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Grau de Química

Treball Final de Grau

The effect of nanoparticles on the physicochemical and thermal properties of different organic eutectic mixtures.

L'efecte de les nanopartícules sobre les propietats fisicoquímiques i tèrmiques de diferents mescles eutèctiques orgàniques.

Lucas Tovar Pichardo

17/06/2003

Universitat de Barcelona

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Campus d'Excel·lència Internacional

La investigación es ver aquello que todos han visto, y pensar aquello que ninguno ha pensado.

Albert Szent-Györgyi

En primer lugar, quiero agradecer a Marc, Adela y Rebeca por su constante apoyo a lo largo de todo el proyecto, estando siempre presentes y ayudando en todo lo que estuvo en sus manos. Además, les agradezco enormemente la orientación y los consejos sobre los estudios y caminos profesionales a seguir.

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REPORT

IDENTIFICATION AND REFLECTION ON THE SUSTAINABLE DEVELOPMENT GOALS (SDG)

This work, entitled "The effect of nanoparticles on the physicochemical and thermal properties of different organic eutectic mixtures," significantly contributes to several Sustainable Development Goals (SDGs) (United Nations, 2025), especially two of the 5 "Ps" of the 2030 Agenda: Planet and Prosperity. This work investigates the improvement of organic Phase Change Materials (PCMs) through the addition of SiO_2 nanoparticles, aiming to enhance their thermal properties such as stability, heat capacity, and conductivity. These materials have significant potential in applications requiring energy efficiency and are therefore a major step toward the development of more sustainable and cleaner energy sources.

First, the project directly addresses SDG 7: Affordable and Clean Energy, specifically targets 7.3 (By 2030, double the global rate of improvement in energy efficiency) and 7.A (By 2030, enhance international cooperation to provide access to clean energy research and technology, including from renewable sources, energy efficiency, and advanced and cleaner fossil fuel technologies, and promote investment in energy infrastructure and clean technologies). PCMs offer great promise when it comes to thermal energy storage, as they allow for more efficient energy storage. The results obtained provide highly important data for the future development of new PCMs and their optimization for application in areas where clean and sustainable energy is required.

This project is also closely related to SDG 12: Ensure sustainable consumption and production patterns, and more specifically to target 12.2 (By 2030, achieve the sustainable management and efficient use of natural resources). This project uses biodegradable fatty acids and esters derived from renewable sources as the main material for PCMs. The choice of these compounds over other, more polluting ones supports the project's goal of minimising environmental impact.

In the long term, if the project could be developed on a large scale, it could have a significant impact on SDG 13: Take urgent action to combat climate change and its impacts, as it can be applied to new buildings, temperature-controlled transport, or space cooling. The implementation of PCMs could contribute to a significant reduction in CO_2 emissions from air conditioning, thus contributing to meeting international climate commitments.

Finally, it is also essential to highlight the relationship with SDG 9: Build resilient infrastructure, promote sustainable industrialization, and foster innovation, as this project contributes to research aimed at developing functional materials. The knowledge it generates can contribute to future research on the development of these materials and their technological and industrial applications in the energy sector.

This final degree project demonstrates that research into new materials can actively contribute to building a more sustainable and environmentally friendly future, while also driving progress in the field of phase-change materials and thermal energy storage.

CONTENTS

1. SUMMARY

This final project investigated the effect of SiO_2 nanoparticles on the thermal and physicochemical properties of various organic eutectic mixtures, with the aim of improving their properties for application in phase-change materials (PCMs) for energy applications. PCMs are compounds with a high capacity to store and release thermal energy during phase-change processes, making them key materials for achieving greater sustainability and energy efficiency in various sectors such as construction, electronics, and thermal storage systems.

A total of five binary eutectic mixtures consisting of fatty acids and esters were synthesized: capric acid/ethyl stearate (CA/ES), capric acid/ethyl myristate (CA/EM), capric acid/methyl palmitate (CA/PM), ethyl myristate/palmitic acid (EM/PA), and capric acid/palmitic acid (CA/PA). However, only three of them (CA/PA, CA/EM, and CA/ES) were fully characterized due to reagent availability issues. Eutectic concentrations were determined by differential scanning calorimetry (DSC) using the STAR^e Evaluation program, selecting those ratios that showed a single melting peak in the thermogram. From these, modified versions containing 1% by weight of SiO_2 nanoparticles were prepared and dispersed using ultrasound.

The samples were characterized using differential scanning calorimetry (DSC) to determine their enthalpy of fusion and heat capacity (c_p) and using the Hot Disk method to evaluate their thermal conductivity. The results indicate that the addition of nanoparticles increases c_p and thermal conductivity, while the enthalpy of fusion decreases. The latter property presents negative values, as it is an endothermic process in which the material absorbs heat to change phase. The main experimental limitation arose when attempting to measure the thermal conductivity of the CA/PA and CA/ES eutectics. Since their melting temperatures were close to the test temperature, the energy released by the sensor caused the compounds to melt, invalidating the results.

In conclusion, the results obtained show that the incorporation of SiO_2 nanoparticles modifies the thermal properties of the PCMs studied, suggesting a potentially positive impact on their efficiency and use in practical applications. Among these, the most notable uses are in construction materials, electronic components, and thermal energy storage systems, where improved thermal conductivity and thermal stability can provide significant benefits.

Keywords: Phase change materials, eutectics, latent heat, nanoparticles.

2. RESUMEN

En este TFG se ha investigado el efecto de nanopartículas de SiO_2 sobre las propiedades térmicas y fisicoquímicas de distintas mezclas eutécticas orgánicas, con el objetivo de mejorar sus propiedades para poder aplicarlo en materiales de cambio de fase (PCMs) para aplicaciones energéticas. Los PCMs son compuestos con gran capacidad de almacenamiento y liberación de energía térmica durante los procesos de cambio de fase, lo que los convierte en materiales clave para lograr una mayor sostenibilidad y eficiencia energética en diversos sectores como la construcción, la electrónica o sistemas de almacenamiento térmico.

Se sintetizaron un total de cinco mezclas eutécticas binarias formadas por ácidos grasos y ésteres: ácido cáprico/estearato de etilo (CA/ES), ácido cáprico/miristato de etilo (CA/EM), ácido cáprico/palmitato de metilo (CA/PM), miristato de etilo/ácido palmítico (EM/PA) y ácido cáprico/ácido palmítico (CA/PA), aunque solo tres de ellas (CA/PA, CA/EM y CA/ES) fueron caracterizadas completamente por problemas de disponibilidad de reactivos. La determinación de las concentraciones eutécticas se llevó a cabo mediante calorimetría diferencial de barrido (DSC) utilizando el programa STAR^e Evaluation, seleccionando aquellas proporciones que mostraban un único pico de fusión en el termograma. A partir de estas, se prepararon versiones modificadas con un 1 % en peso de nanopartículas de SiO_2, que fueron dispersadas mediante ultrasonidos.

Las muestras fueron caracterizadas mediante calorimetría diferencial de barrido (DSC) para determinar su entalpía de fusión y capacidad calorífica (c_p), y mediante el método Hot Disk para la evaluación de la conductividad térmica. Los resultados indican que la adición de nanopartículas produce un aumento en la c_p y la conductividad térmica, mientras que la entalpía de fusión disminuye. Esta última propiedad presenta valores negativos, ya que se trata de un proceso endotérmico en el que el material absorbe calor para cambiar de fase. La principal limitación experimental surgió al intentar medir la conductividad térmica de los eutécticos CA/PA y CA/ES, que al tener temperaturas de fusión próximas a la temperatura del ensayo, la energía liberada por el sensor provocaba la fusión de los compuestos, invalidando los resultados.

En conclusión, los resultados obtenidos muestran que la incorporación de nanopartículas de SiO_2 modifica las propiedades térmicas de los PCMs estudiados, lo que sugiere un impacto potencialmente positivo en su eficiencia y uso en aplicaciones prácticas. De entre estas, destacan los usos en materiales de construcción, componentes electrónicos y sistemas de almacenamiento de energía térmica donde la mejora de la conductividad térmica y la estabilidad térmica puede aportar grandes beneficios.

Palabras clave: Materiales de cambio de fase, eutécticos, calor latente, nanopartículas.

3. INTRODUCTION

3.1. GLOBAL ENERGY CONTEXT

The global energy system is undergoing a major transformation, driven by the urgent need to reduce greenhouse gas emissions and combat climate change. This transition is not possible without changes in energy consumption and production patterns to achieve energy sustainability. Despite the significant growth in renewable energy, fossil fuels still account for most of the global energy consumption, contributing significantly to greenhouse gas emissions (Gielen et al., 2019). This reliance on polluting energy sources hampers efforts to achieve carbon neutrality and limits progress toward sustainability.

Energy demand is expected to increase significantly, especially in areas experiencing significant population growth and industrialization (Creutzig et al., 2017). This significant energy demand must be balanced with environmental sustainability, and this requires the development of clean energy technologies and improvements in energy efficiency.

While solar and wind energy have seen significant improvements and cost reductions in recent years, their intermittent nature requires advances in energy storage and proper energy management to become a reliable resource (Zakeri & Syri, 2015). Therefore, research into innovative materials and systems to optimize energy generation and storage remains crucial to the global energy transition.

