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Development of thermo-regulating textiles using paraffin wax microcapsules

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ABSTRACT

Polystyrene microcapsules containing paraffin wax were synthesized by suspension like polymerization process, and their suitability for textile applications was studied. Fixation of microcapsules into textile substrate by means coating technique was tested. Different coating products and the mass ratio of microcapsules to coating binder were studied in order to get thermal comfort in fabrics. PCM microcapsules were successfully incorporated into the textiles by using TEXPRINT ECOSOFT N10® and WST SUPERMOR® as polymeric binders without modifying the original properties of textile. The coating fabric with 35 wt.% of microcapsules added related to coating binder (WST SUPERMOR®) showed a energy storage capacity of 7.6 J g^{-1}, a high durability and an adequate stability after washing, rub fastness and ironing treatments.

A difference of 8.8 °C for 6 s was observed for thermo-regulating textiles obtained as compared with a coated one without microcapsules.

1. Introduction

The textile industry has reacted slowly to the many opportunities that microencapsulation can offer. Until now, its exploitation in textiles has been limited owing to a lack of awareness across the industry, and the relative high costs of the global processing. Nowadays, the number of commercial applications of microencapsulated PCM in the textile industry continues to grow into textiles with new properties and added value, e.g., medical textiles and technical textiles [1,2].

A thermo-regulating fabric is an intelligent textile that has the property of offering suitable response to changes in external temperature changes or to external and environmental stimuli. The level of thermal comfort depends on the heat exchange between the human body and the environment that surrounds it; many efforts have been devoted to induce a thermoregulatory effect into textiles. This effect can be obtained with the presence of microcapsules containing phase change materials (PCM) [3].

PCM materials absorb energy during the heating process as phase change takes place and release energy to the environment in the phase change range during a reverse cooling process [4]. More than 500 different PCMs, including water (ice), are known. One of the most commonly used PCMs in storing thermal energy is the paraffin wax [5]. PRS® paraffin wax was used in this work as PCM due to its high heat storage capacity, easy availability and low cost. This paraffin wax cannot be incorporated directly into textiles because of their low melting point and, therefore, need to be microencapsulated [6].

The microencapsulation of PCMs involves enclosing them in thin and resilient polymer shells so that the PCMs can be changed from solid to liquid and back again within the shells [7]. Applications of PCM containing microcapsules into textiles include apparel, blankets, medical field, insulation, protective clothing and many others. The technology for incorporating PCMs into textile structure to improve their thermal performance was developed in the early 1980s under NASA research programme [8].

Microcapsule production may be achieved by means of physical and chemical techniques. The use of some techniques has been limited to the high cost of processing, regulatory affairs, and the use of organic solvents, which are concern for health and the environment [9]. In this way, a method based on a suspension free radical polymerization process for the encapsulation of phase change materials has been selected [10–12]. This PCM encapsulation method is simply, inexpensive and technically easy.

Although PCM microcapsules impart thermoregulatory properties to materials, they can affect other comfort-related properties and manufacturing of textiles, especially when the topical application of microcapsules results in drastic changes in the surface characteristics of materials. The extent of change in these properties depends on the loading amount of PCM microcapsules. Therefore, the properties of fabrics treated with PCM microcapsules need to be measured and considered before use in a garment [13].

The microcapsules can be applied by stamping works, exhaustion dyeing, impregnation, spraying and coating or by direct incorporation in the fibre without highly modifying its touch

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and colour [14–16]. Shin et al. [13] incorporated melamine-formaldehyde microcapsules containing eicosane on polyester knit fabrics by means pad dry cure method with a polyurethane binder. Mengjin et al. [17] developed a new kind of thermo-regulating fibre based on PVA and paraffin. Recently, Koo et al. [18] have attempted to demonstrate the application of PCM microcapsules to waterproof nylon fabrics by using a dual coating method.

The aim of this work was to develop a thermo-regulating textile by using PCM microcapsules and a coating technique. For that, the influence of different coating formulations and mass ratio of microcapsules to coating formulation were evaluated in order to obtain an adequate thermoregulatory textile. Furthermore, a study of thermoregulatory effect of the coating fabrics produced was carried out using an infrared thermography (IR) camera. Subsequently, after the fixation of the PCM microcapsules on the textile substrate, standardized treatments as washing, rub fastness and ironing were carried out on fabrics to evaluate its durability and mechanical stability. Thermal properties of textile samples were examined by differential scanning calorimetry (DSC). Furthermore, environmental scanning electron microscopy (ESEM) and optical microscopy (OM) techniques were used to check the presence, surface distribution, preferred join position and to analyse the structure of microcapsules into the textile.

