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Thermo-Regulated Cotton: Enhanced Insulation through PVA Nanofiber-Coated PCM Microcapsules

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Abstract: The innovative integration of phase change materials (PCMs) into textiles through microencapsulation presents a transformative approach to developing thermally regulated fabrics. This study explores the synthesis and characterization of microcapsules containing a coconut oil core and an ethylcellulose shell, and their application on cotton fabrics coated with polyvinyl alcohol (PVA) nanofibers. The dual-layer system involving microcapsules and nanofibers is designed to enhance the thermal insulation properties of textiles by regulating heat through the absorption and release of thermal energy. The microencapsulation of PCMs allows for the effective incorporation of these materials into textiles without altering the fabric's inherent properties. In this study, the coconut oil serves as the PCM, known for its suitable phase change temperature range, while ethylcellulose provides a robust shell, enhancing the microcapsules' structural integrity. The application of a PVA nanofibers layer not only strengthens the thermal regulation properties but also protects the microcapsules from release while the fabric is manipulated, thereby prolonging the functional life of the fabric. Comprehensive testing, including scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR), confirms the successful application and durability of the microcapsules on the textiles. Thermal imaging studies demonstrate the fabric's enhanced capability to maintain a consistent temperature, highlighting the potential of this technology in applications ranging from smart clothing to energy-efficient building materials or automotive isolation. The integration of PCMs in textiles via microencapsulation and nanofiber technology marks a significant advancement in textile engineering, offering new opportunities for the development of smart and sustainable materials. The study demonstrates the promising potential of integrating PCMs into textiles using microencapsulation and nanofiber technologies. Despite the initially modest insulation improvements, the methodology provides a robust foundation for further research and development.

Keywords: coconut oil; ethylcellulose; sustainability; textile; shell; core; FTIR; SEM

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1. Introduction

PCMs leverage latent heat that can be stored or released within a narrow temperature range, changing their state with temperature fluctuations. The integration of phase change materials (PCMs) into textiles represents a significant advancement in the development of smart and functional fabrics. PCMs are substances with a high latent heat of fusion, which means they can absorb or release a large amount of energy at a specific temperature. This unique property enables textiles containing PCMs to maintain a stable temperature, reducing the thermal gap experienced by the wearer in varying environmental conditions. When integrated into textiles, these materials provide thermal regulation through energy absorption during heating and energy release during cooling. This capability is particularly valuable in clothing for cold environments, offering improved comfort and energy efficiency [1]. The application of microencapsulation technology in embedding PCMs into textiles has opened new avenues for improving thermal comfort and energy efficiency.

Phase change material (PCM) microencapsulation involves enclosing the PCM within a protective shell to form microcapsules. In the context of textiles, these microcapsules can be applied to fabrics for various functional benefits such as preventing the oil from leakage when melting.

Microcapsules, consisting of a core material where the PCM is contained and a protective shell, offer a method to incorporate PCMs into fabrics without altering their inherent properties [2]. Various organic and inorganic PCMs exist with a range of melting and solidifying temperatures. Techniques like microencapsulation have been developed to integrate PCMs into textiles, enhancing properties like thermal storage and stability. These advancements are crucial in fields like solar engineering, building materials, and technical textiles [3]. Despite the promising applications, challenges such as material selection, optimal integration techniques, and long-term stability remain. Ongoing research focuses on enhancing the efficiency and applicability of PCMs in textiles, aiming to overcome these hurdles for broader usage [4,5]. Natural compounds such as paraffins and biodegradable polymers are increasingly favored for this purpose due to growing environmental concerns associated with synthetic chemicals [6]. The ecological impact of producing and disposing of PCM-incorporated textiles is not fully understood. This includes concerns about the sustainability of the raw materials and the potential environmental hazards of the microcapsules themselves. In this context, the use of ethylcellulose for the microcapsule shell, coco oil as the core, and PVA nanofibers as a biodegradable polymer covering, presents an eco-friendly and sustainable approach. Ethylcellulose is a versatile polymer known for its excellent film-forming properties and biodegradability, which makes it an environmentally friendly material [7]. Coconut oil is a natural PCM with a suitable phase change temperature range for textiles [8,9], and PVA (polyvinyl alcohol) nanofibers offer a unique combination of strength, flexibility, and biodegradability.

One of the challenges is the durability of PCM microcapsules within textiles. Nelson [10] focuses on the growth of commercial applications of microencapsulation in textiles, emphasizing the need for robust encapsulation processes to ensure the effective impartation of finishes and properties on textiles, which could be compromised during fabric manipulation. Repeated washing, exposure to UV light, and mechanical stress can affect the performance and lifespan of these microcapsules [11]. While significant progress has been made in the development and application of PCM microcapsules in textiles, further research is needed to address the gaps in long-term performance, environmental impact, and the standardization of testing methods.

Mineral wool is widely used for building insulation due to its thermal and acoustic properties. However, the space requirements for mineral wool insulation can be a limitation, especially in designs where compactness and space utilization are crucial. Mineral wool insulation typically requires a substantial thickness to achieve desired thermal resistance, which can be problematic in buildings where space is at a premium. This limitation necessitates a closer look at alternative solutions that are not only thinner but also more sustainable.