3.2. THERMAL ENERGY STORAGE

Thermal energy storage (TES) systems are extremely useful for storing energy for later use, significantly improving energy efficiency and flexibility. These systems absorb, store, and release energy in the form of heat when needed, allowing us to use energy at times other than its production, generating energy when it's cheaper and consuming it when prices are rising. This is especially useful in systems powered by renewable energy, where energy can be stored during favourable weather conditions, such as on sunny or windy days, and used later when environmental conditions are less than optimal (Zalba et al., 2003).

TES technologies are typically classified into three categories: sensible heat storage, latent heat storage, and thermochemical storage. Sensible heat systems store energy by increasing the temperature of a material, typically water or molten salts, while latent heat systems rely on a material's phase changes, such as melting and solidification, to store and release energy at almost constant temperatures. Thermochemical storage, on the other hand, involves reversible chemical reactions and can achieve very high energy densities (Dincer, 2002).

Among these, latent heat thermal energy storage stands out due to its high storage density and its ability to maintain very constant temperatures during energy emission and absorption processes (Lin et al., 2018) .The effectiveness of this system directly depends on the thermal conductivity, melting point, latent heat, and the materials used as Phase Change Materials (PCMs). Among the materials that can be used, PCMs based on organic products, such as fatty acids and esters, are especially attractive due to their thermal stability, chemical inertness, and non-corrosive nature (Carley & Konisky, 2020).

3.3. PHASE CHANGE MATERIALS (PCMS)

Phase change materials (PCMs) are substances that absorb or release large amounts of energy in the form of latent heat when undergoing a phase transition, typically from solid to liquid and vice versa. This phase transition occurs at a virtually constant temperature, making them excellent candidates for thermal regulation of temperature-sensitive systems. Based on their chemical composition, PCMs are generally classified as organic (paraffins, fatty acids, etc.), inorganic (salt hydrates, etc.), organic-inorganic hybrids or eutectic mixtures, each with its own advantages and disadvantages (Lv et al., 2022) (Kenisarin & Mahkamov, 2007). Table 1 shows the most notable properties of each type of PCM.

Table 1. Advantages and disadvantages of the different types of PCM.

PCM type	Advantages	Disadvantages
Organic	Stable cycling, low supercooling, non-corrosive	Low thermal conductivity, flammable
Inorganic	High latent heat, good thermal conductivity	Corrosive, phase segregation, supercooling
Organic-Inorganic hybrids	Good heat storage performance, low supercooling, versatility	Lax packaging, liquid leakage
Eutectic mixtures	Adjustable melting points, sharp phase changes	Complex formulation

These characteristics allow PCMs to passively perform efficient thermal regulation in various applications, such as building HVAC, solar energy systems, and electronic device cooling. Their high energy density and virtually isothermal behavior during phase change make PCMs highly effective for managing thermal loads (Zhang et al., 2007).

3.3.1. Advantages and challenges of organic PCMs

Organic PCM, such as paraffins and fatty acids, excel in thermal energy storage applications due to their thermochemical properties. These materials typically feature high latent heat values, chemical stability, corrosion resistance, and minimal subcooling effects, all of which are essential for thermal cycling and a longer PCM lifespan. Furthermore, organic PCMs typically have a wide melting point range, allowing for a wide range of possibilities depending on the application temperature, allowing them to be used in both residential and industrial settings (Zalba et al., 2003).

Despite these advantages, organic PCMs also present several limitations that restrict their performance. One of the main disadvantages is their relatively low thermal conductivity, which limits the heat transfer rate and therefore the charging and discharging cycles of the thermal storage systems. Furthermore, some organic PCMs, such as paraffins, are flammable, limiting their use due to the safety issues they could generate on a large scale. Another notable issue is the fact that they sometimes experience phase segregation and volume variations during melting-solidification cycles, which can affect their thermal properties and system reliability over time (Sarier & Onder, 2012).

3.4. EUTECTIC MIXTURES

Eutectic mixtures are systems of two or more homogeneously mixed compounds that, when mixed in a characteristic ratio, melt and solidify at a single, precise temperature known as the eutectic point. This melting point is typically lower than that of the individual components of the mixture and can therefore be applied in thermal energy storage (TES) due to their well-defined phase transition and relatively high latent heat. These mixtures have significant advantages such as high thermal stability and a well-defined phase transition. Furthermore, biodegradable, non-toxic, and low-cost organic mixtures can be synthesized, facilitating their use in sustainable energy storage systems. (Zhou et al., 2019).

The thermal properties of eutectic mixtures, particularly their enthalpy of fusion, heat capacity, and transition temperature, are key factors determining the effective application of the compound. One of the significant advantages of these materials is their ability to store large amounts of energy within a narrow temperature range, making them highly useful in applications requiring precise thermal regulation. Additionally, the melting and solidification cycles are typically reproducible after multiple thermal cycles, indicating good thermal stability (Zhou et al., 2019).

3.5. ADDITION OF NANOPARTICLES

In recent years, the incorporation of nanoparticles into PCMs has attracted significant interest. This approach aims to overcome the limitations of conventional PCMs, mainly the low thermal conductivity and stability after several phase change cycles. Among the various nanomaterials, silica (SiO_2) nanoparticles are a good resource due to their chemical stability, low cost, and good compatibility with organic compounds (Kibria et al., 2015).

The inclusion of SiO_2 nanoparticles in organic eutectic mixtures offers the opportunity to improve the thermal properties of PCMs used for latent heat energy storage. The nanoparticles facilitate heat distribution within the material thanks to their large surface area and favourable heat transport properties. This translates into greater heat absorption and release during phase transitions (Yu et al.,

2021). In addition to thermal conductivity, nanoparticles can also significantly improve the thermal properties of PCMs, such as heat capacity, latent heat of fusion, and phase change kinetics. Furthermore, they can reduce supercooling by acting as nucleating agents, resulting in longer PCM lifespans due to more stable and repeatable thermal cycles (Yuan et al., 2023).

3.6. APPLICATIONS

Phase change materials (PCMs) have garnered significant interest due to their ability to store and release large amounts of latent heat during phase transitions. This property makes PCMs suitable for use in a wide variety of applications where thermal regulation, energy efficiency, and temperature stability are critical.

In the building and construction sector, PCMs are widely used to moderate indoor temperatures. When PCMs are incorporated into walls, ceilings, or floors, they help maintain a stable temperature by absorbing excess heat during the day and releasing heat during colder periods, such as at night. In this way, the energy demand required by active systems such as heating or air conditioning is reduced, thus contributing to more sustainable energy consumption patterns (Madad et al., 2018).

In electronic devices, the trend toward component miniaturization and increased power generates temperature problems that can reduce device lifespan and performance. PCMs act as passive thermal buffers by temporarily absorbing excess heat during operation and releasing it when temperature is not a concern, ensuring thermal stability. Their use is being explored in heat sinks, high-performance processors, electronics, and LEDs (Bianco et al., 2022).

The automotive industry has also found applications where PCMs would be very useful, especially in battery thermal management systems (BTMS) for electric vehicles (EVs). These vehicles typically operate with lithium-ion batteries, which are very sensitive to temperature fluctuations. Exceeding safe temperature ranges can lead to battery degradation, reduced lifespan, or even road safety risks. PCMs integrated into battery modules act as thermal buffers, absorbing the heat generated during charging and discharging cycles (Liu et al., 2020).

In the textile industry, PCMs can be incorporated into fibres and coatings to regulate body temperature by storing excess body heat and releasing it when the temperature drops, thus maintaining the body at a stable temperature without sudden fluctuations. This technology paves the way for the development of smart clothing for outdoor use, sportswear, and even medical garments. One innovative application is the use of trimethylolethane-based PCMs in leather coatings for thermosensitive garments (Reyes et al., 2024) (Mondal, 2008).

TES is one of the most important applications for PCMs, especially in systems that use intermittent renewable sources, such as solar or wind, or simply look to increase their energy efficiency. In these contexts, PCMs can store excess thermal energy and release it when needed, helping to offset imbalances between supply and demand (Jayathunga et al., 2024).

The versatility of PCMs in these fields demonstrates their potential to overcome multiple challenges in energy, comfort, and performance. Continued advances in this field of research, particularly in eutectic formulations and the integration of nanoparticles, are expected to further expand the applications and efficiency of PCMs.

4. OBJECTIVES

The main objective of this work is to investigate the effect of incorporating nanoparticles of silicon dioxide (SiO_2) on the thermal properties of organic eutectic mixtures used as phase change materials (PCMs). By synthetising some binary mixtures from long-chain fatty acids and esters, this study wants to evaluate how the presence of nanoparticles affects the different thermal parameters such as melting enthalpy (ΔH_m), specific heat capacity (c_p), transition temperature and thermal stability. To achieve the main goal of this work, the following specific objectives have been developed:

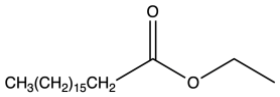
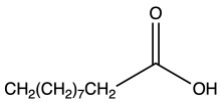
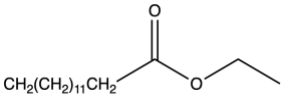
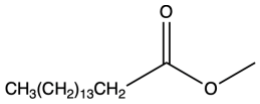
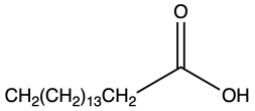
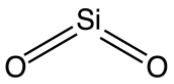
1. Synthesize different eutectic systems based on bibliographic research and laboratory availability.
2. Verify the eutectic composition with the differential scanning calorimetry (DSC).
3. Select the most appropriate eutectic systems to incorporate 1 wt.% SiO_2 nanoparticles.
4. Evaluation of the thermal properties, including melting enthalpy (ΔH_m), specific heat capacity (c_p), transition temperature …
5. Evaluation of the chemical structure (FTIR)
6. Apply rigorous statistical analysis methods to experimental data to validate the reliability of results, quantify variability, and support conclusions throughout the study.

