximum TC improvement of 55.7%. Rashid et al. [20] investigated and reviewed the recent advancement in solidification enhancement of PCM using fins and nanoparticles. The heat recovery potential improvement of an NE-PCM was investigated by Mahdi and Nsofor [21]. The results elucidated that nano-PCMs have improved heat recovery performance and that the degraded concentration of nanoparticles accelerated the PCM solidification. Venkatraman et al. [22] made an investigational study on improved PCM discharging performance using steatite nanoparticles at different concentrations. The discharge performance was significantly improved, and heat was stored and maintained without external energy. Baskakov et al. [23] investigated paraffin with reduced graphene oxide as a thermal battery material. The TC of the paraffin was increased, which resulted in a temperature rise up to 72 °C with two minutes of microwave heating. Xie [24] studied the thermal property enhancement of a salt hydrate PCM with copper nanoparticles and investigated its ability in battery thermal management. Li et al. [25] investigated the improvement of TC using modified carbon nanotubes. The modification strategy improved the dispersion stability significantly. Qiu et al. [26] performed an experimental study on TC enhancement of PCM using modified fly ash. As a result, the LH and TC were improved by 51.7% and 67.7%, respectively.

Carbon-based nanostructures, including carbon nanotubes (CNTs), graphene, and carbon nanofibers (CNFs), offer unique properties that can significantly enhance the thermal properties of materials when incorporated into composites. Carbon-based nanostructures have exceptionally high thermal conductivities due to their one-dimensional (CNTs), twodimensional (graphene), or three-dimensional (CNFs) structures. When incorporated into composite materials, these nanostructures form conductive networks that facilitate the efficient transfer of heat, resulting in enhanced thermal conductivity of the composite. In applications where heat dissipation is critical, such as electronic devices and thermal management systems, carbon-based nanostructures can help dissipate heat more effectively. The high surface area and TC of nanostructures enable rapid heat transfer away from heatgenerating components, reducing operating temperatures and improving device reliability. Carbon-based nanostructures can be functionalized or integrated with PCMs to enhance their TES properties. The high surface area and thermal conductivity of nanostructures facilitate the efficient transfer of heat between the PCM and the surrounding environment, improving the charging and discharging rates of TES systems. Mayilvelnathan and Valan Arasu [27] studied the performance of a TES system using an erythritol PCM with graphene and reported that the TES system efficiency using NE-PCM was increased by 16.29% and 28.48% during stockpiling and retrieval, respectively. Bahiraei et al. [28] experimentally investigated the performance improvement of carbon-based NE-PCM. The graphite-based nanocomposites showed a significant TC improvement for 7.5 and 10 wt% concentration. The thermal efficiency of the eutectic PCM was improved by adding expanded graphite, as studied by Panda et al. [29]. With the 3 wt% of expanded graphite, the TC improved by 232.24%. N-octadecane and graphene nanoparticles at 2 wt% and 5 wt% were used by Zarma et al. [30] to develop a nano-enhanced PCM. The concentrated photovoltaic system's cell temperature was lowered using the generated nano-enhanced PCM, and the PCM containing 5 wt% graphene had a higher solar mean temperature. Lin et al. [31] created a composite PCM of palmitic acid, including graphene nanoplatelets (GNPs) and SiO₂ nanoparticles. According to the results, the composite PCM containing 5 wt% GNPs improved TC by 1.65 times and was advised for thermal energy storage. Sahan et al. [32] investigated the paraffin PCM containing carbon nanotubes and activated carbon. According to the findings, PCM containing carbon nanotubes and activated carbon had increased thermal conductivities by 34.1% and 39.1%, respectively. Moreover, PCM containing carbon nanotubes increased its thermal energy storage capacity by 9.6%, but activated carbon saw no change in this regard. Xu et al. [33] produced a PCM material including water, sodium polyacrylate, and multi-walled carbon nanotubes (MWCNTs). They found that adding 0.1 wt% of MWCNTs enhanced the TC by 19.17%. Bharathiraja et al. [34] investigated

the thermal properties of MWCNTs and SiO₂ nano-enhanced paraffin wax as a thermal storage medium. By increasing the MWCNT and SiO₂ concentration, the melting point was increased slightly, and thermal conductivity was increased. Xu et al. [35] prepared 1-Hexadeol NE-PCM using nano-TiO₂, nano-ZnO₂, nano-Ag, and graphene nanoplates and investigated their properties. Thermal conductivity was improved significantly by graphene nanoplates.