Alternative insulation materials, such as sheep wool, have been gaining attention in recent years. Sheep wool insulation, for instance, offers comparable thermal insulation characteristics to mineral wool and, in some cases, even outperforms it in specific applications. Importantly, sheep wool is more ecological, featuring fewer health risks and a lower environmental impact than traditional mineral wool. Studies have shown that sheep wool insulation has comparable building physics and acoustic properties to mineral wool, making it a suitable alternative in terms of performance while being more environmentally friendly and sustainable [12]. Additionally, the development of innovative mineral fiber insulating panels with lower thicknesses and enhanced sustainability, acoustic insulation, and fire resistance characteristics have been investigated, showing promise for building refurbishment, especially in historical buildings [13]. These advancements suggest a shift towards more compact and sustainable insulation solutions, offering viable alternatives to traditional mineral wool. Cotton, as an alternative insulation material for buildings, has been explored in various studies. A novel environmentally friendly thermal insulation material has been developed using binderless cotton stalk fiberboard (BCSF). This material is made from cotton stalk fibers without any chemical additives, employing high-frequency hot-pressing. The results indicated that BCSF with densities ranging from 150–450 kg/m^3 has thermal conductivity values close to those of expanded perlite and vermiculite in the same density range, making it suitable for ceiling and wall applications to save energy [14].

One study investigated the functionalization of cotton fabric using hollow glass microspheres and titanium dioxide (TiO_2) nanoparticles, focusing on various properties including thermal insulation. The presence of these microspheres on the surface of cotton improved the thermal resistance by 78%, demonstrating superior thermal insulation. This suggests that similar nanomaterials, when applied to cotton fabrics, could enhance their thermal insulation properties [15]. Another study developed reactive multiwall carbon nanotube (MWCNT) nanofluid-coated cotton fabrics, which showed improved thermal conductivity while preserving electrical insulation. This research demonstrates the potential of nanomaterial coatings in enhancing the thermal properties of cotton fabrics [16]. Additionally, a study on electrospun nanofibers deposited on clothing textiles, including cotton, showed that nanofiber layers significantly influence the thermal and physiological properties of the fabric, which could be relevant for thermal insulation applications [17]. These studies highlight the potential of nanofibers and nanomaterials in enhancing the thermal insulation properties of cotton fabrics, although direct studies on nanofiber layers on cotton fabrics specifically for thermal insulation are limited.

This paper aims to explore the potential of these materials in developing advanced thermal regulation textiles. The study delves into the synthesis and characterization of microcapsules with a coconut oil core and ethylcellulose shell, their integration into cotton fabrics, and how a protective layer of PVA nanofibers influences the thermal behavior. The implications of these developments for the enhancement of thermal insulation in functionalized textiles will be analyzed.

2. Materials and Methods

2.1. Materials

Ethylcellulose form Ethocell (Merck Life Science S.L.U., Madrid, Spain) was used as the microcapsule shell. Coconut oil (La Masia, Spain) was used as the active ingredient.

Polyvinyl alcohol (PVA) Mw 61,000 g/mol, purchased from Sigma-Aldrich, St. Louis, MO, USA, was used to obtain the 9% (w/v) solution in distilled water for the PVA nanofibers and 1% (w/v) was used as a surfactant for the microencapsulation process.

Chemically bleached 100% cotton fabric with plain rapport and 210 g/m^2 was used to support the microcapsules.

2.2. Methods

2.2.1. Microcapsules Preparation

Microcapsules were prepared by a solvent evaporation method. The coco oil, which is in liquid form during the encapsulation process, becomes the core of the microcapsules. As the temperature drops, the coco oil solidifies, encapsulated within the ethylcellulose shell. The ethylcellulose, dissolved in acetone, forms the shell around the coco oil droplets. As the acetone evaporates, the ethylcellulose solidifies, creating a rigid structure that encapsulates the coco oil. The presence of the PVA surfactant ensures that the coco oil droplets are well-dispersed and stabilized during the shell formation process.

The shell was prepared by dissolving ethylcellulose 18% into acetone (w/v). The solution was mixed using a magnetic stirrer for two hours in a closed reactor to prevent acetone evaporation. Ethylcellulose solution was formed.

A 1% (w/v) PVA (polyvinyl alcohol) solution, to act as a surfactant, was also prepared. This helps stabilize the emulsion and improve the encapsulation efficiency.

Coco oil is solid at room temperature; in order to melt it, it was heated at 55 °C for 10 min. Afterwards, the melted coco oil 40% (v/v) was mixed with 1% PVA solution and stirred at 1500 rpm for 15 min, and the coco oil emulsion was prepared. The ethylcellulose was added dropwise, while the coco oil emulsion was magnetically stirred (900 rpm) and kept at 35 °C in an open reactor. The mixture was stirred to evaporate the volatile component (acetone) and to make the shell structure hard enough around the active ingredient for approximately 2 h. Then, the emulsion was characterized by Image J analysis software (version 1.53).

2.2.2. Microcapsules Application

Coco microcapsules were applied to cotton fibers by a padding procedure. The coco microcapsules were dispersed in water (60 g/L) and the fabric was immersed in a padding mangle; afterwards, the fabric was passed through two squeezing rolls with an 87 % pick up. Then, the fabric was allowed to dry flat.

2.2.3. Nanofibers

To obtain the PVA solution, a 9% (w/v) PVA solution was prepared by heating the water to 80 °C with magnetic stirring until the PVA was completely dissolved. The electrospinning process was carried out with a BIOINICIA electrospinning system. While electrospinning the nozzle-collector distance was 15 cm. The collector was placed vertically. The same flow rate (0.5 mL/h) and voltage values of 20 kV were used. Subsequently, after adjusting the voltage, electrospinning was carried out for periods of 30 min. Two samples were prepared, one to coat the cotton fabric (NF) and another to coat the cotton fabric treated with microcapsules as described in Section 2.2.2 (MC-NF).

