APPLICATION AND EVALUATION OF THE PROPERTIES OF NANOENCAPSULATED PCMS IN TEXTILES

https://doi.org/10.56238/sevened2025.021-011

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ABSTRACT

The development of smart fabrics, which actively act in the situations to which they are subjected, has been the subject of research in several industrial areas. Among these materials, textiles containing phase change materials (PCMs) stand out, which act on thermal regulation through the solid-liquid transition, absorbing and releasing heat during this process. This study aimed to select, encapsulate and evaluate the performance of PCMs applied to textiles, aiming at thermal control.

Waxes were selected as phase change materials, encapsulated by means of the interfacial polymerization technique, forming microcapsules capable of retaining PCM during thermal cycles. Thermal characterization was performed by differential scanning calorimetry (DSC), while particle morphology and size distribution were analyzed by scanning electron microscopy (SEM) and laser diffraction. Subsequently, the encapsulated PCMs were applied to a flat fabric, being evaluated for thermal conductivity and heat storage capacity through techniques such as infrared thermography and measurement of thermal conductivity and effusivity.

The results indicated that textile articles containing encapsulated PCMs had slower heat absorption, greater thermal retention capacity and gradual release of stored heat, when compared to fabrics without PCMs. The results demonstrate that the incorporation of encapsulated PCMs into fabrics represents a promising strategy for applications where thermal control is desirable, such as functional clothing and technical materials.

Keywords: Phase Change Materials (PCM). Encapsulation. Smart Fabrics. Thermal Regulation.

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INTRODUCTION

The search for functional materials with controlled thermal properties has driven research in the textile area, especially in the development of smart fabrics that provide greater comfort and energy efficiency. Among these technologies, phase *change materials* (PCMs) stand out, capable of absorbing, storing and releasing heat during the solid-liquid phase transition, promoting more efficient thermal control. This ability allows fabrics impregnated with PCMs to act as passive thermal regulators, delaying sudden temperature variations and ensuring greater comfort for the user (DAS et al., 2024).

The incorporation of PCMs in textiles has been studied for applications in various areas, such as sportswear, protective clothing, military uniforms, passive air conditioning materials, and textiles for hospital use (VARSHNEY et al., 2024). These applications are justified by the need for greater thermal stability in environments of varying temperatures, where the use of conventional materials is not able to provide the same level of thermal comfort. In addition, the use of PCMs reduces the energy demand in air conditioning systems, which makes them attractive for the sustainable development of new materials.

While the benefits of PCMs are widely recognized, their direct application in fabrics faces technical challenges. One of the main problems is the stability of PCMs during thermal cycles, as in the phase transition, many materials undergo leakage and degradation, reducing their long-term effectiveness (Iqbal et al., 2019). To overcome these limitations, the encapsulation of PCMs has become a widely studied strategy, allowing the protection of the active material within a polymeric matrix, ensuring greater stability and extending its useful life (PAUSE, 2010).

Among the encapsulation techniques, microencapsulation, macroencapsulation and encapsulation in hollow fibers stand out. Microencapsulation is the most widely used in the textile industry due to its ability to produce reduced-size capsules, improving adhesion to fabrics and allowing more efficient control of heat release (SHAHID et al., 2024). The most common polymers for the formation of the capsules include polyurethane, polystyrene, and polyacrylate, which ensure good mechanical strength and thermal stability. Another promising technique is the incorporation of PCMs into textile fibers, either by coating or by adding phase-change materials directly into the fiber structure during their manufacture (LIMA, 2013).

In addition to thermal stability, the efficiency of encapsulated PCMs also depends on their capacity to store and release heat. Materials with high enthalpy of fusion are preferred as they can store more heat energy without compromising tissue integrity. Recent studies

demonstrate that the appropriate choice of PCM and encapsulation technique can directly impact the durability and thermal effectiveness of modified fabrics (XU et al., 2024).

The increasing demand for innovative materials in the textile industry has driven the development of new methods to enhance the application of PCMs in fabrics. Research in this area seeks solutions that maximize the thermal stability of encapsulated PCMs, improve adhesion to the textile substrate, and optimize the performance of the final material (HOSSAIN ET AL., 2023). In addition, there is a continuous effort to develop economically viable industrial processes that allow the large-scale production of these materials, without compromising their thermal and mechanical properties (TRAN et al., 2019).

This study aims to select the materials to be encapsulated, encapsulation and evaluation of the performance of PCMs applied to cotton fabrics to improve their thermal properties. Two waxes were analyzed as phase change materials, encapsulated by the interfacial polymerization technique. The thermal performance of the encapsulated materials was characterized by differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and laser diffraction. In addition, the encapsulated PCMs were applied to the tissue, and their thermal efficiency was evaluated by infrared thermography techniques and measurements of conductivity and thermal effusivity.

The proposal of this research aims to contribute to the advancement of knowledge about smart textile materials, promoting the development of innovative products with optimized thermal regulation capacity. With this, it is expected to enable the application of encapsulated PCMs in the textile industry, favoring their use in various segments, from technical clothing to materials aimed at thermal comfort in controlled environments.

METHODOLOGY

MATERIALS

Cetyl Palmitate (CPC) was supplied by the company Dhaymers, is a fatty ester, formed by the reaction between palmitic acid and cetyl alcohol, being a waxy, white, light-odor solid, commonly used in cosmetics and pharmaceutical products that presented a melting point between 48 °C and 53 °C, with a melting enthalpy of 174.97 J/g and was selected for its high heat storage capacity and thermal stability in heating and cooling cycles.

Ucuúba butter (CMU), was supplied by the company Beraca, is a vegetable fat extracted from the ucuúba seed, with a melting range between 43 °C and 47 °C and a melting enthalpy of 80.38 J/g, being selected because it is of natural origin, has a thermal

range compatible with human comfort, has good chemical stability and sustainable application as an alternative to synthetic waxes.

Methyl methacrylate (BASF BRASIL S.A – technical grade) as a monomer and hydroxyethylmethacrylate – HEMA (Aldrich) as a functional monomer, colloidal silica (NALCO BRASIL, 30 % w/w in water) as a protective colloid and potassium persulfate (VETEC, 99%) as an initiator of the polymerization reaction were also used.

The Phase *Change Materials* (PCMs) used in the preparation of the microcapsules were selected according to the properties and conditions presented in Table 1.