5. EXPERIMENTAL SECTION

5.1. MATERIALS

In this work, various chemical compounds were used as raw materials for the preparation of eutectic mixtures with and without nanoparticles. To ensure reproducibility, their key properties, such as chemical name, CAS number, molecular formula, melting point, purity, supplier, and chemical structure, are summarized in Table 2. This information is essential for understanding the composition and quality of the materials used in experiments.

Table 2. Summary of the chemical compounds used in this study, including chemical name, CAS number, molecular formula, melting point, purity, supplier, and chemical structure. *Structures drawn by the author using ChemDraw.*

Reagent name	CAS number	Molecular formula	Melting point [ºC]	Purity [%]	Supplier	Chemical structure
Ethyl stearate (ES)	111-61-5	$C_{20}H_{40}O_2$	~ 32 - 38	> 97	Sigma-Aldrich	
Capric acid (CA)	334-48-5	$C_{10}H_{20}O_2$	~ 31 - 32	> 98	Sigma Aldrich	
Ethyl myristate (EM)	124-06-1	$C_{16}H_{32}O_2$	~ 22 - 24	> 98	Fisher Scientific	
Methyl palmitate (MP)	112-39-0	$C_{17}H_{34}O_2$	~ 28 - 34	> 97	Sigma-Aldrich	
Palmitic acid (PA)	57-10-3	$C_{16}H_{32}O_2$	~ 62 - 64	> 98	Sigma-Aldrich	
Silica nanoparticles	7631-86-9	SiO_2	>1600	99.5	Sigma-Aldrich	

5.2. PREPARATION OF EUTECTIC MIXTURES

To determine the concentrations of the different eutectics, a first approximation was made through a literature search. Different values were found for the molar ratios of the different eutectic mixtures, and from these, different batches with concentrations close to the theoretical value were prepared to determine the location of the eutectic point. The detailed tables (5-9) with the theoretical and experimental masses for each composition are provided in Appendix 1. All batches were prepared following the same procedure. First, the corresponding proportions of each organic compound were weighed on a precision balance with a resolution of 0.001 g in a 50 mL beaker with a final sample mass of 10 g. The beaker was then placed in a water bath at a temperature above the melting point of the eutectic mixture and stirred for 10 minutes (Figure 1), counting from the moment the entire contents of the beaker were melted. Once the stirring time was over, the sample was transferred to a plastic vial and stored in a refrigerator to avoid temperature fluctuations that

could affect the PCM. Table 3 summarizes the concentrations of the different eutectic mixtures and their respective stirring temperatures.

Table 3. Overview of the eutectic mixtures prepared in this study, including the molar ratios reported in the literature, the different molar ratios tested experimentally, and the treatment temperatures applied to each eutectic mixture.

Eutectic mixture	Bibliographic molar ratio	Tested molar ratios	Temperature during stirring [ºC]	Physical state (25 °C)
CA / ES	X = 0.34 (ES)	X = 0.32 0.34 0.36 (ES)	50	Solid
CA / PA	X = 0.83 (CA)	X = 0.82 0.83 0.84 0.85 0.86 (CA)	70	Solid
CA / ME	X = 0.64 (EM)	X = 0.62 0.64 0.66 (EM)	25	Liquid
CA / MP	X = 0.395 (MP)	X = 0.375 0.395 0.415 (MP)	45	Liquid
PA / MP	X = 0.895 (MP)	X = 0.875 0.895 0.915 (MP)	60	Solid

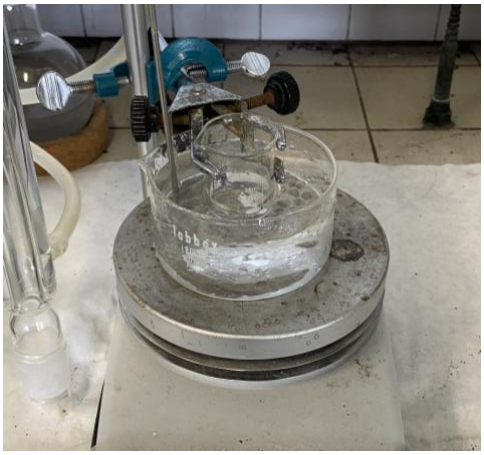


Figure 1. Melting and stirring of the fatty acid eutectic mixture in a water bath on a hot plate.

5.3. CONFIRMATION OF EUTECTIC CONCENTRATIONS BY DIFFERENTIAL SCANNING CALORIMETRY (DSC)

To determine the correct eutectic composition, we use a differential scanning calorimeter (DSC 822e Star3+, from Mettler Toledo) with STAR^e evaluation software, which enables us to identify the eutectic point based on the heat flow curve. To prepare the samples for analysis, a small portion of each sample is placed in 100 μL aluminium crucibles with an approximate mass of 10 mg, determined on a Mettler Toledo balance with an accuracy of 0.01 mg. All crucibles are sealed with a perforation in the lid to prevent overpressure. For each eutectic, a sample is prepared for each concentration. An additional sample is also included for each eutectic at the concentration indicated in the bibliography. The test was performed with a heating rate of 2 K/min and a nitrogen flow of 50 ml/min.

5.4. INCORPORATION OF SiO_2 NANOPARTICLES

To incorporate the SiO_2 nanoparticles, we work in fume hoods with a gas extraction system and use personal safety measures such as gloves, goggles, and an FFP3 mask. These safety measures are important because SiO_2 nanoparticles, with a size between 10 and 20 nm, can remain suspended in the air as an invisible cloud for long periods, thereby increasing the risk of involuntary inhalation. These nanoparticles can cause various lung problems after repeated exposure, and for this reason, we must take specific safety measures.

To incorporate the nanoparticles into the validated eutectic mixtures, we first weigh 1% of the nanoparticles by mass. As in this case the samples are 5 g, we add a weight of 50 mg of SiO_2 nanoparticles. Next, we must properly disperse the nanoparticles; in this case, this was done with an ultrasonic homogeniser. Two of the prepared PCMs are in the solid phase at room temperature, so sonication was carried out in a thermostatically controlled bath at 40°C. During sonication, the vial is covered with aluminium foil to prevent moisture from entering during the procedure, and 15-second ultrasonic cycles with 5-second pauses are performed for a total

of 10 minutes. The liquid samples at room temperature were subjected to the same procedure, this time without the use of a water bath.

5.4. SAMPLE PREPARATION FOR CHARACTERIZATION

5.4.1. Differential Scanning Calorimetry (DSC)

To determine the specific heat capacity (c_p) and enthalpy of fusion (ΔH_m) of each sample, approximately 10 mg of each formulation was weighed into 100 µL aluminium crucibles using a Mettler Toledo balance with an accuracy of 0.01 mg. The crucibles were sealed with perforated aluminium lids to prevent overpressure during temperature changes. All analyses were performed under a nitrogen atmosphere to prevent oxidation.

For c_p determination, a common heating ramp was used for all samples, from −15 °C to 50 °C at a rate of 10 K/min. To obtain accurate c_p values using DSC, the use of a reference with well-defined thermal properties is essential. In this context, sapphire is used as a reference standard due to its thermal stability and its highly accurate tabulated c_p.

For enthalpy measurement, all samples followed the same temperature program design, consisting of a heating-cooling-heating cycle with a heating and cooling rate of 2 K/min. Each sample was analysed within its specific temperature range, depending on its eutectic composition:

- CA/PA: 10 °C to 35 °C
- CA/EM: −10 °C to 20 °C
- CA/ES: 5 °C to 35 °C

Each cycle consisted of an isothermal step at the initial temperature for 5 minutes, followed by heating to the upper limit of the range at a rate of 2 K/min. After reaching this temperature, the sample was held isothermally for 3 minutes to ensure a complete phase transition. It was then cooled back to the initial temperature at the same rate, held for 3 minutes again, and subjected to a second identical heating ramp, followed by a final isotherm of 3 minutes. The enthalpy of fusion was calculated from the second heating cycle to reduce the influence of thermal history and improve reproducibility.

5.4.2. Infrared Spectroscopy (IR)

For Infrared spectroscopy (IR) characterisation, we use the Spectrum Two from PerkinElmer, in which we place the different samples on the detection lens and obtain the corresponding IR spectra. For this characterisation, we will place a eutectic sample without nanoparticles and one with nanoparticles to distinguish the bands corresponding to the stretching of the O-Si-O bond. To correctly locate this band, we have a spectrum of a pure SiO_2 sample.

5.4.3. Determination of thermal conductivity

In this study, thermal conductivity measurements were performed using the Transient Planar Source (TPS) technique with a Hot Disk instrument. The sensor, composed of a planar nickel coil encapsulated in polyimide, was positioned so that it was completely covered by the sample in a sandwich configuration. During the test, a constant electric current is applied, and the temperature increase over time is recorded under controlled conditions (23°C), allowing the software to determine the material's thermal conductivity. This method is particularly well-suited for the characterization of low-conductivity organic systems.

5.4.3.1. Liquid sample preparation

For liquid samples, in this case, the CA/EM eutectic measurements are carried out in a teflon conductivity cell. The objective is to ensure that the detector is completely covered by the liquid, without the presence of air bubbles that could interfere with the measurement. Once the assembly was complete, tests were performed to determine the correct measurement parameters, modifying time and power. Time values of 2.5 s, 5 s, and 10 s were used, and power values of 20, 40, 50, and 60 mW. After several tests, it was determined that the optimal measurement parameters were 5 seconds and 40 mW. Three measurements were taken for each batch, with a 3-minute rest period between measurements.