2.2.4. Characterization

For the morphological characterization, FESEM (ULTRA 55, Carl Zeiss, Jena, Germany) scanning electron microscopy (SEM) was used, using an accelerating voltage of 2 kV on the surfaces to be analyzed on each of the samples and at the magnifications considered appropriate in each case. The sample was previously coated with gold/platinum to confer on the sample the conductivity required for correct observation.

Fourier transform infrared spectroscopy (FTIR) was performed for the characterization of the starting materials (oils and PVA) as well as for the characterization of the nanofibers obtained. A JASCO FT/IR-4700 type A spectrophotometer (Jasco Spain, Madrid, Spain) with the ATR accessory was used. Sixteen spectra with a resolution of 4 cm^{-1} were performed for each sample.

Fabrics were placed a 30 cm distance from a 100 °C heater and held by two wooden bars (red in Figure 1), bars which were placed on a frame (black in Figure 1) placed around the heater and below the camera. Every period of 15 s for 2 min, we measured the fabric temperatures by means of a thermochromic camera (model Testo 865, México D.F., Mexico), which was placed at 50 cm of the fabric. At the end of the second minute, we removed the heater and kept measuring cooling temperatures each 15 s for 2 min. Figure 1 shows the scheme of the system established for heating and measuring. In this case, the sample represented is the one which contains the nanofibers to show the nanofibers veil position. Three different specimens were measured for every sample.

The fabric was subjected to an energetic shaking process to evaluate the stability and adherence of microcapsules on its surface. The procedure involved holding the fabric firmly at one end and vigorously hand shaking the fabric back-and-forth in the air for a period of 1 min, with 60 shakes. This mechanical agitation simulates the forces the fabric might encounter during handling and use. After the shaking process, the fabric was examined for any signs of microcapsule detachment.

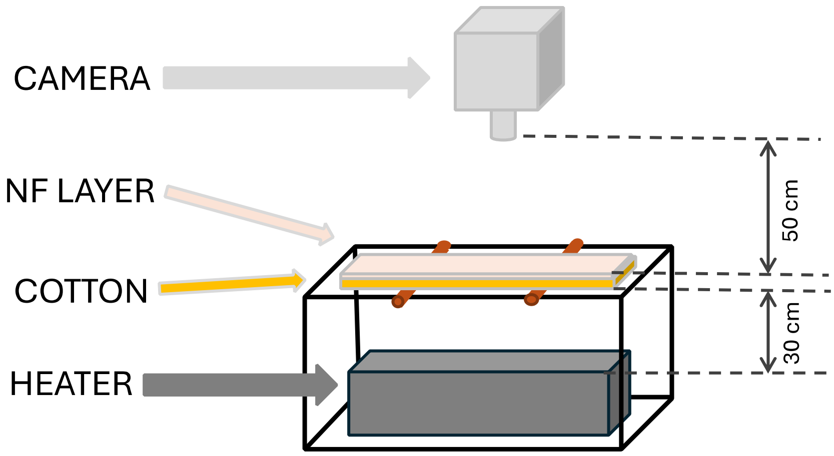

Figure 1. Scheme for the thermal measurement system.

3. Results

3.1. Microcapsules Characterization

Figure 2 presents an optical micrograph and a histogram detailing the size distribution of the microcapsules. The microcapsules appear as spherical entities with varying diameters, demonstrating the encapsulation process's effectiveness in producing uniformly shaped particles. The microcapsules have diameters ranging from 5 µm to 24.5 µm. By means of Image J software, the total amount of microcapsules from the image in Figure 2 is 131; the most frequent size range is between 5.0 µm and 6.0 µm. This indicates that the majority of the microcapsules fall within this size range. There is a noticeable decreasing trend in frequency as the microcapsule diameter increases. Smaller microcapsules (5.0 µm to 6.0 µm) are more abundant, while larger microcapsules (24.0 µm to 25.0 µm) are much less frequent.

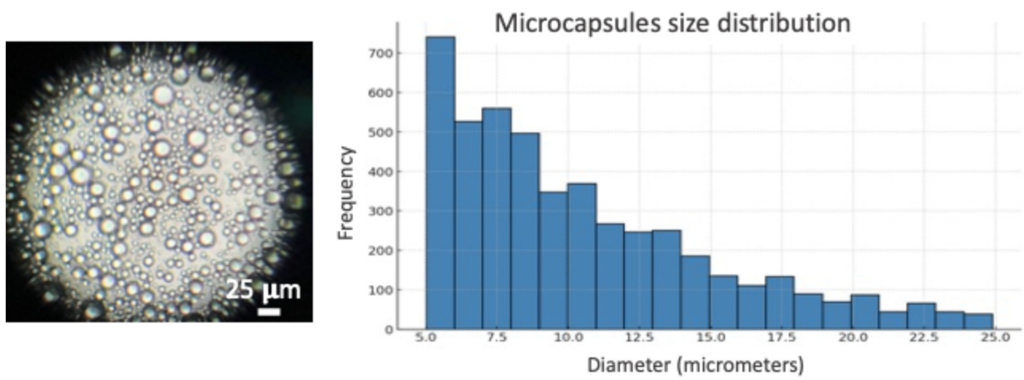

Figure 2. Microcapsule characterization. Histogram for microcapsules size distribution.