Table 1 - Properties and conditions evaluated for the selection of Phase *Change Materials* (PCMs)

Properties	Conditions evaluated in the selection
Melting temperature range compatible with textile applications	The transition temperature of the materials is close to the temperature of the human skin, preferably between 28 °C and 45 °C, in order to provide thermal comfort to the user.
High enthalpy of fusion	Materials with high latent heat storage capacity were prioritized, as they ensure greater thermal efficiency.
Thermal and chemical stability	The selected waxes were to withstand multiple thermal cycling without degradation or loss of performance.
Encapsulation Process Compatibility	The materials must have good emulsification characteristics and allow the formation of stable microcapsules by interfacial polymerization.
Commercial availability and affordability	The choice also considered the economic feasibility for industrial applications.

Source: the author.

Thermal analysis of the waxes was also performed to select the materials to be used as PCMs. The waxes were analyzed by differential scanning calorimetry (DSC), using the DSC/TGA 1 Star System (Mettler-Toledo) equipment. The samples, with a mass between 5 and 10 mg, were stored in open alumina crucibles. The method consisted of 3 cycles on a heating ramp from 25 °C to 100 °C, followed by cooling from 100 °C to 25 °C, both at a rate of 5 °C/min, under nitrogen flow at 50 mL/min, and this equipment was provided by the Laboratory of Chemical Processes and Particle Technology of IPT.

The acquisition and analysis of the data were carried out by the STARe software and the graphs obtained allowed the identification of the melting (endothermic) and solidification (exothermic) peaks, characterizing the phase transition of the materials. The enthalpy of fusion was determined by the area under the endothermic peak in the heating curve, allowing to estimate the amount of heat absorbed by the waxes during the phase transition. Under these conditions, Cetyl Palmitate (CPC) and ucuúba butter (CMU) waxes were selected.

PRODUCTION OF MICROCAPSULES

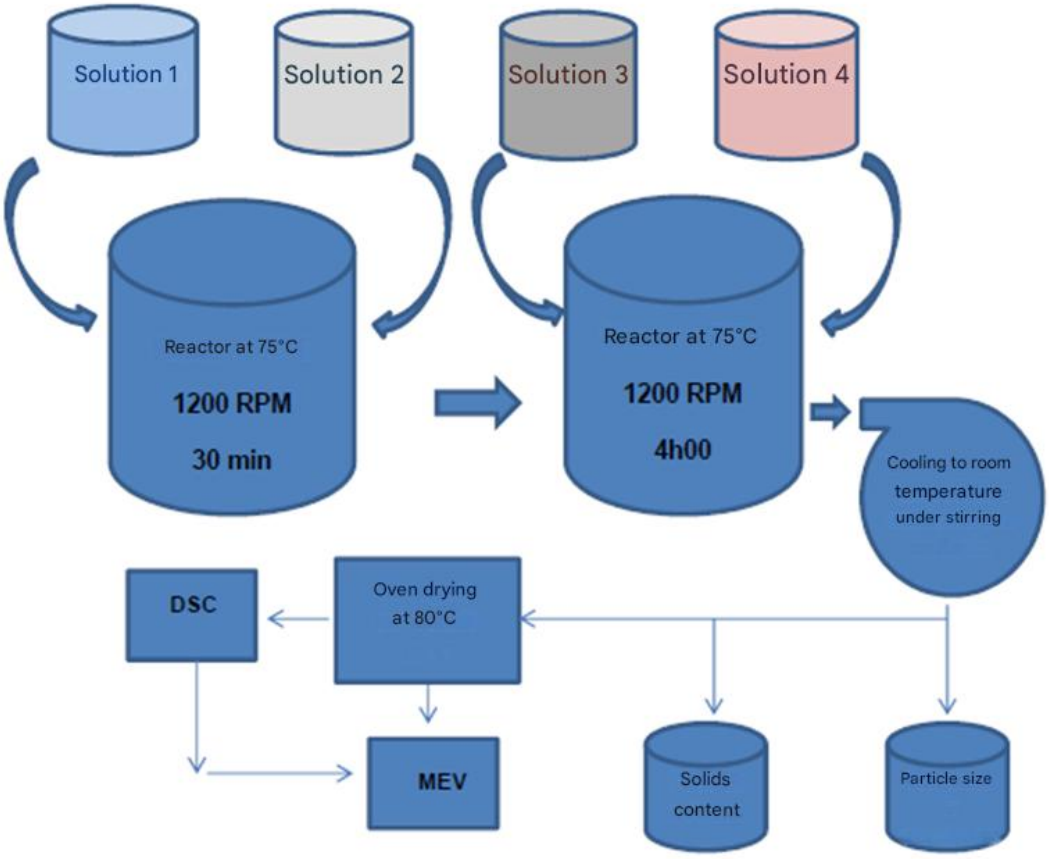
The encapsulation of phase change materials (PCMs) was performed by interfacial polymerization, using different formulations with the objective of optimizing colloidal stability, encapsulation efficiency and monomer conversion.

Production followed the following steps, also shown in Figure 1:

- **Wax melting and emulsion preparation:** Wax (CPC or CMU) was added to a 350 mL jacketed reactor containing water and colloidal silica. The system was kept at 75 °C, under agitation at 1200 rpm for 30 minutes, until complete wax melting.
- **Addition of reagents:** The monomers (methyl methacrylate and HEMA) were then added and, in a fractionated form, the potassium persulfate initiator, dissolved in water. The reaction continued for 4 hours at 75 °C, under the same agitation.
- **Cooling and drying:** After the reaction, the mixture was cooled to room temperature, with constant stirring, and then taken to the oven at 80 °C for drying until powder or pasty material was formed.
- **Analyses for evaluation of control parameters:** After cooling and before drying, the materials were analyzed for solids content using a METTLER TOLEDO infrared moisture analyzer scale, model HB43-S and particle size using the Microtrac S3500 analyzer, Horiba Instruments Inc. After drying, high-resolution scanning electron microscope (SEM-FEG) analyses were performed using FEI QUANTA 3D FEG equipment, available at the Laboratory of Chemical Processes and Particle Technology of IPT and again DSC.

A jacketed reactor with thermal bath, condenser with cooling bath, sealing to avoid volatile losses, and agitation system with disc impeller (CAWLESS) were used.

Figure 1 - Flowchart of the production stages of the PCMs.



Source: the author.

Throughout the production, four solutions were used, as shown in Figure 1, added to the reactor at different times, namely: Solution 1 (Water + colloidal silica), Solution 2 (CPC or CMU wax), Solution 3 (Methyl methacrylate + HEMA functional monomer) and Solution 4 (Potassium persulfate + water).