Figure 2. Setup for measuring thermal conductivity of the liquid eutectic sample.

Figure 3. Setup for measuring thermal conductivity of the liquid eutectic sample, covered with an insulating lid.

5.4.3.2. Solid sample preparation

For solid-state samples, in this case, the AC/AP and AC/EE eutectics, a different assembly is performed. First, all batches are placed in an oven at a temperature of 40°C until they completely melt. The contents are then poured into square silicone molds and allowed to cool until solidified and stabilised at laboratory temperature. This is done to ensure a flat surface and correct batch-to-batch reproducibility. Once the samples have the desired shape, we proceed with the assembly. We place each sample in a Petri dish and the conductivity sensor on the flat side as we observe in Figure 4 below. A thermal insulator is placed on top of the sensor to prevent interference as we can see in Figure 3. Once the tests were performed to establish the correct measurement parameters, we observed that the results were not as expected, giving us much higher conductivity values than expected. Therefore, we decided to run more tests, but this time placing two samples in a "sandwich" shape, covering both sides of the sensor. Tests were performed with time values of 20, 30, 40, 80 and 120 s and powers of 5, 10, 20 and 30 mW. We were able to observe that the best results were obtained with a time of 80 s and a power of 5 mW. Tests were performed with pairs of batches with and without nanoparticles, with three measurements for each sample and 10 minutes between each measurement.

Figure 4. Setup for measuring thermal conductivity of the solid eutectic samples.

6. RESULTS AND DISCUSSION

6.1. CONFIRMATION OF EUTECTIC COMPOSITION

The determination of eutectic concentrations was the initial step in the characterization of the PCMs. This process was carried out using differential scanning calorimetry (DSC), the data of which were processed and interpreted using STARe evaluation software. The eutectic composition of each binary system was identified by analysing the obtained thermograms, where species with a single, well-defined peak were at or very close to the eutectic point. The thermograms used for this analysis are provided in Appendix 2 (Figures 14–17). Conversely, samples showing two distinct peaks indicated compositions outside the eutectic range, where phase changes occur in two separate stages due to phase segregation.

In Figure 5, we can find one representative example of the concentration determination process is presented, illustrating how the eutectic composition was identified for MP/PA binary mixture.

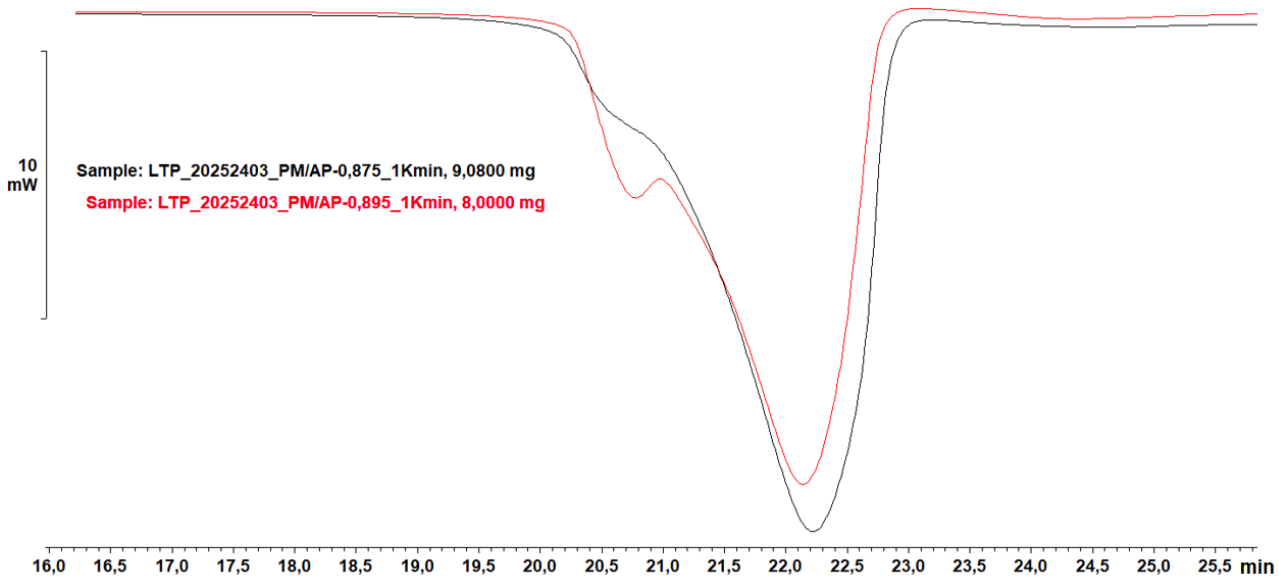


Figure 5. DSC thermograms of MP/PA at two different concentrations (X = 0.875 (MP) and X = 0.895 (MP)), obtained using STARe software.

The DSC thermogram presented above reveals a clear difference between the two analysed concentrations of the MP/PA mixture. The sample with a molar ratio of X = 0.875 (MP) shows a second peak to the left of the main peak, indicating the presence of a second phase transition. This indicates that the mixture is not in the correct proportion since the presence of this second peak rules out the possibility of being at the eutectic concentration (Quant et al., 2023). In contrast, the thermogram corresponding to X = 0.895 (MP) shows a single peak with only a slight shoulder, indicating a more uniform phase transition. This slight shoulder tells us that we are not yet exactly at the eutectic concentration but that we are much closer at a concentration of X = 0.895 (MP).

Initially, the molar ratios of four eutectic systems were prepared and determined: capric acid/ethyl myristate (CA/EM), capric acid/ethyl stearate (CA/ES), methyl palmitate/capric acid (MP/CA), and methyl palmitate/palmitic acid (MP/PA). Due to limited reagent availability, the MP/CA and MP/PA systems were excluded from further study. Instead, a new eutectic system based on capric acid/palmitic acid (CA/PA) was prepared. The determination of the eutectic concentration was performed for the binary systems; however, the determination of conductivity, c_p, and enthalpy was limited to the three selected eutectics: CA/EM, CA/ES, and CA/PA.

6.2. Differential Scanning Calorimetry (DSC) results

6.2.1. Heat capacity (c_p)

Specific heat capacity (c_p) is a key parameter when performing the thermal analysis of a material, as it reflects its ability to absorb and store heat energy. In the context of phase-change materials (PCMs), c_p is a very important parameter, as it determines the efficiency of thermal energy storage systems (Dzindziora et al., 2021). The following section presents the c_p values obtained for the three eutectic systems studied, with and without the incorporation of SiO_2 nanoparticles. The results are displayed using box plots (Figures 6-8), where we can more precisely distinguish the distribution and variability of the results across multiple batches.

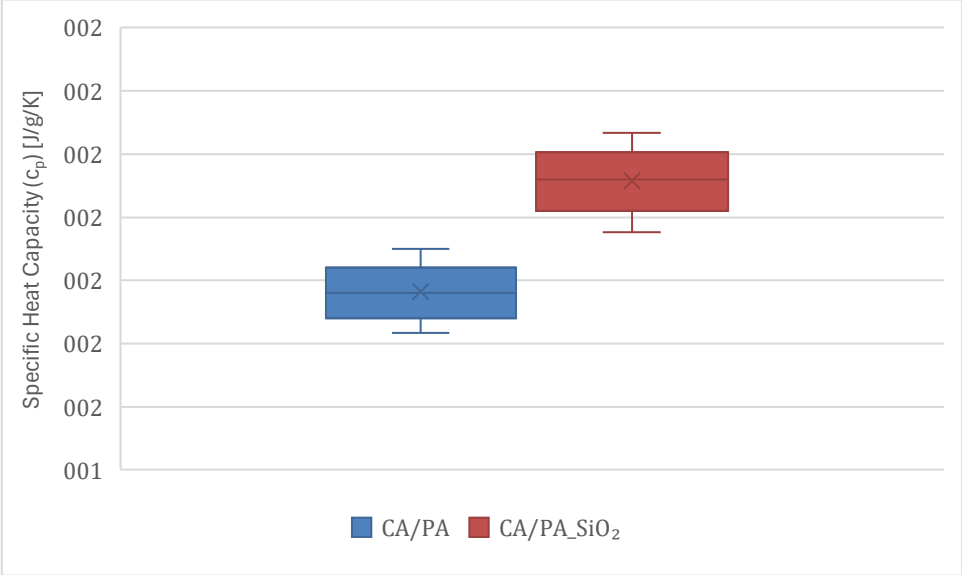


Figure 6. Box plot showing the distribution of specific heat capacity (c_p) values for the capric acid / palmitic acid eutectic system, comparing samples with and without 1 wt% SiO_2 nanoparticles. Each box includes data from different batches.

The outliers in Figure 7, although represented in the box plot to show their presence, are not considered in the calculation of the median or quartiles. Therefore, although outliers are relevant from a variability perspective, they do not affect the central parameters of the data set.