Microcapsules were placed on the cotton fabric and SEM and FTIR analysis were conducted before the PVA nanofibers were used to coat them. Figure 3 illustrates the cotton fibers with some round spheres on them. Those round particles are attributed to the coco microcapsules.

The FTIR spectra plot illustrates the absorbance spectrum for coco oil, ethylcellulose, cotton fabric, and cotton fabric with microcapsules (Figure 4). To identify the presence of microcapsules on the cotton fibers, we can look for new features in the microcapsules´spectrum (MC) which are distinct from cotton fabric (Co), but present in coco oil spectra (coco) and ethyl cellulose (EC).

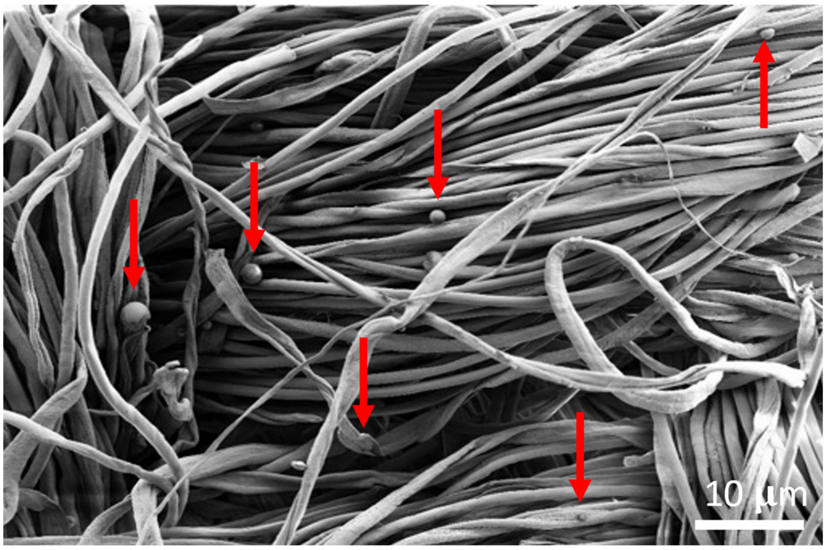

Figure 3. SEM image for coco oil microcapsules on cotton fabric once the fabric has been heated for five cycles. Arrows indicate the microcapsules position.

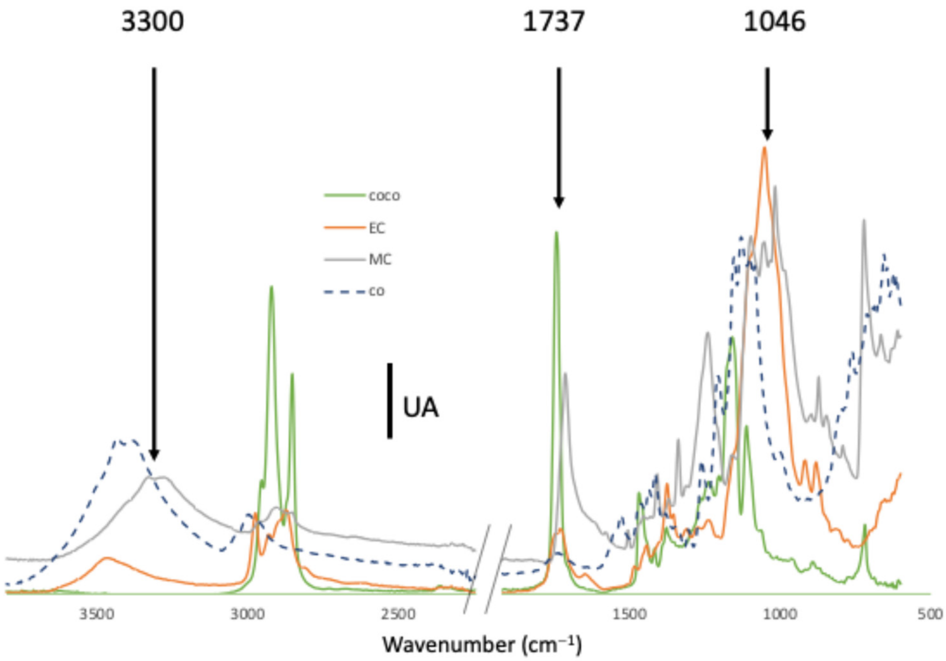

Figure 4. FTIR spectra for coco oil microcapsules on cotton fabric once the fabric has been heated for five cycles.

Ethylcellulose presence can be demonstrated by the peak centered around 1046 cm. This peak is characteristic of ethylcellulose [18,19] and clearly alters the spectrum for cotton fabric with microcapsules (MC). The prominence of this peak in EC and its presence in MC but not in coco oil or cotton indicates that the ethylcellulose component is a significant part of the MC material. It indicates that the microcapsules indeed contain ethylcellulose, likely forming the shell that encapsulates the core material (possibly coco oil in this case).

