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100% BIO-BASED MICROENCAPSULATED PHASE CHANGE MATERIALS AS REGULATOR OF TEMPERATURE OF TEXTILE FABRIC

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Abstract: *Phase change materials (PCM) are very useful in many fields due to their capacity to absorbe and release heat energy when it is necessary. In this paper it was managed to microencapsulate 100% biodegradable PCMs to apply them into textile fiber in order to regulate its temperature. The microencapsulation method used in the present work was solvent evaporation with oil-in-water emulsification. Scanning Electron Microscope (SEM) was conducted to confirm the successful microencapsulation. Differential Scanning Calorimeter (DSC) was used to evaluate thermal properties of the core material and also efficience of the microencapsulation. Coconut oil and bee wax were used as PCM due to their melting point (Coconut oil - 23°C, Beeswax – 60°C) and, Polylactide (PLA) and Ethyl cellulose (EC) as shell materials because of their biodegradable nature. The microPCMs were applied onto the non-woven PLA with the help of a binder which in this case was calcium alginate and chitosan and then it was confirmed by SEM the microcapsules were inside the fabric and stick them to it. The thermal regulating properties of modified textiles were investigated by an IR camera. The results obtained from thermal analysis of samples showed that the temperature of the unmodified sample decreases faster than the modified sample.*

Key words: *Coconut oil, bee wax, polylatide, ethyl cellulose, calcium alginate, chitosan.*

1. INTRODUCTION

Phase Change Materials (PCMs) are substances that can absorb and release large quantity of latent heat energy during the change of their phase. This phase change depends on the specific melting point of the PCM and with it calculates the enthalpy of the reaction. Phase change materials (PCM) can be employed in many fields because of their capacity to absorb and release energy when it is necessary.

There are many types of organic PCM with phase change temperatures from -5 ° C to 190 ° C. [1-5] Speaking of thermal comfort applications in textiles or building, PCM's and their mixtures are used with phase change temperatures between 18 ° and 65 ° C.

1.1. Solvent Evaporation

In this microencapsulation technique, the polymer must be dissolved in a volatile solvent, and mixed in a water-based emulsifying solution, in which the solvent must be evaporated, releasing the polymer that encloses the core material also dissolved in the solvent. This technique is widely used in the pharmaceutical industries. Initially the coating solution is prepared by dissolving the coating polymer in a volatile solvent that is immiscible in the emulsifier solution. After that depending on the nature of the core material (hydrophobicity or hydrophilicity) it is dissolved or dispersed in the polymeric coating solution. The mixture is added to the emulsifying solution with continuous stirring until the solvent is distributed in the aqueous phase and evaporated. At this point the material coat contracts around the core material and results in hardened microspheres [6].

1.2. Textile and clothing applications of PCMs.

Textile products and clothing have been fundamental materials for the human being to feel comfortable and protected against certain external factors and thus ensure that the physical conditions of our body are adequate for survival. This protection can comprise a series of functions, maintaining the appropriate environment for the body [7-13]. One of the main functions of clothing is to protect the body from the climate by creating a microclimate-stable next to the skin to support the system of thermal regularity of the body for which the textile fibers must have certain properties.

1.3. Incorporation of microencapsulated PCMs to fibers, fabrics and foams.

The application of PCM in textiles brings many advantages for example: they are very easy to apply to textiles, fabrics, nonwovens and fibers, they usually do not affect the properties that the textile already has, they have a shelflife on a garment that permits normal fabric-care processes, they promote mixing of core materials, they reduce the reactivity of the PCMs with the external environment, they decrease the evaporation of the core material to the outside environment, they increase the area of heat transfer and they provide a constant volume in the core material [8,14-15]. The composition of the material shell and the amount of microcapsules added to the structure are the main factors that provide a suitable management in the textile product. When a sufficient amount of microPCM is added to a textile structure as a suitable component for coating of fabric, fibers, yarns and breathable foams a suitable thermal management in the textile product, which satisfies the end-use needs for which it was created. These will continue to function as long as the coating or the fibers remain intact otherwise the properties provided will be lost [16-17].