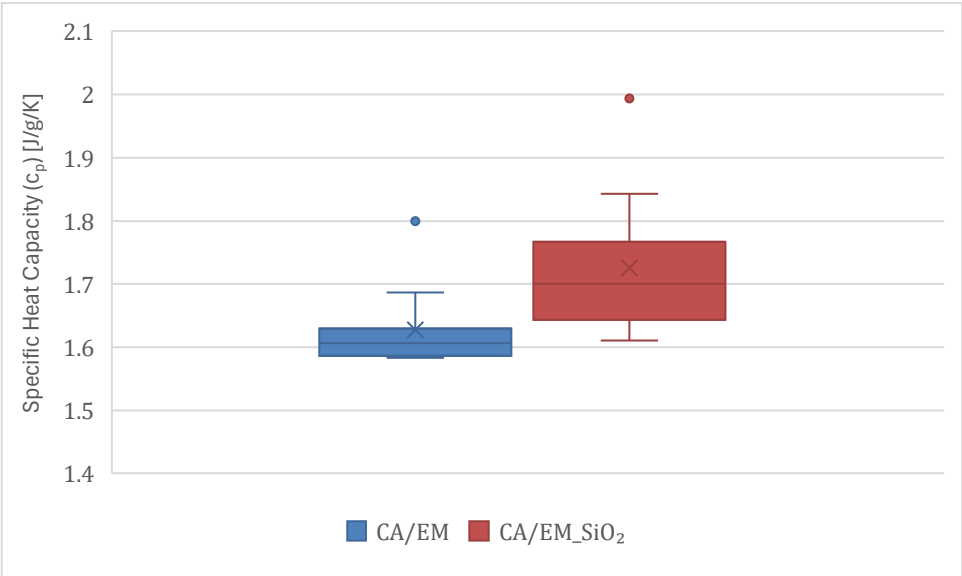


Figure 7. Box plot showing the distribution of specific heat capacity (c_p) values for the capric acid / ethyl myristate eutectic system, comparing samples with and without 1 wt% SiO_2 nanoparticles. Each box includes data from different batches.

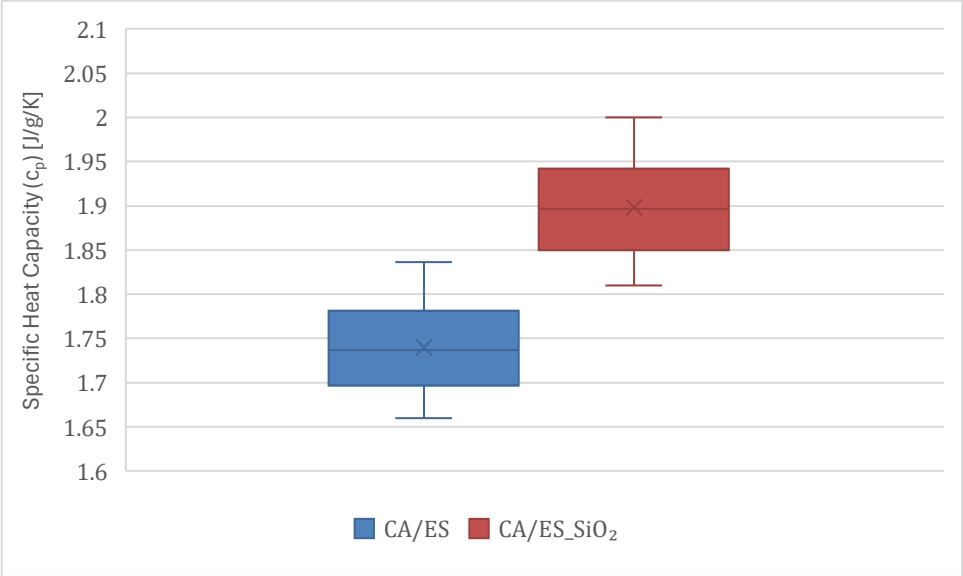


Figure 8. Box plot showing the distribution of specific heat capacity (c_p) values for the capric acid / ethyl stearate eutectic system, comparing samples with and without 1 wt% SiO_2 nanoparticles. Each box includes data from different batches.

The specific heat capacity (c_p) results for the three eutectic systems follow the same trend: the samples modified with 1 wt.% SiO_2 nanoparticles exhibited higher c_p values than the unmodified batches. This trend can be observed in the box plots shown above (Figures 6-8), where the distribution of c_p values for the batches containing nanoparticles is located higher in the figures. The increased c_p indicates a greater capacity of the eutectic mixtures with SiO_2 nanoparticles to store thermal energy, which can be attributed to the presence of SiO_2 nanoparticles, which, thanks to their large surface area and thermal conductivity, act as heat transfer enhancers, thus increasing the heat capacity value. These results support the hypothesis that the incorporation of nanoparticles can positively influence the thermal properties of organic eutectic PCMs.

6.2.2. Enthalpy of fusion (ΔH_m)

The enthalpy of fusion is a key parameter in the evaluation of phase change materials (PCMs) as it directly reflects the material's ability to store and release thermal energy during the melting and solidification processes. For PCMs, a higher enthalpy value indicates a greater latent heat storage capacity per unit mass, which is crucial for applications such as thermal energy storage (Lin et al., 2018). In DSC analysis, the enthalpy change is represented by the area under the endothermic or exothermic peak corresponding to the phase transition. The enthalpy values plotted are negative, corresponding to the melting process. Positive values are plotted because we treat the magnitude as an absolute value.

The enthalpy of fusion (ΔH_m) was determined for the three eutectic mixtures, both in their pure form and after the incorporation of 1 wt.% silica nanoparticles (SiO_2).

The results are presented using box plots in Figures 9-11, corresponding to each eutectic system. Each box plot summarizes the ΔH_m values from independent batches, allowing a clear visualization of the variability and differences between samples with and without nanoparticles. Each set of results for a property is represented by a box enclosed by three horizontal lines: the lower one indicates the first quartile (Q1), corresponding to 25% of the distribution; the upper one represents the third quartile (Q3), corresponding to 75%; and the middle line marks the median, i.e., the second quartile (Q2), which accounts for 50% of the variability in the data. The box thus shows the interquartile range, which encompasses the central set of values excluding the 25% furthest from the median. The vertical lines extending from the box indicate the minimum and maximum values considered within an acceptable range, defined as 1.5 times the difference between Q3 and Q1 (Svobodova-Sedlackova et al., 2022). Values outside this range are considered outliers.

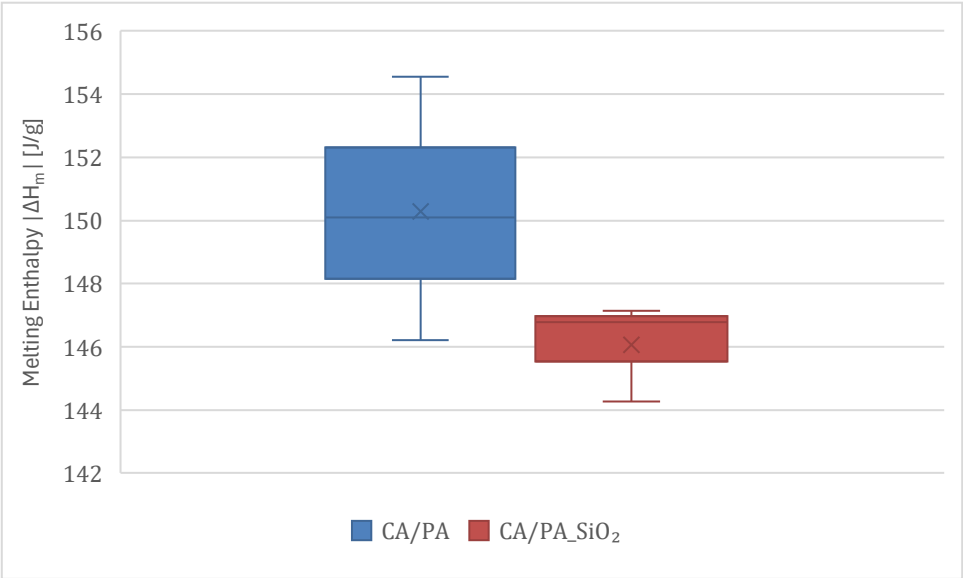


Figure 9. Box plot of melting enthalpy (ΔH_m) for the CA/PA eutectic mixture. Negative ΔH_m values are shown as absolute values. Data represent three independent batches for both the pure eutectic and the 1 wt% SiO_2 nanoparticle formulation.

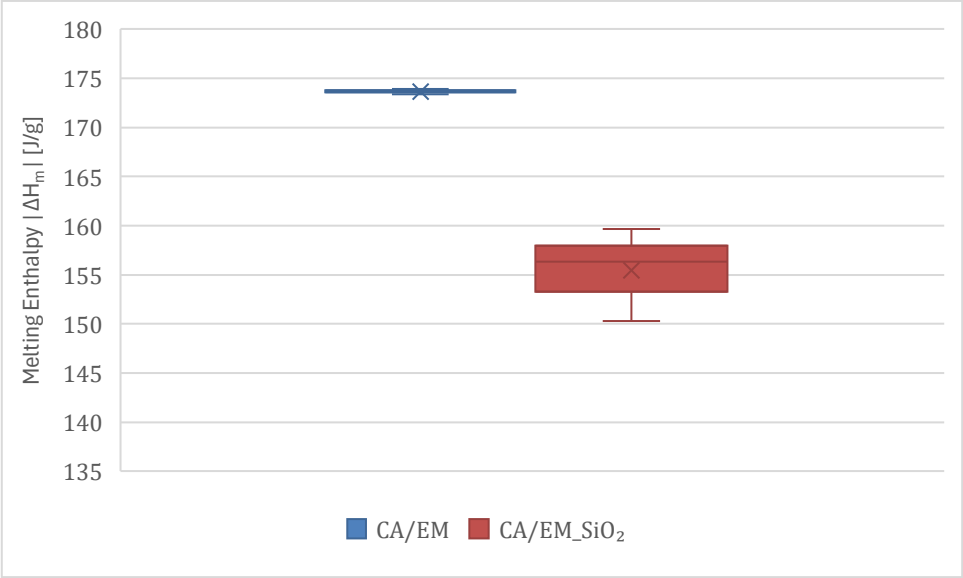


Figure 10. Box plot of melting enthalpy (ΔH_m) for the CA/EM eutectic mixture. Negative ΔH_m values are shown as absolute values. Data represent three independent batches for both the pure eutectic and the 1 wt% SiO_2 nanoparticle formulation.