Pure cotton (Co) does not typically show a strong absorption near 1707 cm^{-1} [20], as cellulose does not contain carbonyl groups in its native form. The presence of a distinct peak at 1707 cm^{-1} in the FTIR spectrum of the cotton fabric with microcapsules (MC), which is not present in the pure cotton (Co) fabric but is observed in both coco oil and ethylcellulose (EC), can indeed be indicative of the presence of microcapsules on the fabric. This peak likely corresponds to specific chemical functionalities associated with the microcapsules. The peak at around 1737 cm^{-1} in the spectra of coco oil and ethylcellulose typically corresponds to C=O (carbonyl) stretching vibrations, which are common in ester or other carbonyl-containing groups [21,22]. This is a characteristic peak for many organic compounds, especially those that contain ester groups, as in ethylcellulose. The shift from 1737 cm^{-1} in coco and EC to 1707 cm^{-1} in MC suggests a change in the chemical environment of the carbonyl group when the microcapsules are formed and applied to the cotton fabric. The interaction between the coco oil and the ethylcellulose may alter the electronic environment around the carbonyl groups, leading to a shift in the absorption frequency. It might indicate the formation of new chemical bonds or interactions, such as hydrogen bonding, due to the encapsulation process, that affect the carbonyl group's vibration frequency [20–22].

When the OH stretching region between 3800 and 3000 cm^{-1} is analyzed, a shift towards a lower wavelength is observed. The encapsulation of coco oil with an ethylcellulose shell might create a microenvironment around the cotton fibers that differs significantly from that of pure cotton. This altered microenvironment could influence the hydrogen bonding dynamics of the OH groups in the cellulose, leading to a shift in the absorption band. The OH stretching vibration is sensitive to hydrogen bonding. When microcapsules are introduced, these OH groups may form new hydrogen bonds with the materials of the microcapsules (such as ethylcellulose), leading to a shift in the OH stretching frequency. The new hydrogen bonds could be weaker or different in nature, causing a shift to lower wavenumbers [18,23].

The peak at 1046 cm^{-1} in the FTIR spectrum of the microcapsules (MCs) made of ethylcellulose and coco oil can be attributed to the C–O–C stretching vibrations of the ethylcellulose. Ethylcellulose (EC) contains ether linkages (–C–O–C–), which typically show strong absorption bands in the range of 1050 to 1150 cm^{-1}, as can be observed in Figure 4 for the pure EC. Therefore, the peak at 1046 cm^{-1} is characteristic of the ether bonds present in the ethylcellulose polymer used as the shell material in the microcapsules. This absorption indicates the presence of the ethylcellulose component within the microcapsules.

3.2. Nanofibers Characterization

Covering fabric with microcapsules in a PVA nanofiber veil serves the primary purpose of protecting the microcapsules. The presence of a PVA nanofiber layer ensures that the microcapsules, which are applied to the cotton fibers and cannot react with the fibers, remain intact and protected from external factors that could compromise their integrity and functionality. This protective layer is particularly important in maintaining the microcapsules' stability and efficacy, especially when they are subjected to various environmental conditions or mechanical stresses such as fabric manipulation. In order to protect the coco microcapsules, a PVA nanofibers veil was placed on the fabric. For SEM observation part of the veil was peeled off. In Figure 5, the microcapsules can be observed on the cotton fibers where the nanofiber veil is partially removed.

The FTIR spectra (Figure 6) shows the spectrum for PVA polymer, the one for cotton fabric (co), and the ones for the fabric with microcapsules without nanofibers (MC) and with PVA nanofibers (NF).

The analysis of the spectra shows how PVA nanofibers, when compared with Figure 4, offer the same shift towards lower wavenumbers for the OH stretching region around 3300 cm^{-1}. The ethylcellulose is also confirmed as the observable peak centered around 1046 cm^{-1}. A peak centered around 1840 cm^{-1} is clearly appreciable for cotton fabric with microcapsules and nanofibers and was not observed before placing the PVA nanofibers (MC). When compared with the pure PVA nanofiber spectrum it is evident this is due to the PVA nanofiber layer placed on the fabric. However, in general FTIR spectroscopy, the region around 1800 cm^{-1} is often associated with carbonyl (C=O) stretching vibrations. This can appear in various forms depending on the specific chemical structure and interactions within the material. For PVA, which typically does not contain carbonyl groups in its unmodified form, a peak in this region could indicate a chemical modification or the presence of impurities which contain the carbonyl group [23].

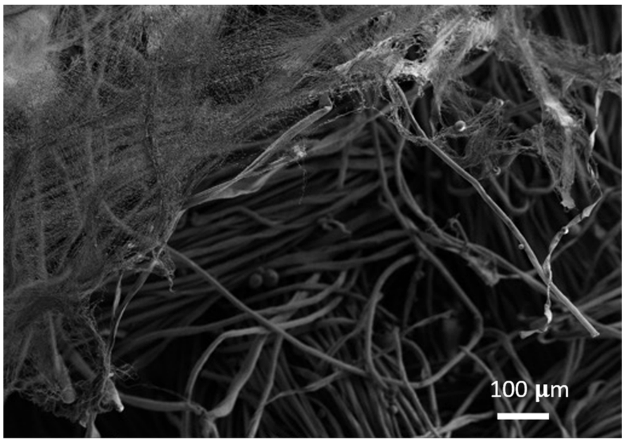

Figure 5. SEM image for coco oil microcapsules on cotton fabric and covered by PVA nanofibers once the fabric has been heated for five cycles.