Fatty acids and fatty alcohols are of great interest among the different PCMs due to their good stability, high thermal storage density, and low corrosiveness. Exploring the improvement using various nanoparticles under different conditions and comparative investigations enables us to improve the thermal efficiency of a TES system. Both carbon-based and metallic nanoparticles contribute equally to improving the PCM thermal characteristics. In this study, two different NE-PCMs were developed using different concentrations of carbon-based MWCNTs and metallic CuO nanoparticles and fatty alcohol as a PCM. Their various thermophysical properties were comparatively examined using several characterization methods, and the findings are displayed. It is anticipated that the findings of this study will contribute to our understanding of the thermophysical modifications in PCMs due to the addition of nanoparticles and provide possible information on the NE-PCM with MWCNTs and CuO nanoparticles to be used as potential TES materials.

2. Materials and Methods

Lauryl alcohol (LA), also called 1-Dodecanol, is a long-chain fatty alcohol that appears to be a clear transparent liquid at 30 °C. It is immiscible in water but soluble in organic solvents. LA (purity: 99%) supplied by Sigma-Aldrich, India, was used as PCM. Copper oxide nanoparticles and MWCNTs supplied by Sigma-Aldrich, India, were used as nanoinclusions. Sodium dodecyl sulphate (SDS) (purity: 95%) from Loba Chemie, India, was employed to stabilize the nanoparticles in the PCM. The properties of the nanoparticles and surfactant are presented in Table 1. All the materials were used for the experiment as supplied without any further processing.

Material	Properties	
CuO nanoparticles	Size: 80 nm Surface area: 18 m ² g ⁻¹	
MWCNTs	Density: 0.28 gcm ⁻³ Length: 10–30 μm Outside diameter: 20–30 nm Inside diameter: 5–10 nm Purity: >95 wt%	
SDS	Density: 1.01 gcm ⁻³	

Table 1. Properties of the nanoparticles and surfactant.

The NE-PCM was formulated through physical mixing followed by an ultrasonic dispersion technique. Ultrasonic dispersion techniques are commonly employed for the synthesis and dispersion of nanoparticles within various matrices, including PCMs. When applied to nano-enhanced PCMs, ultrasonic dispersion techniques facilitate the uniform distribution of nanoparticles throughout the PCM matrix, resulting in enhanced thermal properties and performance. Ultrasonic dispersion involves the application of high-frequency acoustic waves (ultrasound) to a liquid medium containing nanoparticles and the PCM matrix. The ultrasonic waves create cavitation bubbles in the liquid, which collapse violently near the nanoparticles. This leads to localized high temperatures and pressures, promoting the dispersion of nanoparticles and their interaction with the PCM matrix. Ultrasonic dispersion techniques can be easily scaled up for industrial production, making them suitable for large-scale synthesis of nano-enhanced PCMs. As a first step in the process, LA was heated to 45 °C, and a suitable proportion of SDS surfactant was added to it and mixed

well through a magnetic stirrer and hot plate. Then, an appropriate amount of MWCNTs and CuO nanoparticles was added to the PCM and surfactant mixture and stirred for 30 min. Then, the mixture was probe sonicated at a frequency of 20 kHz for 1 h, and the temperature was maintained at a constant throughout the process. Finally, the resultant NE-PCM was cooled to room temperature. In the preparation process, 1, 3, and 5 wt% of nanoparticles were used, and the surfactant to the nanoparticle concentration was 1:10.