After synthesis, the microcapsules containing the phase change materials were dried at 80 °C in an oven until powder or pasty mass was obtained. The average particle size was determined by laser diffraction, with dispersion of the samples in water under ultrasound. The solids content was obtained by gravimetric analysis, with drying of the samples in an oven at 100 °C for 24 hours. The thermal analysis was performed by DSC, under the same conditions applied to pure waxes, to verify the maintenance of the melting enthalpy and thermal stability after encapsulation. Finally, the morphology of the microcapsules was evaluated by scanning electron microscopy (SEM), using the JSM-6510 JSM-6510 equipment, allowing the observation of the integrity of the polymeric wall and distribution of the encapsulated particles.

SOLIDS CONTENT OF ENCAPSULATED PCMS AND POLYMERIZATION EFFICIENCY

The analysis of the solids content of the suspensions containing encapsulated PCMs was used to estimate the polymerization efficiency, by comparing the theoretical and experimental values obtained. The difference between these values indicates the fraction of monomer that was lost by volatilization or that did not participate in the reaction. The efficiency was calculated based on Equation 1, and the results are presented in Table 2, including the theoretical and experimental values and the percentage of encapsulation achieved in the different formulations.

$$EP = \frac{(\mathrm{TSe}-\mathrm{TSsm})}{\mathrm{TSm}}\, x100 \qquad \text{Equation 1}$$

Where - EP: Polymerization efficiency; TSe: Experimental solids content; TSsm: Monomer-free solids content; TSm: Solids content of the monomer.

APPLICATION OF COTTON FABRIC ENCAPSULATED PCMS

The applications of the encapsulated PCMs were carried out on a flat fabric with a 100% cotton composition, an average weight of 121 g/m² and an average thickness of 0.31 mm, with a screen-like pattern. Specimens of (10 x 10) cm were used and for fixation of the encapsulated PCMs in the tissues, the Acrylux HTX 52 resin (Chemical Lumen) was used. Three different types of specimens were prepared: A tissue specimen without resin or PCM application, a tissue specimen with only 10% (m/m) resin application, and a tissue specimen with 10% (m/m) resin and 30% (m/m) PCM. The prepared solutions were deposited on the specimens to obtain a 100% *pick-up*, which were then dried in an oven at 100 °C for 30 min.

THERMAL ANALYSIS OF TISSUE CONTAINING ENCAPSULATED PCMS

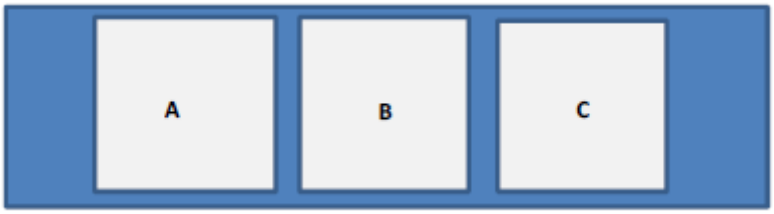
The fabric impregnated with encapsulated PCM was analyzed for its thermal behavior in the absorption and release of heat, in addition to the heat transferred in these materials, for this purpose two different analyses were used: infrared thermography and TCI – *Thermal Conductivity Analyser.*

Infrared thermography analysis was used to evaluate the thermal behavior of tissues containing encapsulated PCMs during the heating and cooling processes. The equipment used was a FLIR® T600 thermal imaging camera, which allowed the detection of infrared radiation emitted by the specimens and its conversion into thermal images.

To perform the assay, the tissues were positioned on a metal plate, which was heated on a plate at approximately 100 °C for 30 seconds. After this time, the plate was removed and cooled to room temperature (~20 °C). Thermal images were captured at regular intervals: at 10, 20 and 30 seconds during warm-up, and at 1 min, 2 min and 3 min during cool-down.

Three sample conditions were compared: fabric without any application (control), fabric with application of only 10% m/m of resin, to evaluate its influence on the material, and fabric with application of 10% m/m of resin and 30% m/m of encapsulated PCM. The thermal behavior was evaluated by means of the point of highest temperature in each image and the graphical analysis of the thermal profiles was performed using the FLIR ThermaCAM Quickview® software. Figure 2 shows the arrangement of the specimens on the plate, where position A is the tissue considered "control" (without any application), position B is the tissue containing the application of 10% m/m of the resin only and position C the tissue containing the application of 10% m/m of the resin and 30% m/m of PCM.

Figure 2 - Tissue positioning in infrared thermography analysis



Source: the author.

The evaluation of the thermal conductivity and effusivity of the treated tissues was carried out to investigate the behavior of heat flow through the materials, especially after the impregnation of encapsulated PCMs.

Thermal conductivity measures the ability of the fabric to transfer heat by steady-state conduction, and is essential for understanding the thermal response speed of the material. Fabrics containing PCMs tend to have lower conductivity, which means less immediate heat propagation, a desirable characteristic because it favors the storage and controlled release of thermal energy.

Thermal effusivity, on the other hand, represents the ability of the fabric to exchange heat with the environment or with human skin. Materials with lower effusivity are perceived as "warmer" to the touch, while those with higher effusivity feel "colder". This property is relevant to perceived thermal comfort, and its measurement allows to verify whether the fabric treated with PCM effectively modifies the thermal sensation in practice.

The tests were carried out with the TCi™ equipment from C-Therm Technologies, which operates through the transient method of the origin plane. The samples were placed on the sensor, and seven successive measurements were performed for each material, the first value being discarded and the following six were considered, according to the methodology recommended by the equipment manufacturer.

The data obtained allowed us to compare the untreated tissues, with resin application only and with resin application plus 30% (m/m) of encapsulated PCM.

RESULTS AND DISCUSSION

SOLIDS CONTENT OF ENCAPSULATED PCMS AND POLYMERIZATION EFFICIENCY

Table 2 shows the theoretical and experimental values and the percentage of encapsulation achieved in the formulations.

Table 2 - Characterization of the encapsulated PCMs regarding the solids content formed and the polymerization efficiency

Wax Composition	Solids content Theoretical (%)	Content of Solid experimental (%)	Content of solids monomer (%)	Content of solids without monomer (%)	Efficiency of polymerization (%)
Cetil Palmitate	10,9	8,96	5,7	5,3	64,21
Ucuúba butter	10,9	7,63	5,7	5,2	42,63

Source: the author.