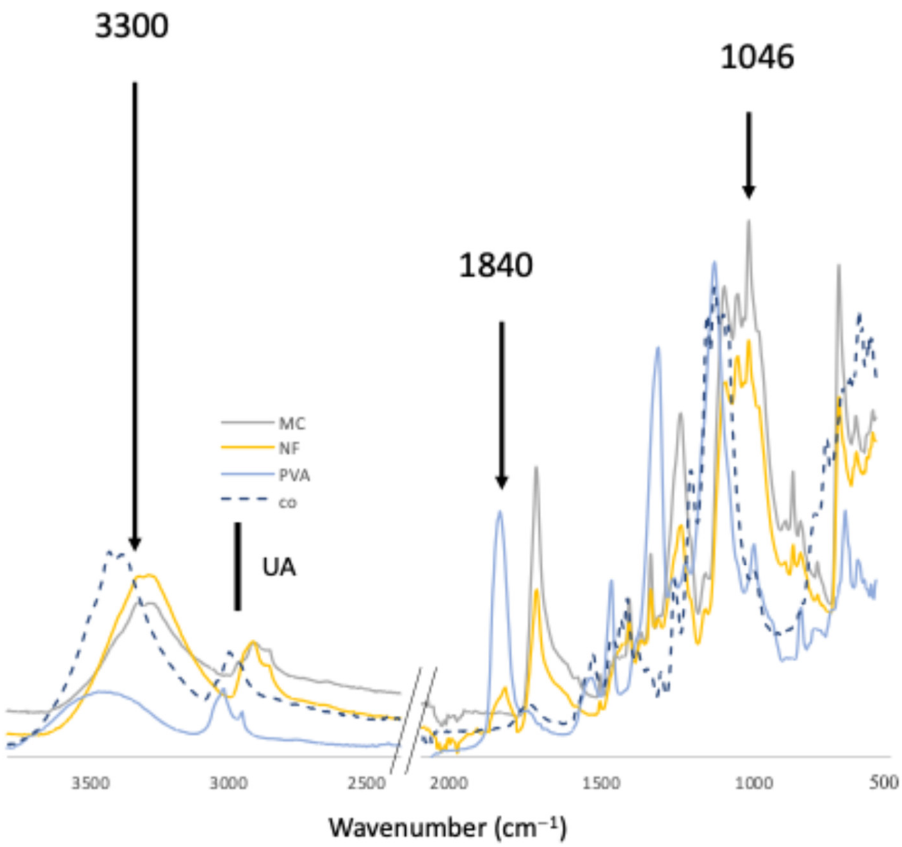

Figure 6. FTIR spectra for PVA nanofibers covering coco oil microcapsules on cotton fabric once the fabric has been heated for five cycles.

3.3. Thermal Treatment

Regarding the thermal behavior of fabrics, heating and cooling was applied, and the fabric temperature was measured every 15 min. Figure 7 shows the heating and cooling response from cotton fabric with different treatments: the cotton fabric itself (Co) is compared with cotton fabric covered by a nanofiber veil (NF); cotton fabric with coco microcapsules (MC); and cotton fabric with coco microcapsules and covered with a nanofiber veil (MC-NF). All of them are presented with solid lines. The graph shows temperature changes over time, and it reveals different behavior for each sample at the heating process whereas only small differences can be appreciated for the cooling process. Furthermore, dashed lines demonstrate the thermal behavior from cotton fabrics with microcapsules once they had been shaken.

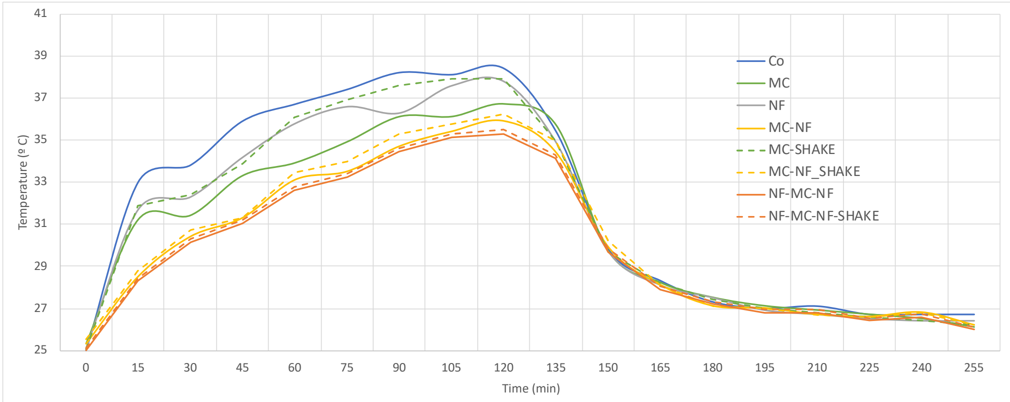

Figure 7. Thermal behavior of different fabrics during the heating and cooling process. Solid lines indicate fabric after treatment. Dashed lines indicate fabrics treated and shaken.