2. EXPERIMENTAL.

In the present paper it is shown the procedure to microencapsulate organic phase change material (bee wax and coconut oil) by interphase precipitation during evaporation of the solvent from water/oil emulsion. 100% biodegradable polymer materials were used as shell (Poly(lactic acid) (PLA) and Ethyl cellulose (EC)).

2.1 Materials.

- Coconut Oil as phase change material - commercial product.
- Beeswax as phase change material - commercial product.
- Different sort of Poly(lactic acid) (PLA) as shell material – a commercial product of NatureWorks®LLC
  - PLA 4060D - a amorphous resin
  - PLA 6201D - a thermoplastic fiber-grade resin
  - PLA 6400D - thermoplastic fiber-grade resin
- Ethyl cellulose (EC) as shell material - a commercial product manufactured by Aldrich (4cP viscosity measured for 5% toluene/ethanol 80:20 solution).

- Dichloromethane, chloroform, ethyl acetate were used as organic solvent - a commercial product manufactured by Chempur.
- Poly(vinyl alcohol) (PVA) (Mw = 1000, manufactured by Aldrich) as a surfactant/emulsifier.
- For the coating of the PCM microcapsules on textile a coating binder was used as follows:
  - Sodium alginate (ALDRICH product of 15-20 cP viscosity, (1% solution in H_2O, temperature 25ºC)) at a 1,5% w/w. concentration of in aqueous solution, and 10% w/w. calcium chloride dihydrate (Chempur) solution – as a coagulant.
  - Chitosan (ALDRICH product of 200-800 cP viscosity, (1 wt.% in 1% acetic acid, 25ºC) at a 1% w/w concentration of in 1% acetic acid solution.
- Polylactide nonwoven (a commercial product manufactured by FET Taiwan, SLN-2539W5, 1,5 den/38 mm FB) was applied for the modification with PCM microcapsules.

2.2. Methods and results

Microcapsules preparation.

Microcapsules with polylactide or ethyl cellulose shell were obtained by solvent diffusion from the emulsion. The oil-in-water emulsion was obtained by homogenization of the organic phase (polylactide or ethyl cellulose and coconut oil or bee wax in organic solvent) in the aqueous phase containing a surfactant/emulsifier – 1% solution of poly(vinyl alcohol). In this technique, a poly(vinyl alcohol)(PVA) emulsifier is used to reduce the interfacial tension between the dispersed droplets and the continuation phase, in addition to protecting the droplets from aggregation.

The microcapsules were created after complete removal of the solvent from the droplets by evaporation during the mixing process. The manufactured microcapsules were washed, decanted and dried. Among the effective parameters affecting the microcapsule preparation process can be mentioned: the type and amount of polymer used as the shell layer, the type and amount of core material, the rate of homogenization or the type of solvent used.

Scanning Electron Microscopy (SEM).

The surface structure of the obtained microcapsules was analyzed using microphotography obtained by means of the High-resolution electron microscope Nova Nanosem 230 and Scanning electron microscope JSM-5200LV (JEOL). The observations were carried out under high conditions at 10 kV and different magnifications.

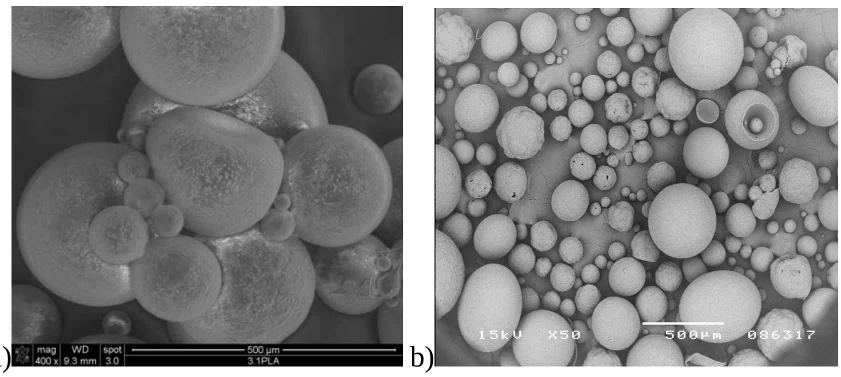
a)  b)

Fig. 1: *SEM images of: (a) sample COPLA3 made with PLA 4060D and (b) sample COPLA14 made with PLA 6201D.*

This technique it was also used to make the measurements of the microcapsules diameter.