The formulations showed polymerization efficiencies (PE) of 64.21% and 42.63%, respectively. These results are in accordance with data from the literature in which Sari et al. (2009) reported a PE of 43% in the production of capsules containing n-octacosanol, encapsulated in poly(methyl methacrylate), this value is close to that obtained in the formulation of ucuúba butter. Fang et al. (2009) found 53.5% for micrometric capsules, while Sánchez et al. (2007) reported 49% for capsules of about 10 μm. Thus, the values obtained in this study are within the range considered technically acceptable, especially when considering that the capsules produced are on a nanometric scale.

The analysis of PE provided an indication as to the efficacy of the encapsulation process. Values between 40% and 65% are consistent with systems involving polymeric emulsion and volatile monomers, as is the case of the interfacial polymerization employed. Therefore, the results obtained confirm that the methodology adopted was adequate to obtain capsules with a yield comparable to that found by other authors.

OVEN DRYING

The visual evaluation of the samples after drying in an oven was an important indication of the efficiency of the encapsulation of the PCMs. In the formulations of cetyl palmitate and ucuúba butter, the formation of dry powder was observed, observed by the example of the formulation using cetyl palmitate presented in Figure 3a, indicating that the wax was correctly encapsulated by the polymer matrix. The absence of visible residues of molten wax on the surface of the samples suggests that the active material was contained inside the microcapsules, and that, even after exposure to a temperature higher than the melting point of the PCMs, there was no extravasation of the contents.

This behavior contrasts with the formulation used as an example, and presented in Figure 3 b, of a sample with ucuúba butter, which during the stages of defining the study parameters, there was the formation of clots, which can be associated with the presence of non-encapsulated wax, which melts during drying, indicating incomplete or inefficient encapsulation.

Thus, the result of the drying in an oven not only validated the efficiency of the process, but also served as a criterion to select the most appropriate formulations for subsequent characterizations. The success of the formulations in this trial also corroborates the adequacy of the monomer/wax ratio used and the use of the functional monomer HEMA, which has been shown to favor the formation of stable and complete capsules. In Figure 3 it is possible to observe the behavior of the formulations after the drying process.

Figure 3 - Appearance of encapsulated PCMs after drying in an oven

(a) Encapsulated of cetyl palmitate PCM, after drying in an oven with powder formation

(b) Formulation with ucuúba butter in which there was no encapsulation (example), with the formation of clots

Source: the author.

AVERAGE PARTICLE SIZE

The formulations were analyzed for the average size of the particles by means of the laser diffraction technique, after showing good performance in the oven drying test. The results indicated that the formulation containing ucuúba butter generated the smallest particles, with an average diameter of 7.27 μm**,** while the formulation containing cetyl palmitate presented particles with an average diameter of 22.13 μm, as shown in Table 3.

Table 3 - Average particle size of the encapsulated materials

Formulation	D10 < 10% (µm)	D10 < 50% (µm)	D10 < 90% (µm)	Average Diameter (µm)
Cetil Palmitate	2,32	23,83	38,66	22,13
Ucuúba butter	0,85	3,62	18,45	7,27

Source: the author.

The observed difference was attributed to the viscosity of the wax melted at the time of emulsification. Ucuúba butter has the lowest melting point (~45 °C) and lower viscosity, facilitating the formation of smaller droplets during emulsion and resulting in smaller encapsulated particles. Cetyl palmitate, on the other hand, with a higher melting point (~54 °C), forms a more viscous emulsion, favoring the formation of larger particles.

This relationship between melting temperature, viscosity and particle size is in accordance with what is described by Nosari (2012), who directly relates lower viscosity to the formation of finer particles during encapsulation processes. It should be noted, however, that the values obtained by laser diffraction can represent clusters, and not isolated particles, and it is necessary to complement them with SEM data for greater precision.

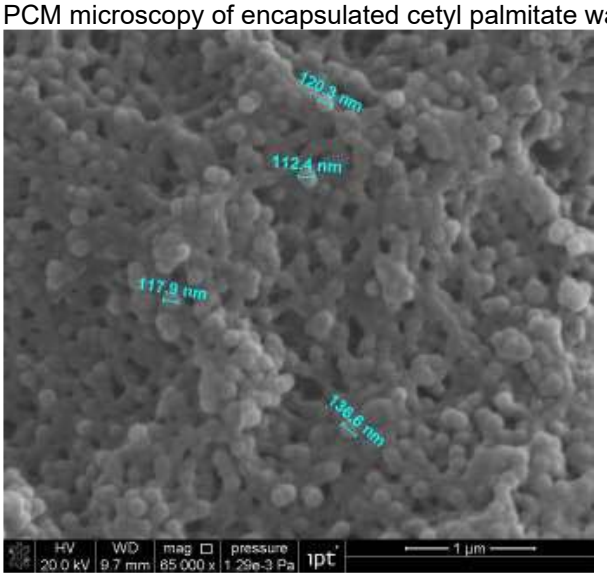
From the point of view of textile application, smaller particles are desirable because they cause less interference in the touch, fit and appearance of the fabric.

ANALYSIS OF SEM-ENCAPSULATED PCMS

For the analysis of the samples in the SEM, the material was used after drying in an oven, which was washed in solvent, in which about 0.5 g of the dried samples were added in a test tube together with enough hexane to cover the entire sample. This mixture was then stirred and this procedure was repeated three times. Subsequently, this material went through heating and cooling cycles in the DSC, in order to evaluate its integrity after thermal stress.

As shown in Figure 4 of the PCM of cetyl palmitate wax, obtained by SEM, the encapsulate presented well-defined particles, with regular morphology and dimensions on the nanometric scale, reaching about 120 nm. The results indicate that the encapsulation technique was effective in the formation of nanocapsules, with uniform distribution and structural integrity. In addition, it was observed that the capsules kept their physical structure intact even after undergoing three consecutive thermal cycles in the DSC analysis, evidencing good thermal stability of the system.

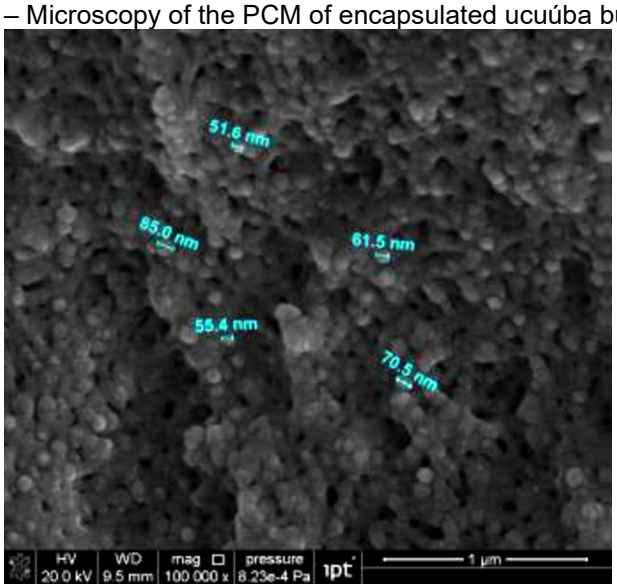
Figure 4 - PCM microscopy of encapsulated cetyl palmitate wax



Microscopic SEM image of the PCM of cetyl palmitate wax produced using a functional HEMA monomer, obtained at 65 000 x magnification.
Source: the author.