The thermophysical properties of the prepared NE-PCMs were examined through various characterization techniques. Differential scanning calorimetry (DSC) is a thermal analysis technique used to measure the heat flow into or out of a sample as a function of temperature or time. It is commonly employed in various fields to study the thermal properties of materials, including phase transitions, purity determination, reaction kinetics, and stability. The basic principle of DSC is based on comparing the heat flow to a sample and a reference material as they undergo a controlled temperature program. Any energy difference between the sample and reference is recorded as a function of temperature or time. A typical DSC instrument consists of a sample holder with two compartments: one for the sample and one for the reference material. Both compartments are placed in separate furnaces and are maintained at the same temperature throughout the experiment. As the temperature changes, the instrument measures the heat flow required to keep both compartments at the same temperature. The phase change temperatures and LHs were estimated through DSC analysis using NETZSCH DSC 204 F1 Phoenix. The equipment has a measurement temperature range of -180 °C to 700 °C, and the accuracy of the enthalpy measurement is <1%. The equipment was calibrated with indium as standard material before conducting the measurement. The measurement was performed between -10 and 60 °C at a heating rate of 10 °C.min⁻¹ under a nitrogen atmosphere. Before conducting the analysis, the equipment was calibrated with the standard reference material. Thermogravimetric analysis (TGA) is a thermal analysis technique used to study the weight changes in a sample as a function of temperature (or time) under controlled atmosphere conditions. It is widely used in various fields, including materials science, chemistry, pharmaceuticals, and environmental science, to investigate thermal stability, decomposition kinetics, composition, and purity of materials. The basic principle of TGA involves measuring the weight of a sample as it is subjected to a programmed temperature ramp or isothermal conditions. Any weight loss or gain is indicative of changes occurring within the sample. A typical TGA instrument consists of a sample holder, a balance, a furnace, and a temperature controller. The sample is placed in a crucible or pan, which is then loaded onto a balance. The balance continuously measures the weight of the sample as the temperature changes. The TGA was performed using NETZSCH STA 449 F3 Jupiter. The equipment has a measurement temperature range of -150 °C to 2400 °C, and the resolution of the mass loss measurement is $0.1 \ \mu g$. The measurement was performed from 25 to 280 °C at a heating rate of 10 °C.min⁻¹. The sample was placed in an open pan to estimate the decomposition temperature.

The KD2 Pro thermal property analyzer is an advanced instrument used for measuring the thermal properties of various solid and liquid materials. The KD2 Pro operates on the transient line source (TLS) technique. In this method, a needle-like probe, which serves as both a heat source and a temperature sensor, is inserted into the material of interest. A known amount of heat is applied to the probe for a short duration, and the resulting temperature change is measured. By analyzing the rate of temperature change over time, the KD2 Pro calculates the thermal properties of the material. The TC was determined through a KD2 Pro thermal property analyzer. The TC at the solid phase is measured using the SH-1 sensor with 10% measurement error, and at the liquid phase, the KS-1 sensor with 5% measurement error was used. During the measurement, the temperature of the sample was controlled by placing it in a double jacket container through which water from the constant thermal bath was circulated. The sample temperature was maintained at 10, 30, and 40 °C, and respective thermal conductivities were measured. The viscosity was measured using Anton Paar SVM 1001 viscometer. The equipment has a viscosity

measurement repeatability and reproducibility of 0.1% and 0.35%, respectively. The Anton Paar SVM 1001 viscometer is a sophisticated instrument used for measuring viscosity, a crucial property in various industrial applications. The SVM 1001 is designed to provide accurate and reliable viscosity measurements across a wide range of sample types and viscosities. The SVM 1001 viscometer operates on the rotational viscometry principle. It measures viscosity by rotating a spindle immersed in the sample fluid. The resistance to the spindle rotation is proportional to the viscosity of the fluid. By measuring the torque required to rotate the spindle at a constant speed, the instrument calculates the viscosity of the sample. In order to measure the viscosity of the NE-PCM in its liquid phase, the measurement was executed at 30 °C at all concentrations. The measurement error was about 0.1%, and it was repeated 3 times to ensure the repeatability.