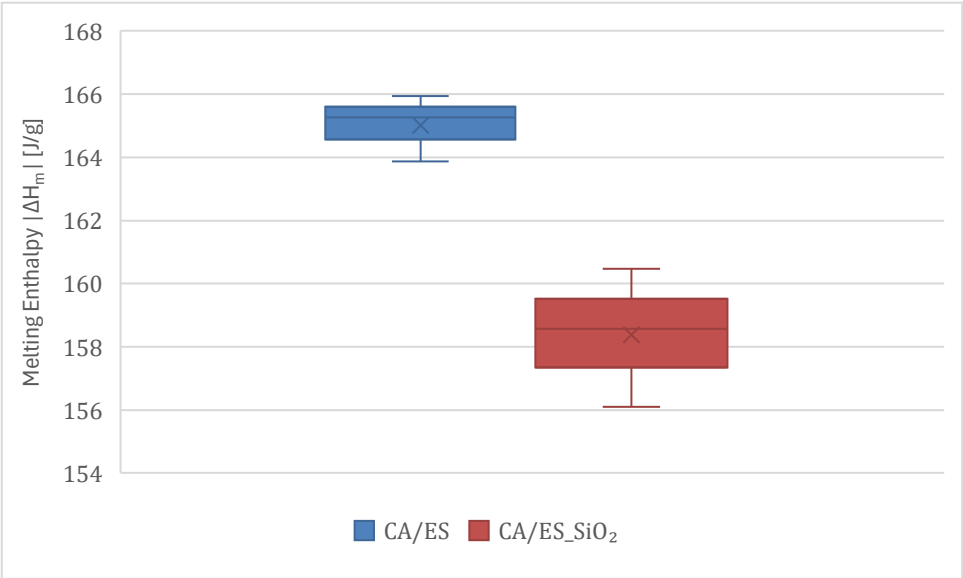


Figure 11. Box plot of melting enthalpy (ΔH_m) for the CA/ES eutectic mixture. Negative ΔH_m values are shown as absolute values. Data represent three independent batches for both the pure eutectic and the 1 wt% SiO_2 nanoparticle formulation.

In all three eutectic systems, the samples containing SiO_2 nanoparticles exhibited lower enthalpy values, indicating a lower latent heat of fusion. This suggests that the presence of nanoparticles could interfere with the crystallization process, potentially reducing molecular packing or hindering nucleation. These structural effects may indicate a lower degree of crystallization of the sample, thus decreasing the energy required to melt it.

6.3. THERMAL CONDUCTIVITY RESULTS

Determining thermal conductivity is a crucial aspect in the characterization of phase change materials (PCMs), as it indicates the rate at which thermal energy is absorbed and released during phase transitions. PCMs are materials with low intrinsic thermal conductivity and therefore typically exhibit inefficient heat transfer, limiting their effectiveness in thermal energy storage systems (Lin et al., 2018). Therefore, analysing thermal conductivity is essential to evaluate the performance of the different synthesized PCMs and the influence of adding SiO_2 nanoparticles.

Thermal conductivity measurements were only successfully obtained for the eutectic mixture composed of capric acid and ethyl myristate (CA/EM). At the measurement temperature (23°C), this eutectic mixture was completely liquid, allowing for stable and reproducible data acquisition using the method used. The results are presented in the box plot below, which shows the distribution of thermal conductivity values for this sample, with and without the addition of silica nanoparticles.

In contrast, the other two eutectic mixtures, CA/PA and CA/ES, could not be properly characterized under the same conditions. Their melting temperatures were too close to the ambient measurement temperature, and therefore, the energy released by the sensor during the measurement process caused local melting of the samples instead of uniform heating. This phase transition interfered with the thermal response, giving us inconsistent and unrepresentative results. For this reason, these two samples were excluded from the present analysis.

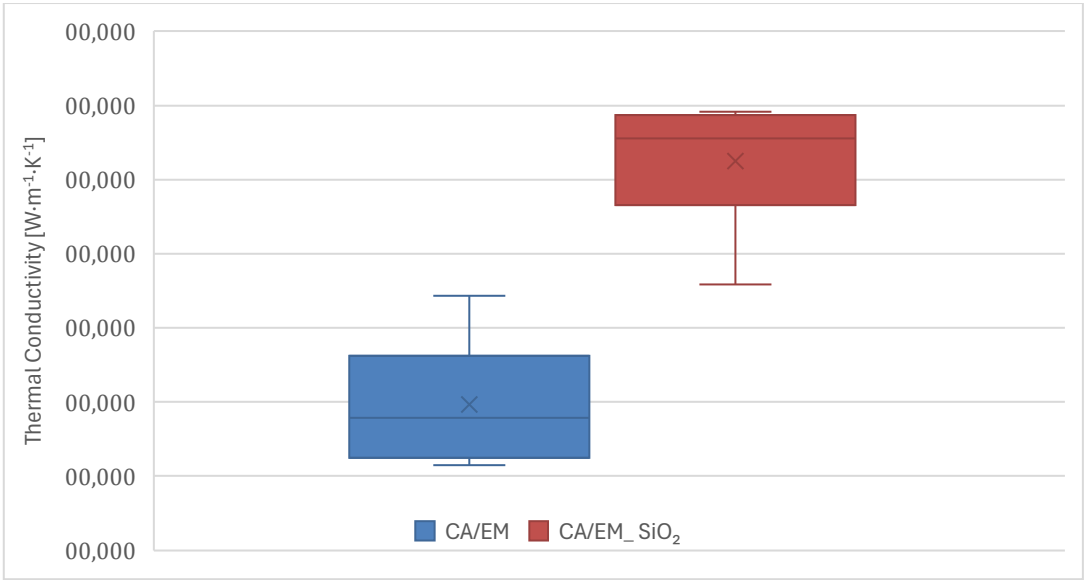


Figure 12. Box plot comparison of the thermal conductivity [$W \cdot m^{-1} \cdot K^{-1}$] between CA/EM and CA/EM_ SiO_2

As shown in Figure 12, the eutectic mixture with silica nanoparticles (CA/EM_SiO_2) exhibits a clear increase in thermal conductivity compared to the sample without nanoparticles (CA/EM). The median value for the nanoparticle-enhanced formulation is significantly higher, and the entire interquartile range shifts upward. This suggests that the presence of 1 wt % SiO_2 improves the heat transfer properties of the eutectic system.

6.4. FTIR Characterization of Eutectic Systems with and without SiO_2 Nanoparticles

To further evaluate the molecular interactions of the eutectic mixture, FTIR spectra were recorded for three samples: the eutectic mixture of capric acid and ethyl myristate (CA/MS), the same eutectic system with 1 wt.% SiO_2 nanoparticles (CA/MS_SiO_2), and pure SiO_2 nanoparticles. The resulting spectra are presented in Figure 13.

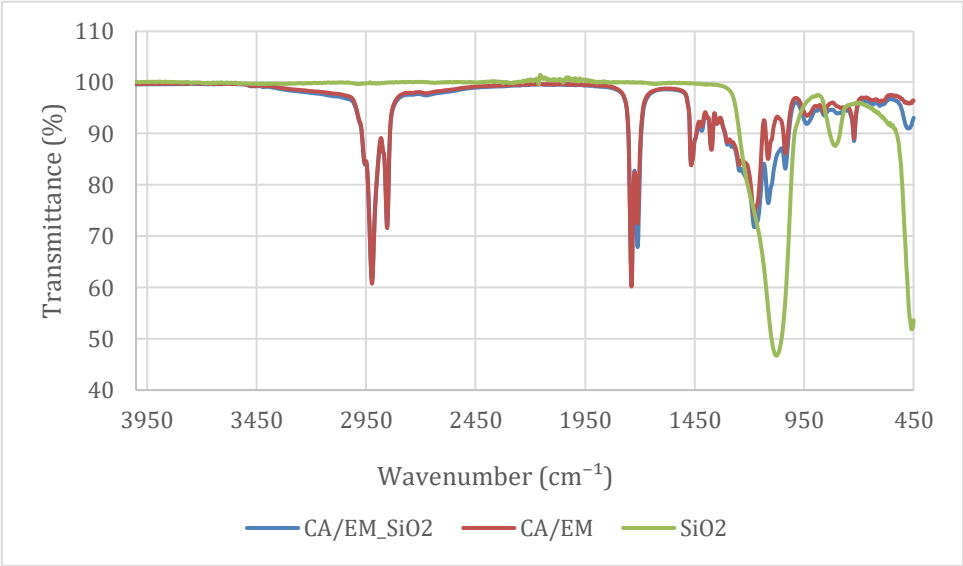


Figure 13. FTIR spectra of the CA/MS eutectic mixture, the CA/MS composite with 1 wt.% SiO_2 nanoparticles, and pure SiO_2 nanoparticles.