As shown in Figure 5, the PCM encapsulated with ucuúba butter and HEMA functional monomer also resulted in the formation of well-defined particles, with sizes ranging from 50 to 85 nm, characterized at the nanometric scale. Microscopy indicates that encapsulation was effective, with the formation of regular and homogeneously distributed capsules. As in the formulation with cetyl palmitate, it was found that the material kept its physical structure intact after three thermal cycles of heating and cooling performed in the DSC analysis, evidencing the morphological stability of the system.

Figure 5 – Microscopy of the PCM of encapsulated ucuúba butter wax



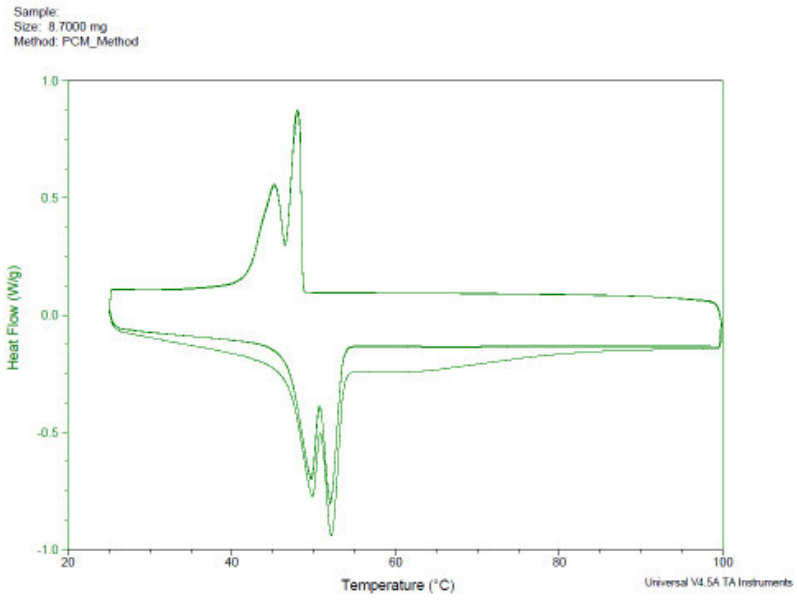
Microscopic SEM image of the PCM of ucuúba butter wax produced using a functional HEMA monomer, with a magnification of 100,000 times.
Source: the author.

CHARACTERIZATION OF DSC-ENCAPSULATED PCMS AND ENCAPSULATION EFFICIENCY

The encapsulated materials were submitted to three consecutive thermal cycles of DSC analysis, with a temperature range of 25 °C to 100 °C and a rate of 5 °C/min, in order to evaluate their thermal stability and structure after the process. The curves obtained confirmed that the morphology of the capsules was preserved even after the cycles. DSC analysis also allowed us to determine the enthalpy of fusion of encapsulated PCMs, which is essential for calculating encapsulation efficiency, as proposed by Sari et al. (2010). The amount of PCM retained inside the capsules directly impacts the thermal storage capacity of the system. Figure 6 illustrates, by means of overlapping curves, the three thermal cycles of the encapsulated PCM of cetyl palmitate, revealing the consistency of the thermal behavior throughout the analyses.

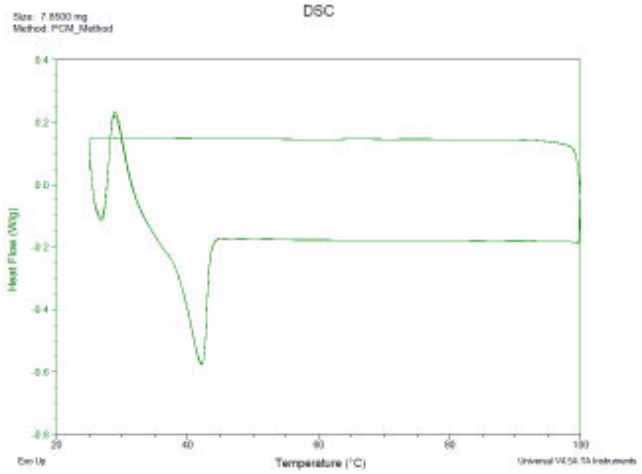
The DSC curve in Figure 6 shows consistent melting and recrystallization peaks over the three thermal cycles, where the cetyl palmitate encapsulates showed temperatures around 54 °C and the ucuúba butter encapsulates around 45 °C, indicating stability of the encapsulated PCM **and** absence of changes in transition temperatures**.** According to Sari et al. (2010), this behavior suggests that the capsule structure remained intact, with no signs of chemical degradation. The same pattern was observed in the formulation containing encapsulated ucuúba butter, as shown in Figure 7.

Figure 6 - DSC curve of encapsulated PCM from the formulation of cetyl palmitate wax



Source: the author.

Figure 7 - DSC curve of the encapsulated PCM from the formulation of ucuúba butter wax



Source: the author.

The encapsulation efficiency (EE) of the encapsulated phase change materials (PCMs) was determined based on the ratio of the enthalpy of fusion of the encapsulated PCM to the enthalpy of the pure wax used, as described by Sari et al. (2010). This metric makes it possible to evaluate the functional yield of the encapsulation, that is, the amount of active material effectively retained in the capsules capable of storing thermal energy.

The enthalpy of fusion of pure wax was 174.97 J/g for cetyl palmitate and 80.38 J/g for ucuúba butter. After encapsulation, the values observed were 78.75 J/g for the formulation using cetyl palmitate and 36.20 J/g for the formulation using ucuúba butter. Based on these data, the encapsulation efficiency (EE) was calculated using Equation 2, suggested by Sari et al (2010).

$$EE = \frac{\Delta H \text{ encapsulated}}{\Delta H \text{ pure wax}} x100 \qquad \text{Equation 2}$$

Applying the equation:

Formulation with cetyl palmitate: EE = (78.75 / 174.97) × 100 ≈ 45.03%

Formulation with ucuúba butter: EE = (36.20 / 80.38) × 100 ≈ 45.06%

Both systems presented an encapsulation efficiency of approximately 45%, a value considered satisfactory for polymeric systems obtained by interfacial polymerization at the nanometric scale. These results are in line with previous studies. For example, Fang et al. (2009) and Sánchez et al. (2007) reported values of 53.5% and 49%, respectively, for capsules at micrometric and submicron scales.