3. Results

The DSC measurement results are shown in Figure 1a,b. The onset melting and solidification points for LA were 22 and 20 °C, respectively. The LH of melting and solidification were 217 and 213 Jg^{-1} , respectively. The DSC results of NE-PCMs are shown in Figure 1. The data consolidated from the DSC analysis of PCMs at different concentrations of MWCNTs and CuO nanoparticles are presented in Table 2. According to the findings, the phase change temperature was reduced to a smaller extent for all concentrations of two distinct nanoparticles, but the changes are not significant. Similarly, the LHs are also reduced with the inclusion of nanoparticles. Here, it is noted that the excessive inclusion of nanoparticles has an adverse effect on the heat storage capacity. The melting occurs as a single-step endothermic process. The solidification process occurs with two peaks due to the two-stage exothermic phase change process. This phenomenon is generally observed in long-chain fatty alcohols [36]. When PCMs with both nanoparticles were compared, more LH reduction was observed in the case of CuO than in the MWCNTs, but the difference is not significant. The drop in LH with the rise in nanoparticle concentration is due to the reduction in LA quantity per unit mass of NE-PCM, as the LH is directly proportional to the PCM mass. Moreover, the degree of supercooling decreased for the NE-PCM compared to the pure PCM. The nanoparticles improve the heat transfer and promote the crystallization process, which reduces supercooling.

Sample	Melting			Solidification			Degree of _ Supercooling
	Onset (°C)	Peak (°C)	LH (Jg ⁻¹)	Onset (°C)	Peak (°C)	LH (Jg ⁻¹)	(°C)
LA	22	27	217	20	16	213	11
LA + 1 wt% MWCNTs	21.5	26.1	212	20.5	17.5	210	8.6
LA + 3 wt% MWCNTs	21.6	26.3	202.5	20.7	17.4	201	8.9
					15.6		
LA + 5 wt% MWCNTs	21.6	26.5	195	20.7	17.5	193	9
					15.8		
LA + 1 wt% CuO	21.9 2	25 (200	20.3	17.1	- 198	8.5
		25.6			14.9		
LA + 3 wt% CuO	21.8	25.6	197	20.3	17.3	- 195	8.3
					14.4		
LA + 5 wt% CuO	21.9	25.8	193	20.4	17.3	- 191	8.5
					13.8		

Table 2. Results from the DSC measurement.

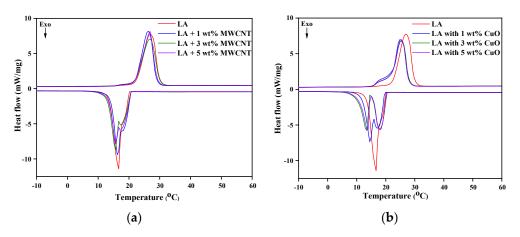


Figure 1. DSC thermograph of LA with nanoparticles (a) with MWCNTs and (b) with CuO.

The thermogravimetric analysis results are shown in Figure 2a,b. The findings elucidate that the decomposition temperature of the NE-PCMs has no significant alteration in both cases of MWCNTs and CuO nanoparticles. For NE-PCMs with MWCNT nanoparticles, there was a minor reduction in the decomposition temperature with the increase in MWCNT concentration. For LA, the decomposition occurs at 178 °C, and it was reduced to 162 °C for 5 wt% MWCNT concentration. In the case of CuO nanoparticles, the decomposition temperature somewhat increased to 182 °C. These changes are negligible and will not affect the phase change process.

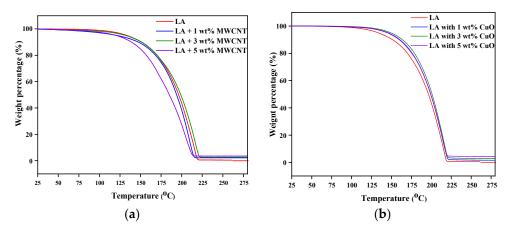


Figure 2. TGA result of LA with nanoparticles (a) with MWCNTs and (b) with CuO.