For ease of interpretation, Table 4 summarizes the main characteristic vibrational modes observed for capric acid, ethyl myristate, and SiO_2. These include stretching and bending vibrations associated with the functional groups present.

Table 4. Summary of the main vibrational modes observed in the FTIR spectra of capric acid, ethyl myristate, and SiO_2 nanoparticles. Characteristic wavenumbers (cm^{-1}) and their corresponding vibrational assignments are listed, highlighting the functional groups.

Wavenumber [cm^{-1}]	Assignment	Present in
~ 2923	C–H stretching	Capric acid / Ethyl myristate
~ 2854	O-H stretching	Capric acid
~ 1736 and 1713	C=O stretching	Capric acid / Ethyl myristate
~ 1415	C-H bending	Capric acid / Ethyl myristate
~ 1070	Si–O–Si asymmetric stretching	SiO_2
~ 810	Si–O–Si symmetric stretching	SiO_2
~ 450	Si–O–Si rocking	SiO_2

Capric acid and ethyl myristate are long-chain fatty compounds that display characteristic vibrational modes in the infrared region. The most prominent bands include strong C=O stretching vibrations around 1699 cm^{-1} for the carboxylic acid group in capric acid and around 1740 cm^{-1} for the ester group in ethyl myristate. Both compounds also have intense C-H stretching bands in the 2920–3000 cm^{-1} region due to the aliphatic chains, as well as C-H bending vibrations near 1465 and 1375 cm^{-1}. In ethyl myristate, C-O stretching modes appear between 1000–1300 cm^{-1}, further confirming the presence of the ester function. These bands allow clear identification and differentiation between the acid and ester structures (Ali et al., 2020).

After this comparison, it is worth highlighting that, in the spectrum of the CA/MS_SiO_2 composite, the absorption bands around 1070, 810, and 450 cm^{-1} (Gunde, 2000), corresponding to the asymmetric and symmetric stretching vibrations and the rocking of the O-Si-O bond, respectively, appear more intense compared to the pure CA/MS system. This increase in intensity coincides with the corresponding feature in the spectrum of pure SiO_2, confirming the presence of silica nanoparticles in the composite.

These spectral modifications indicate that the silica nanoparticles are not only physically embedded in the eutectic matrix but also contribute clearly distinguishable vibrational features in the FTIR profile. The overlap and intensification of the Si-O bands confirm their incorporation and provide indirect evidence of their dispersion at the molecular level.

7. CONCLUSIONS

This study has contributed to a better understanding of the behaviour of PCMs based on binary organic mixtures when SiO_2 nanoparticles are added. The main objective of the study was to evaluate the effect of adding nanoparticles on the thermal properties of the PCMs, more specifically to see how it affects the enthalpy of fusion, the heat capacity (c_p), and the thermal conductivity.

The eutectic systems used to characterize and quantify their thermal properties were capric acid/palmitic acid (CA/PA), capric acid/ethyl stearate (CA/ES), and capric acid/ethyl myristate (CA/EM). These were selected after DSC analysis of five original combinations. The eutectic concentration was identified by analysing the melting peaks, where a single, narrow, well-defined peak indicates a stable binary eutectic. All eutectic systems were analysed by DSC, but only the last three underwent characterization to determine thermal parameters due to material limitations. Furthermore, thermal conductivity measurements could only be performed for the AC/MS eutectic, as the melting points of the other two eutectics were very close to the working temperature. This caused the heat flux generated by the sensor to cause melting rather than heating the sample, compromising data reliability.

The results show that the addition of 1 wt.% silica nanoparticles produced changes in all thermal parameters. Specifically, the mixtures with the presence of nanoparticles showed a reduction in the enthalpy of fusion, along with increases in specific heat capacity and thermal conductivity. These results are highly relevant, as higher c_p and conductivity can significantly improve heat storage and heat transfer, respectively, while the decrease in enthalpy, although a drawback, can be compensated for through system design. All measurements were performed in triplicate for each sample, reinforcing the reproducibility of the data obtained.

These results are relevant to various energy applications, such as thermal management in electronic components, building materials, battery systems for electric vehicles, and textile engineering. Due to the improvement provided by nanoparticles in the thermal response of PCMs, certain intrinsic limitations of PCMs, such as slow heat transfer and low thermal efficiency, can be overcome.

A possible improvement of these components in future projects would be the microencapsulation of these PCMs, with the aim of improving their stability, shape retention, and compatibility with composite systems. Although this idea is beyond the scope of this work, it represents a logical continuation to expand the applicability of PCMs.

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APPENDICES

APPENDIX 1: THEORETICAL AND EXPERIMENTAL MASSES FOR EUTECTIC MIXTURES

Table 5. Theoretical and experimental masses of CA and ES at various molar fractions in the CA/ES eutectic system.

Molar ratio	Organic compound	Theorical mass	Experimental mass
X=0,32 (ES)	CA	5,394 g	5,395 g
	ES	4,606 g	4,608 g
X=0,34 (ES)	CA	5,169 g	5,173 g
	ES	4,831 g	4,834 g
X=0,36 (ES)	CA	4,949 g	4,946 g
	ES	5,050 g	5,049 g

Table 6. Theoretical and experimental masses of EM and CA at various molar fractions in the EM/CA eutectic system.

Molar ratio	Organic compound	Theorical mass	Experimental mass
X=0,62 (EM)	CA	2,917 g	2,917 g
	EM	7,083 g	7,075 g
X=0,64 (EM)	CA	2,742 g	2,742 g
	EM	7,258 g	7,276 g

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Table 7. Theoretical and experimental masses of MP and CA at various molar fractions in MP/CA eutectic system.

Molar ratio	Organic compound	Theorical mass	Experimental mass
X=0,375 (MP)	MP	4,851 g	4,851 g
	CA	5,149 g	5,150 g
X=0,395 (MP)	MP	5,062 g	5,062 g
	CA	4,938 g	4,937 g
X=0,415 (MP)	MP	5,269 g	5,269 g
	CA	4,731 g	4,732 g

Table 8. Theoretical and experimental masses of MP and PA at various molar fractions in the MP/PA eutectic system.

Molar ratio	Organic compound	Theorical mass	Experimental mass
X=0,875 (MP)	MP	8,807 g	8,816 g
	PA	1,193 g	1,191 g
X=0,895 (MP)	MP	8,999 g	8,995 g
	PA	1,001 g	1,008 g

Table 9. Theoretical and experimental masses of CA and PA at various molar fractions in the CA/PA eutectic system.

Molar ratio	Organic compound	Theorical mass	Experimental mass
X=0,82 (CA)	CA	3,767 g	3,768 g
	PA	1,233 g	1,230 g
X=0,83 (CA)	CA	3,832 g	3,831 g
	PA	1,168 g	1,170 g
X=0,84 (CA)	CA	3,895 g	3,897 g
	PA	1,105 g	1,129 g
X=0,85 (CA)	CA	3,960 g	3,959 g
	PA	1,040 g	1,039 g
X=0,86 (CA)	CA	4,025 g	4,024 g
	PA	0,975 g	0,979 g

APPENDIX 2: DSC THERMOGRAMS OF EUTECTIC SYSTEMS AT DIFFERENT COMPOSITIONS

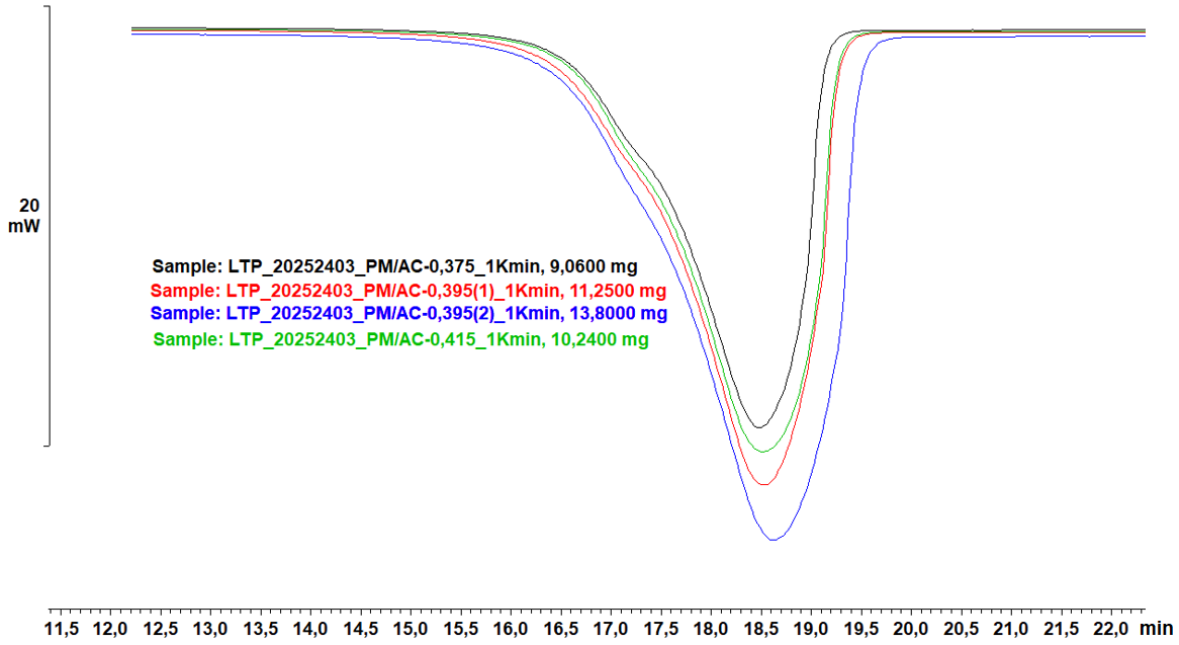

Figure 14. DSC thermograms of the MP/CA eutectic system, at molar fractions of MP (X = 0.375, 0.395 —replicates 1 and 2—, and 0.415).