Differential Scanning Calorimeter (DSC).

DSC measurements were done on a differential calorimetry of TA Instruments Thermal Analysis -- DSC Standard Cell RC equipped with a refrigerated cooling system. DSC analyses were conducted under a nitrogen atmosphere and the temperature interval cycle was set to -40 - 40°C (Coconut Oil) or 0 – 80oC (Bee wax) at 10°C/min. The tests were made in order to identify the thermal capacity and phase change enthalpy values. The latent heat capacity of the Bio-based PCM microcapsules was determined by calculating of the area under the peaks that represent the solid–solid and solid–liquid phase transitions. Moreover DSC gave the melting point of the core materials thereby it could know the temperature which they would start to absorb or release heat energy depending on the cooling or heating process.

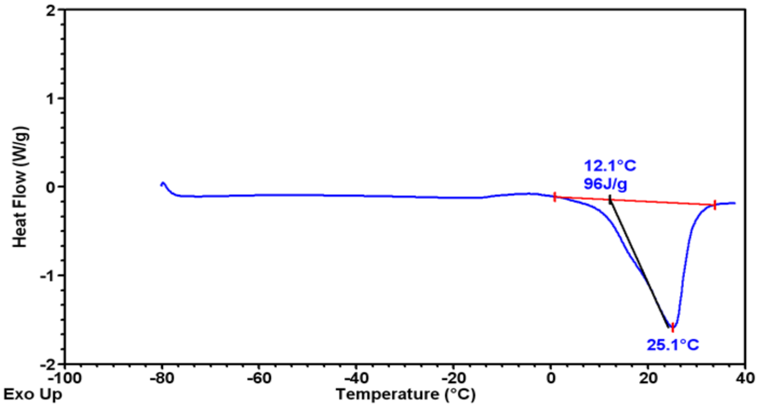


Fig. 2: *DSC thermograms of pure coconut oil: heating cycle.*

Infrared thermography.

Infrared thermography was used to investigate the thermal changes in the fabric, i.e. the temperature distribution but also the thermoregulation effect. Thermo-graphic camera is used to measure the amount of heat energy that some material releases and the most important thing is how it decreases or increases the temperature of that material after time. In addition, this technique provides visual information of the temperature changes that are occurring in the material that has been exposed to temperature, either low or high.

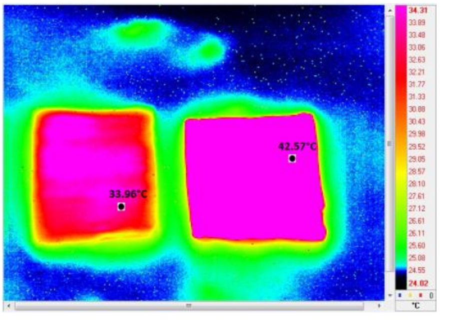


Fig. 2: *IR images of non-woven PLA unmodified (sample on the left) and modified with COEC3 (sample on the right) at 0 sec.*

3. CONCLUSIONS

The important contribution in this work is the creation of a new technology of textile PCM application being environmental friendly with 100% biodegradable materials, useful and also inexpensive. Coconut oil and bee wax are candidates for core as temperature regulator due to their capability to absorb and release large amount of heat energy by changing their phase. It is possible to microencapsulate these two organic PCMs in polylactide (PLA) and ethyl cellulose (EC) as well. PLA as shell material is the best option due to this polymer is enough resistance to the normal situations woven or non-woven textile materials are exposed. Ethyl Cellulose (EC) is also a good option due to its biodegradable properties. The microcapsules were obtained as a result of interphase precipitation during evaporation of the solvent from water/oil emulsion.

The method used to obtain microcapsules allowed to obtain microcapsules with an average diameter of 26 μm. This indicates that the microcapsules are tiny and easy to coat onto the fabric surface.

The thermal regulating properties of modified textiles were investigated. The results obtained showed that the temperature of the unmodified sample decreases faster than the modified sample, which means that the modified sample can absorb more thermal energy and stay in it for a longer time due to the microencapsulated phase change material that is inside. Modified nonwovens with microcapsules containing coconut oil or bee wax provide cooling effect, which confirms the effectiveness of the modification.

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