The TC estimated in both solid and liquid phases of the PCM and NE-PCMs are presented in Figure 3a,b. The TC was observed more in the solid phase than in the liquid phase due to the closely packed PCM and nanoparticles. Furthermore, the NE-PCM with MWCNTs showed higher TC than the NE-PCM with CuO nanoparticles. This is owing to the higher TC of MWCNTs. The nanoparticles enable microconvection between the PCM molecules and improve heat transfer. The maximum improvement in TC for the MWCNT-enhanced PCM was 82.6% and 79.1% in the solid and liquid phases, respectively. Similarly, the TC improved by 49.3% and 33.5% for the CuO-enhanced PCM in the solid and liquid phases, respectively.

The viscosity measurement results at different nanoparticle concentrations are shown in Figure 4. The viscosity upsurges with a rise in the concentration of nanoparticles. In the case of MWCNTs, the PCM impregnates into the tubular structure. The carbon network acts as a support structure for the PCM. Therefore, the viscosity was observed to be higher when compared to metallic CuO nanoparticles. The network structure of MWCNTs resists the flow of the NE-PCM, thereby increasing the viscosity. Furthermore, the surfactant added is responsible for the increase in viscosity. The surfactant is added in proportion to the nanoparticle concentration. With the increase in nanoparticle concentration, the surfactant quantity also increases. The surfactant forms a network-like structure that resists the PCM's flow and increases viscosity.

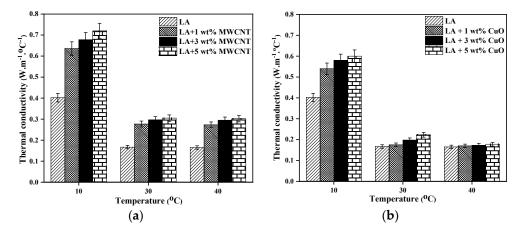


Figure 3. TC result of LA with nanoparticles (a) with MWCNTs and (b) with CuO.

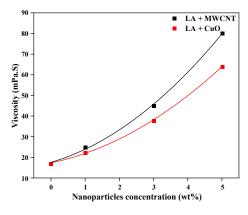


Figure 4. Viscosity of LA with nanoparticles.

4. Conclusions

In this work, two types of NE-PCMs were prepared using MWCNTs and CuO as nanoparticles and lauryl alcohol as the PCM. The findings elucidated that the LH was reduced with the rise in nanoparticle concentration, and more reduction was observed in the case of metallic nanoparticles than the MWCNTs. There is no significant alteration in the decomposition temperature. In the PCM with MWCNTs, the highest latent heat drop during melting and freezing was approximately 10.1% and 9.3%, respectively; in the PCM with CuO, the maximum latent heat drop was around 11% and 10.3%, respectively. The lowest supercooling for the PCM containing MWCNTs and CuO nanoparticles was 8.6 °C and 8.3 °C, respectively. The TC was improved by a maximum of 82.6% and 49.6%, in the case of MWCNT- and CuO-nanoparticle-based PCMs. Greater improvement was observed for carbon-based nanostructure. Furthermore, the viscosity was measured as higher for MWCNTs than CuO, and it tends to upsurge with the rise in nanoparticle concentration. Overall, it can be concluded that MWCNTs provide more favorable results for the thermal property enhancement of PCMs than CuO metallic nanoparticles. Therefore, an MWCNT-based NE-PCM could be a promising TES material for heat storage applications.

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Abbreviations

CSP	Concentrated solar power
CNT	Carbon nanotube
CNF	Carbon nanofiber
DSC	Differential scanning calorimetry
GNF	Graphene nanoplatelet
LH	Latent heat
LHTES	Latent heat thermal energy storage
LA	Lauryl alcohol
MWCNT	Multi-walled carbon nanotube
NE-PCM	Nano-enhanced phase change material
PCM	Phase change material
SDS	Sodium dodecyl sulfate
TES	Thermal energy storage
TC	Thermal conductivity
TGA	Thermogravimetric analysis
TLS	Transient line source

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