There are two distinct phases visible in the graph: heating until 120 min and cooling from 120 to 255 min. During the heating phase, all fabrics show a rise in temperature over time. The rate of temperature increase is evident for every fabric, and shows that the cotton without treatment has the highest one (38.4 °C). Cotton fabrics with nanofibers (NF) show a slight decrease in the heating, probably due to the fact that the high specific surface does not allow the air to pass through and establishes some kind of isolation, reducing the heating by around 0.5 °C. The nanofiber layer shows a considerable difference in heat transmission, and as could be expected, it reduces the fabric temperature [16]. Apparently, the nanofibers absorbed part of the energy, preventing the heat from reaching the top of the surface as fast as in the cotton fabric. Anyway, it is the microcapsules' presence on the fabric MC and MC-NF when the temperature is reduced around 1.5 °C or 2.5 °C, respectively. The lowest temperatures reached during the heating phase are for MC-NF and MC, suggesting these fabrics may have better heat retention properties or lower thermal conductivity. These are practically unaltered when a second layer of nanofibers is added (NF-MC-NF), regardless of whether the fabric is shaken or not. This can be due to the presence of coco oil microcapsules. The cooling phase shows a similar decrease in temperature for every fabric. According to the final temperature, each specimen tested seems to approach a similar temperature by the end of the cooling phase, indicating they may reach thermal equilibrium with the surrounding environment over time.

The thermal behavior could be due to the coco oil presence; however, to determine the phase change material (PCM) on the coco microcapsules, different cycles were studied (five cycles). Both the original MC-NF fabric and the MC-NF fabric after five cycles follow a similar temperature curve (Figure 8). This suggests that the fabric's thermal response is consistent and does not degrade significantly after repeated cycles. The peak temperature reached during the heating phase appears to be almost identical for both the initial test and the fabric tested up to 50 heating and cooling cycles. This indicates that the heat capacity and thermal conductivity of the fabric remain stable. During the cooling phase, both fabrics again show very similar behavior, closely following each other as they return to the baseline temperature. There is no evident thermal fatigue or change in the cooling rate.

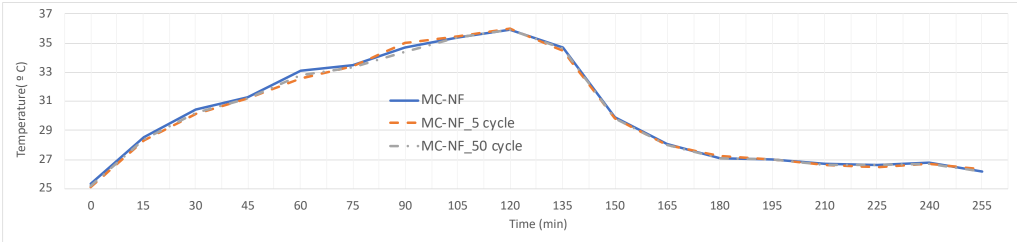

Figure 8. Thermal behavior for cotton fabric with coco microcapsules and nanofibers during the heating and cooling process for one, five, and fifty cycles.

4. Discussion

Investments in thermal insulation not only yield economic benefits by lowering energy costs but also offer substantial environmental advantages. For instance, buildings with enhanced insulation require less energy for temperature regulation, thereby reducing CO_2 emissions [24]. Furthermore, sustainable insulation materials often have superior lifecycle properties, contributing to long-term environmental sustainability. These materials are designed to meet rigorous energy efficiency standards while also being safe and environmentally friendly.

The broad size distribution of microcapsules may influence the thermal properties and application performance of the microcapsules. Smaller microcapsules provide a larger surface area-to-volume ratio, which can enhance heat exchange rates. The SEM analysis provided valuable insights into the microstructural characteristics of the cotton fabric treated with coco oil as a phase change material (PCM). The observed spherical formations on the cotton fibers are indicative of successful encapsulation of the coco oil. These spheres are likely the result of the coco oil being encapsulated within the fibers, forming microcapsules that can effectively manage thermal energy through phase transitions.

The thermal cycling test, consisting of five heating and cooling cycles, is crucial in demonstrating the stability and functionality of the encapsulated coco oil. Phase change materials like coco oil absorb heat as they melt from a solid to a liquid state and release heat when they solidify. The retention of spherical shapes after multiple thermal cycles suggests that the encapsulation process was effective in preventing oil leakage. If the coco oil were not properly encapsulated, it would likely seep out during the melting phase, leading to irregular shapes or loss of material, which would be detectable in the SEM images.

Moreover, the consistency in maintaining the round shapes of the microcapsules throughout the thermal cycles highlights the robustness of the encapsulation. This is essential for the practical application of PCMs in thermal insulation, as it ensures long-term reliability and efficiency. The encapsulated coco oil can repeatedly absorb and release thermal energy without degradation, which enhances the thermal regulation properties of the fabric.

Additionally, the encapsulation of coco oil within the cotton fabric offers several advantages. It integrates the PCM directly into the fabric structure, providing a lightweight and flexible thermal management solution. This can be particularly beneficial in applications where traditional insulation materials are not suitable due to weight or rigidity constraints.

The FTIR spectra reveal distinct chemical modifications in the cotton fabric post encapsulation with microcapsules containing coco oil and ethylcellulose. Specific absorption bands corresponding to functional groups in coco oil and ethylcellulose confirm their presence on the fabric's surface. For instance, the characteristic peaks of coco oil, such as those associated with the C–H stretching vibrations of aliphatic chains, and the C=O stretching vibrations of esters, are evident in the FTIR spectra. Similarly, ethylcellulose is identifiable through its characteristic ether (C–O–C) and hydroxyl (O–H) stretching vibrations.

These spectral signatures substantiate the hypothesis that coco oil and ethylcellulose have been successfully integrated into the cotton fabric, forming microcapsules. The presence of these compounds on the fabric's surface strongly indicates that the microencapsulation process was effective. Microencapsulation is crucial as it ensures the stability and functionality of the phase change material (PCM), in this case, coco oil, by preventing leakage and maintaining structural integrity during thermal cycling.