The obtaining of values close to those found in the literature, added to the morphological stability observed by SEM and the thermal performance analyzed by DSC,

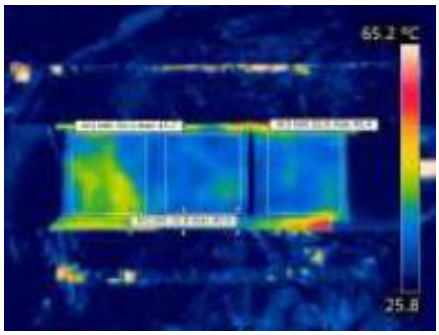
confirms the efficacy of the encapsulation method adopted in this study and its feasibility for application in textile systems with thermal storage properties.

INFRARED THERMOGRAPHY ANALYSIS OF TISSUES CONTAINING ENCAPSULATED PCM

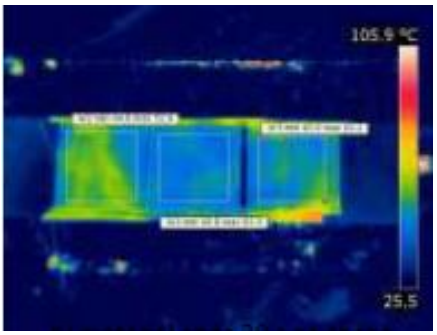
Figure 8 shows the thermal behavior of the specimens of the infrared thermography analysis performed in the comparison of the specimen containing PCM of ucuúba butter, in comparison with a tissue containing only resin and another tissue without application (control).

The graph presented in Figure 9 shows the thermal behaviors of the specimens in the infrared thermography analysis performed in the comparison of the specimen of a flat fabric containing PCM ucuúba butter, compared to a flat tissue containing only resin and another flat fabric without application (control).

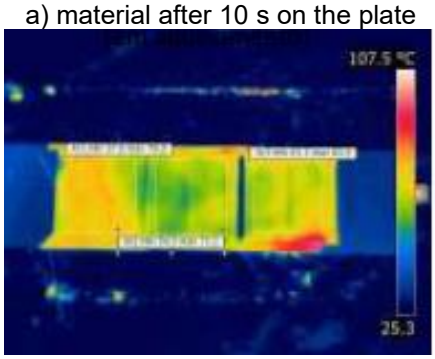
Figure 8 - Thermal behavior of fabrics containing different formulations in heating plate over time, in the analysis of the PCM ucuúba butter.



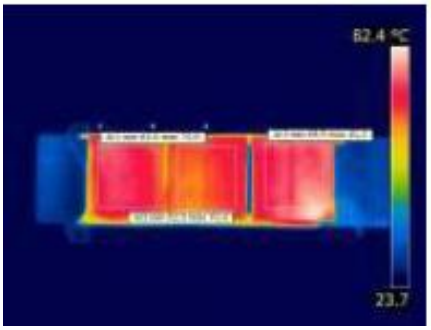
a) material after 10 s on the plate



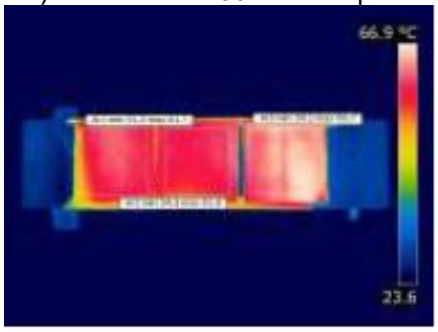
b) material after 20 s on the plate



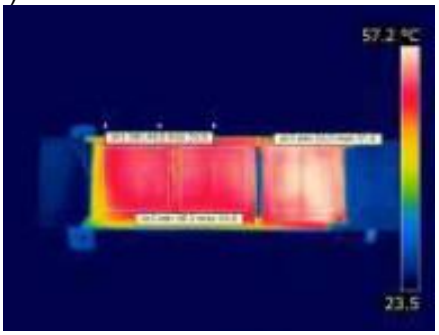
c) material after 30 s on the plate



d) Material after 1 min out of the sheet



e) Material after 2 min out of the sheet



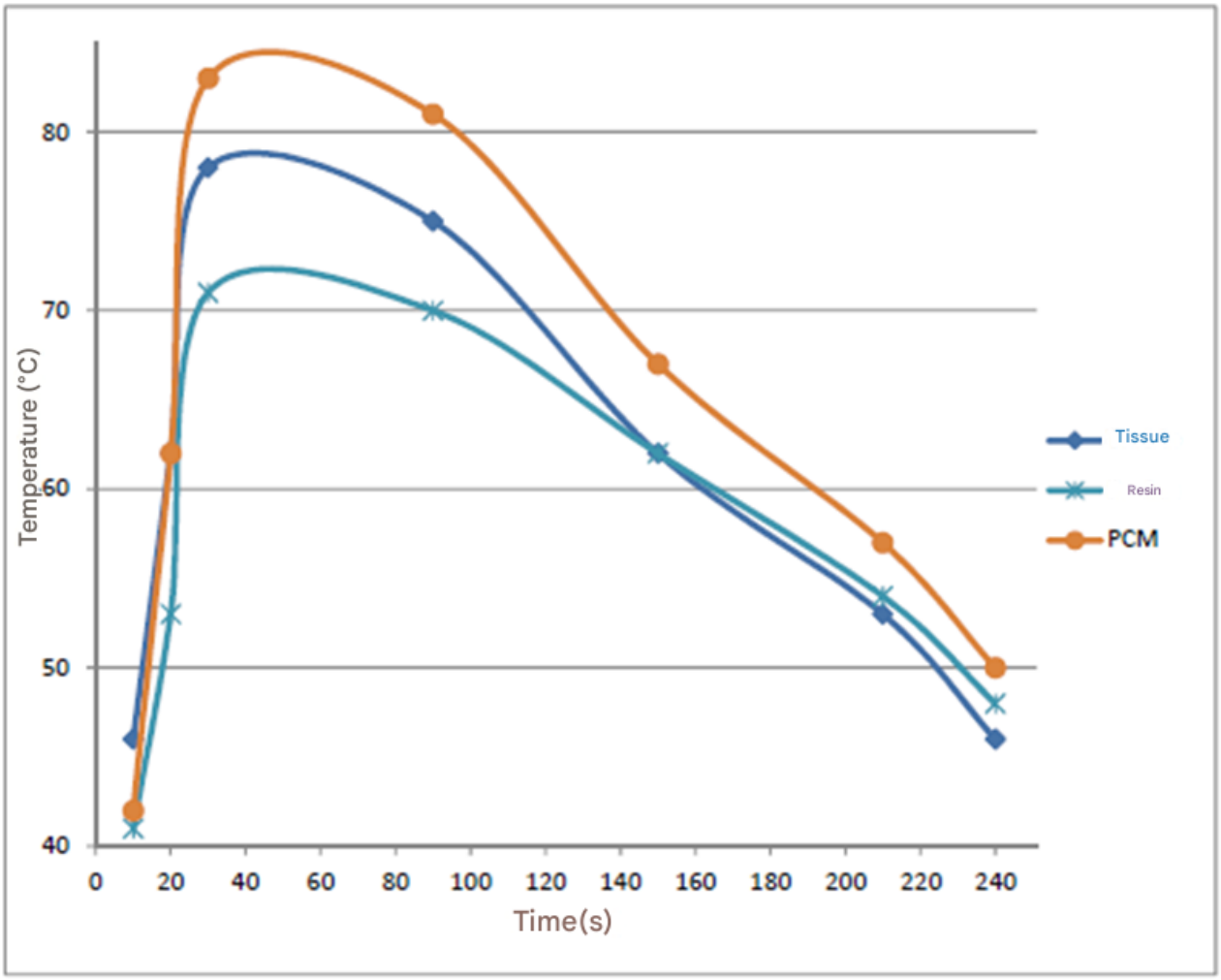
f) Material after 3 min out of the plate