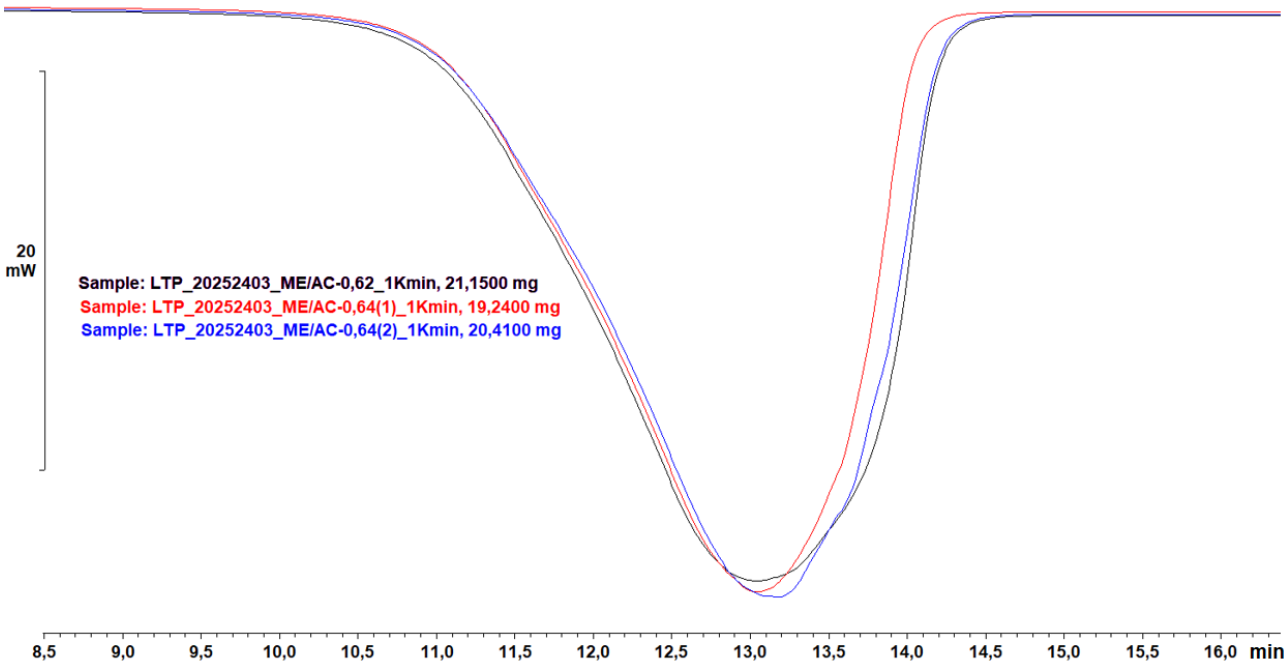

Figure 15. DSC thermograms of the EM/CA eutectic system, at molar fractions of EM (X = 0.62 and 0.64 —replicates 1 and 2—).

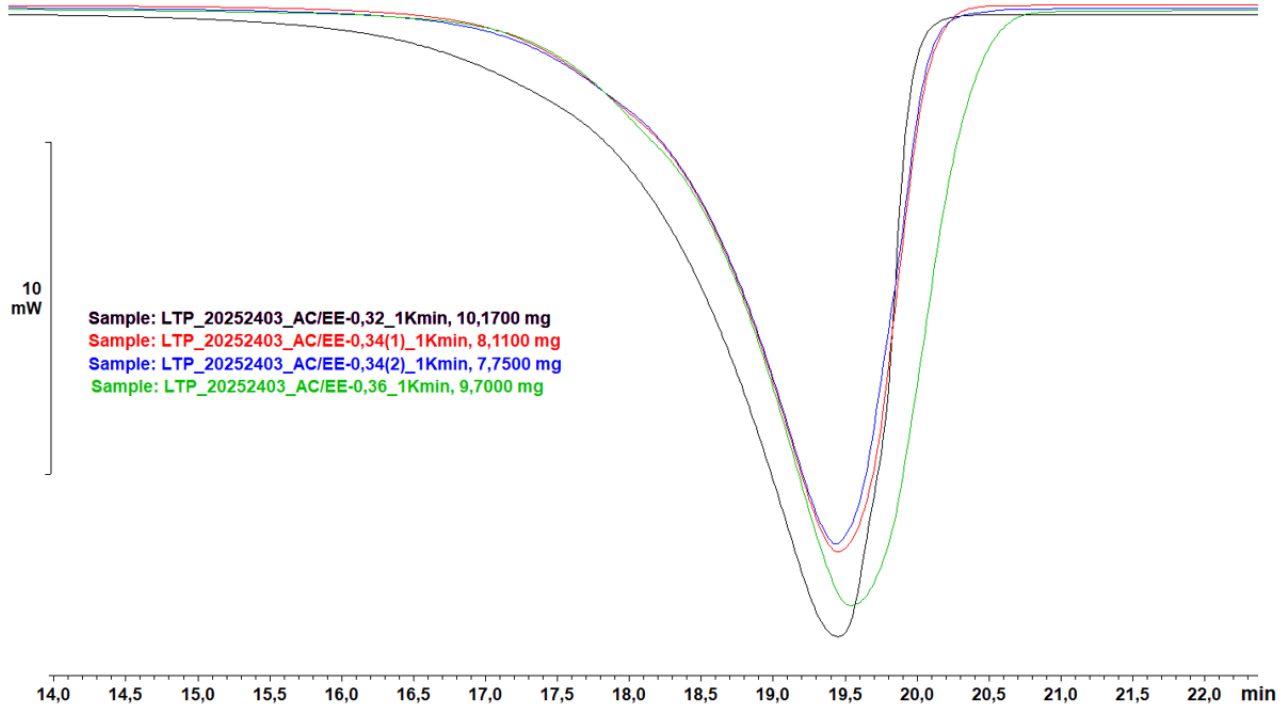

Figure 16. DSC thermograms of the CA/ES eutectic system, at molar fractions of ES (X = 0.32, 0.34 —replicates 1 and 2—, and 0.36).

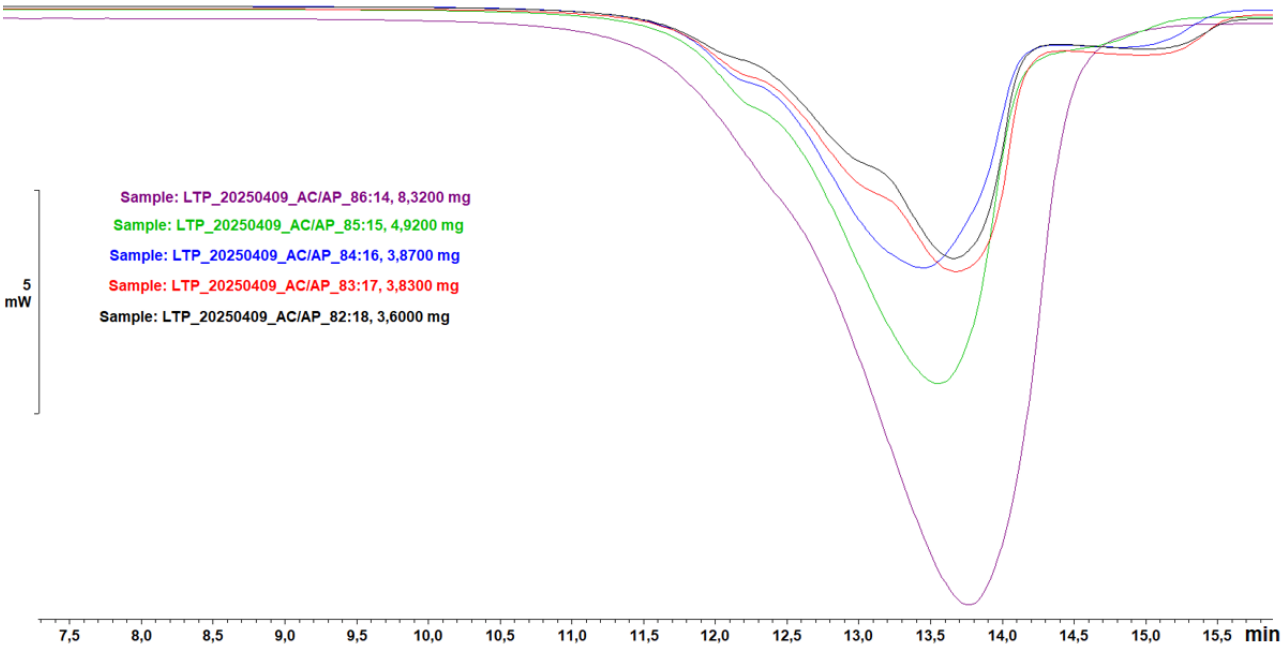

Figure 17. DSC thermograms of the CA/PA eutectic system, at molar fractions of CA (X = 0.82, 0.83, 0.84, 0.85, and 0.86).