The integration of coco oil microcapsules (MC) and a PVA nanofiber veil onto cotton fabric exhibits a significant improvement in thermal management properties. Individually, both the microcapsules and the nanofibers contribute to temperature regulation, but when combined, they produce a synergistic effect that enhances heat retention and overall thermal insulation performance.

The coco oil microcapsules are effective in reducing the fabric temperature during cooling due to their phase change properties. As the temperature rises, the coco oil within the microcapsules absorbs heat by melting, thus preventing the fabric from heating up rapidly. Conversely, during cooling, the coco oil solidifies, releasing the stored heat and maintaining a stable temperature for an extended period. This phase change behavior is crucial for creating a thermally regulated environment, which is beneficial for applications requiring consistent temperature control.

The PVA nanofiber veil adds another layer of thermal insulation by acting as a protective barrier. The nanofibers provide additional insulation by trapping air within their structure, which reduces thermal conductivity. This effect is similar to that of traditional insulating materials, where the trapped air acts as a thermal barrier. Moreover, the nanofiber veil also plays a protective role by encapsulating the microcapsules, preventing them from being released during fabric manipulation or wear. This ensures that the microcapsules remain intact and functional throughout the lifecycle of the fabric.

When the coco oil microcapsules and the PVA nanofiber veil are used together, they create a synergistic effect that significantly enhances the thermal insulation properties of the fabric. The combined system benefits from the phase change properties of the microcapsules and the insulating characteristics of the nanofibers. This dual-layer approach not only improves heat retention but also ensures that the fabric's surface temperature remains manageable, preventing it from becoming excessively hot or cold. The nanofiber veil ensures the structural integrity of the microcapsules, protecting them from mechanical stress and extending their functional lifespan.

This synergistic combination is particularly advantageous in applications where thermal regulation is critical, such as in protective clothing, outdoor gear, or thermal blankets. The enhanced heat retention and stability provided by the microcapsules and nanofiber veil can improve comfort and energy efficiency, making the fabric suitable for a wide range of thermal management applications not only for textile goods but for construction, civil engineering, food packages, cargos, etc. [25–28].

The close match between the first and the fiftieth heating and cooling cycle implies that the ethylcellulose shell created to retain the coco oil inside is hard enough to resist the increase in volume due to the oil expansion when heated. Furthermore, as the fabric's thermal behavior is stable across multiple heating and cooling cycles it evidences the reversibility of the process, as the ethylcellulose shell prevents the oil from leakage when it melts due to the increase in temperature. This could be important for applications where the fabric is subject to thermal cycling, such as isolation materials in buildings. The reversibility of the thermal treatment confirms that the ethylcellulose is part of the shell and the coco oil is kept inside the shell. Furthermore, the SEM study with microcapsules on the surface confirms that the ethylcellulose shell is hard enough to bear the oil pressure when it melts, as the shell is not broken and prevents oil leakage. Thus, this confirms that the little spheres observed by SEM are something more than the solid coco oil.

5. Conclusions

The SEM analysis confirms the successful encapsulation of coco oil in the cotton fabric, as evidenced by the spherical formations and their stability through multiple thermal cycles. This demonstrates the potential of using encapsulated PCMs for efficient thermal insulation, combining the benefits of natural materials like cotton and coco oil with advanced thermal management capabilities. Despite this fact, there is still research to conduct on solvent recovery; such innovations are crucial for developing potential sustainable and effective thermal insulation solutions.

The use of coco oil microcapsules and a PVA nanofiber veil on cotton fabric results in superior thermal insulation performance. The microcapsules reduce fabric temperature fluctuations through phase change properties, while the nanofiber veil enhances insulation and protects the microcapsules. Together, they create a robust, thermally efficient fabric that maintains a stable temperature and ensures long-lasting performance.

The conclusions drawn from this study underscore the transformative potential of PCM-incorporated textiles across various sectors with biodegradable materials. In industrial applications, these textiles can be used to enhance energy conservation and manage thermal processes within manufacturing environments. For civil engineering, the integration of such materials in the construction of buildings promises improved insulation properties, potentially reducing heating and cooling demands and contributing to energy-efficient building designs. In the automotive industry, the application of these thermo-regulated textiles in vehicle interiors could offer greater comfort for passengers by stabilizing cabin temperatures, as well as reducing the energy consumption of climate control systems. Furthermore, the protection by the PVA nanofibers ensures that these materials can withstand the manipulation. This technology heralds a new era of smart materials, paving the way for broader applications in areas that stand to benefit from sustainable and efficient thermal management solutions.

The integration of PCMs into textiles, even if initially showing modest insulation improvements, opens the door to multifunctional fabrics. These fabrics can provide not only thermal regulation but also other benefits such as enhanced comfort, protection against temperature fluctuations, and potential energy savings in clothing or building materials. The methodology demonstrated in this study shows potential for scalability and practical implementation. By refining the production process and ensuring that microcapsules adhere well to fabrics and maintain their functionality over time thanks to PVA nanofibers, this study lays the groundwork for commercial applications across various fields, contingent upon the optimization of conditions, including clothing, bedding, building insulation, and even medical textiles.

The approach of using ethylcellulose for encapsulation and PVA nanofibers for additional stability is innovative. It highlights the potential for using biocompatible and sustainable materials in creating advanced functional textiles. Further research could optimize these materials, leading to greener and more sustainable insulation solutions.

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