Comparative thermal behavior of a flat fabric without any application (left), a flat fabric containing only the resin (center) and a fabric containing PCM of ucuúba butter (right).

Source: the author.

The evaluation of the surface thermal behavior of the tissues, by means of thermographic images (Figures 8 and 9), revealed differences between the treated and untreated samples. During the initial warm-up (first 20 seconds), the non-applied (control) fabric showed greater heat absorption, reaching higher temperatures than the others. On the other hand, the fabrics treated with resin and resin + encapsulated PCM showed lower thermal elevation, and the fabric with resin application only showed more insulating behavior.

Figure 9 - Comparative temperature curves of flat fabric containing PCM of ucuúba butter over time



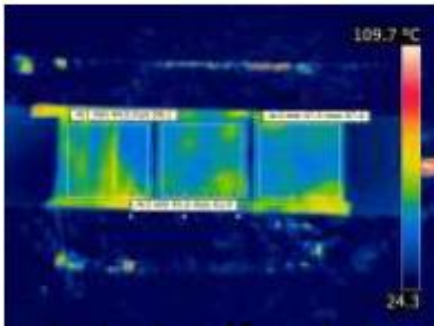
Source: the author.

In the specific case of the formulation with PCM encapsulated with ucuúba butter (Figure 9), a slower heat absorption was observed during heating, a behavior attributed to the energy required for the fusion of the wax present in the microcapsules. This phase transition process acts as a thermal attenuator, reducing the heating speed of the fabric.

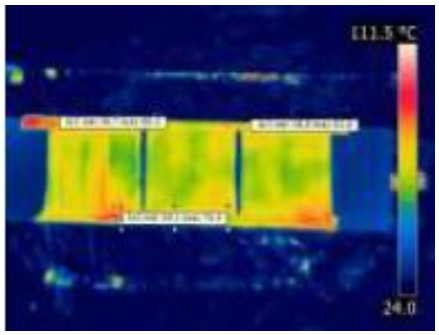
During cooling, the tissue containing PCM stood out for maintaining the higher surface temperature over time**.** This behavior indicates that the heat absorbed in the heating phase was stored in the form of latent heat and released gradually. In contrast, the tissues without PCM showed a faster drop in temperature. These results indicate the efficiency of encapsulated PCM in the thermal regulation of the tissue, contributing to a more controlled heat exchange with the environment.

Similarly, Figures 10 and 11 present the images obtained by infrared thermography and the data obtained from these images in the comparison of the specimen of a flat tissue containing PCM of cetyl palmitate, compared to a flat tissue containing only resin and another flat tissue without application (white).

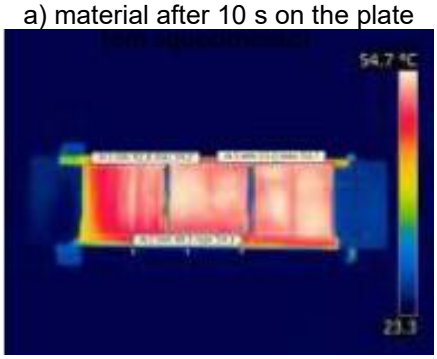
Figure 10 - Thermal behavior of tissues containing different formulations in heating plate over time, in the analysis of cetyl palmitate PCM.



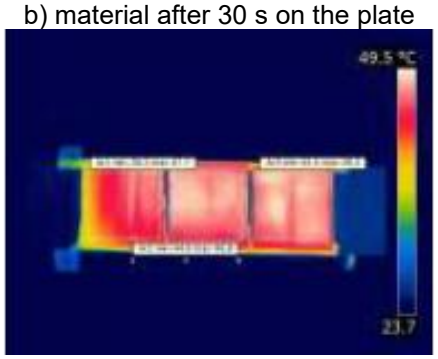
a) material after 10 s on the plate



b) material after 30 s on the plate



c) Material after 1 min out of the sheet



d) Material after 2 min out of the sheet

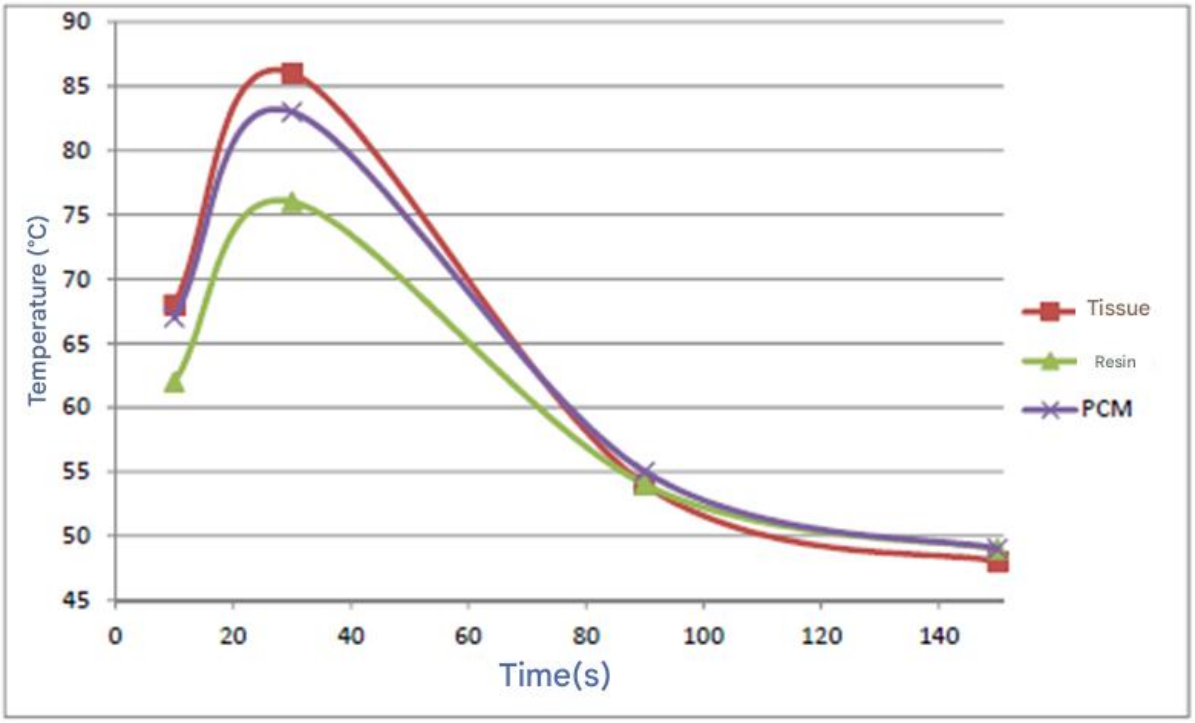
Comparative thermal behavior of a flat fabric with no application (left), a flat fabric containing only the resin (center), and a tissue containing cetyl palmitate PCM (right).
Source: the author.

The results observed for the formulation with cetyl palmitate were similar to those obtained for the formulation with ucuúba butter, where the control tissue absorbed more heat in the first moments of heating, presenting a greater rise in surface temperature, while the tissues with PCM and with resin showed less initial heating.

The presence of encapsulated PCM promoted, after a certain time, heat accumulation inside the microcapsules, which was later released more slowly during cooling. This behavior confirms the ability of PCM to store latent heat during fusion and release it gradually, providing a thermal regulation effect on the tissue.

The data show that the fabric with isolated resin maintains consistently lower temperatures than the other samples, suggesting that the resin acts as a thermal barrier, without contributing significantly to heat storage. Therefore, in PCM samples, the observed elevation and thermal retention can be attributed exclusively to the presence of the phase change material, validating its functionality.

Figure 11 - Comparative temperature curves of flat tissue containing PCM of cetyl palmitate over time



Source: the author.

ANALYSIS OF CONDUCTIVITY AND EFFUSIVITY OF TEXTILE ARTICLES CONTAINING ENCAPSULATED PCMS

The fabrics containing encapsulated PCMs were analyzed for conductivity and effusivity in comparison with the fabrics without application (control) and the fabrics containing only resin. Table 4 shows the effusivity and conductivity results of the materials analyzed.

It can be observed in Table 4 that fabrics containing PCMs have slightly lower **thermal conductivity** compared to fabrics without application (control). This indicates that, when subjected to a heat source, the fabric with PCM is more resistant to thermal conduction, delaying the passage of heat compared to the control fabric. This behavior is desirable in practical applications, as it allows the PCM to act as a temporary thermal barrier, absorbing heat during heating and gradually releasing it upon cooling.

When comparing these results with those obtained by infrared thermography, it is observed that, even with lower conductivity, the tissues with PCM were able to store a

greater amount of heat. This suggests that the absorbed heat is retained inside the microcapsules and is not immediately dissipated, as occurs in tissues without PCM, where heat propagates more quickly and unchecked.

Table 4 - Evaluation of effusivity and thermal conductivity of tissues containing PCMs

Tissues	Effusiveness (W√s/m2K)	Conductivity (W/mK)
No application (control)	180	0,08
With resin	183	0,08
With Resin + PCM Cetyl Palmitate	150	0,07
With resin + PCM ucuúba butter	140	0,07

Source: the author.

Regarding the effusivity data presented in Table 4, it is known that thermal effusivity is directly related to the sensation of "cold" or "hot" to the touch. Materials with greater effusiveness tend to quickly absorb heat from the skin, providing a feeling of freshness. On the other hand, materials with lower effusivity, such as fabrics with encapsulated PCM, absorb heat more slowly, being perceived as "hotter".

This reduction in effusivity in tissues with PCM indicates that the presence of capsules modifies the dynamics of thermal exchange with the environment. PCM, when undergoing its phase change, absorbs heat internally (as latent heat), without immediately transferring it to the external medium, which slows down surface heat exchange.

CONCLUSION

The present work demonstrated the feasibility of applying encapsulated phase change materials (PCMs) in textile substrates with the objective of promoting thermal control. The selected waxes, cetyl palmitate and ucuúba butter, showed thermal behavior compatible with textile applications, being successfully encapsulated by means of interfacial polymerization.

The formulations obtained good results in terms of polymerization efficiency, formation of stable nanocapsules, and encapsulation efficiency, around 45%, values compatible with the literature. Morphological (SEM) and thermal (DSC) analyses confirmed the stability of the capsules and the retention of the desired thermal behavior.

When applied to the fabrics, the encapsulated PCMs promoted relevant modifications in the thermal properties of the materials. The treated tissues showed lower conductivity and thermal effusivity, as well as differentiated thermal behavior during heating and cooling, as demonstrated by thermography analyses. This set of results highlights the

potential of encapsulated PCMs for use in functional textile materials, with applications in thermal comfort and technical clothing.

ACKNOWLEDGMENT

To the Institute of Technological Research – IPT, for the support in all the analyses for conducting the research.

To the Textile Technology and Protection Products Laboratory (LTP) of IPT for providing the work infrastructure.

To the Laboratory of Chemical Processes and Particle Technology of the IPT, for providing the work infrastructure.

To the School of Arts, Science and Humanities EACH of the University of São Paulo, for the support in the Research.

To the companies Lumen Química and Dhaymers, for the supply of the reagents used.

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