2. Experimental

2.1. Materials

For the microcapsules synthesis the following materials were used. Styrene (99 wt.%) of reagent grade (Merck Chemical) was washed with sodium hydroxide to remove the inhibitor and calcium chloride as desiccant was used. Benzoyl peroxide (97 wt.%) was used as initiator (Fluka Chemical). PRS® paraffin wax (478 g mol^{-1}) was used as core material. This paraffin was a mixture of hydrocarbons C_{19}–C_{27} produced and commercialized by the petrochemical company Repsol YPF (Spain), being its energy storage capacity of 202.6 J g^{-1} and its melting temperature range of 40–45 °C. Polyvinylpyrrolidone (K30, 40,000 g mol^{-1}) of reagent grade (Fluka Chemical) was used as stabilizer and methanol to pour the samples. All these reagents were used as received.

A tubular type Shirasu porous glass membrane with pores sizes of 5.5 μm was used to produce a narrow microcapsules size distribution. At the end of every experiment, the used membrane has been retrieved by a treatment with sodium dioctyl sulfosuccinate (AOT) (Fluka Chemical, 96 wt.%), ethanol (Panreac Chemical, 96 wt.%) and hydrochloric acid (Prolabo Chemical, 2 mol L^{-1}) reagents.

The synthesis process was previously described elsewhere [12]. The average particle size in number of the microcapsules obtained was 4.53 μm, which was checked by Malvern Mastersizer Hydro 2000 SM light scattering apparatus. DSC measurements showed an average energy storage capacity of 104.7 J g^{-1} between 0 and 55 °C for the paraffin wax microcapsules.

For the fixation of PCM microcapsules on the textile substrates different commercial coating binders were used: RESIN CENTER BC® (supplied by Color-Center Ltd.), PRIMAL E-358 EMULSION® (Rohm and Haas Ltd.), TEXLAKCOLOR SF® (Eurotext), TEXPRINT ECOSOFT N10® (Eurotext), MINERSTAR NEUTRO® (Minerva color Ltd.), WST SUPERMOR® (Minerva color Ltd.) and CENTERPRINT ECO® (Color-Center Ltd.).

The fabric used was 100% cotton, characterization results of which are shown in Table 1.

Samples were named according to coating binders and wt.% of microcapsules employed. Then, CENTER-30 was a thermo-regulating textile produced using RESIN CENTER BC® as coating binder and 30 wt.% of microcapsules with respect to the weight of coating binder.

Table 1
Fabric substrate characterization.

Sample	Warp (yarn cm^{-1})	Weft (yarn cm^{-1})	Weight (g m^{-2})	Wide (mm)	Length (mm)
Cotton	41.80	25.87	158.45	200	290

2.2. Addition of the microcapsules to the fabrics

Microcapsules were applied into the fabrics by coating technique. For this purpose, a motorized film applicator from Elcometer model 4340 was used in accordance with ASTM D-823C [19]. The coating formulation was prepared mixing homogenously PCM microcapsules and the coating binder. In this study different commercial binders were used for the fixation of the PCM microcapsules on the textile substrate using 345 g of commercial binder per m^2 of fabric.

The virgin textile was set on the motorized film applicator surface assuring the fabric with clips. After that, a thickness of 0.01 mm was selected with standard guides due to a better appearance of produced fabric. The position of the motorized film applicator and the selection thickness was carried out manually. A dragging speed of 5 mm s^{-1} was chosen to allow a homogeneous coating along the film applicator.

Finally, the dry fabric substrate impregnated with the coating formulation and the microcapsules was cured at 95 °C during 11 min.

2.3. Characterization

2.3.1. Optical microscopy (OM)

A Nikon Eclipse E 200 microscopy equipped with the software Nikon Application Suite Interactive measurement was used to analyse the microcapsules.

The optical microscopy technique provides images of microcapsules with a good resolution that allows a study of its morphology, fixation and durability using the transmitted light and reflection mode.

2.3.2. Environmental scanning electron microscopy (ESEM)

ESEM was used to analyse the fixation and integrity of PCM microcapsules into the coating textiles. Textile samples were observed by using XL30 (LFD) ESEM with a wolfram filament operating at a working potential of 20 kV.

2.3.3. Differential scanning calorimetry (DSC)

Measurements of phase change temperatures and energy storage capacities of different materials employed and obtained were performed in a differential scanning calorimetry model DSC Q100 of TA Instruments equipped with a refrigerated cooling system and nitrogen as the purge gas. Measurements were done varying the temperature in the range from −30 to 80 °C with a heating rate of 10 °C/min.

Different samples of each experiment were analysed at least three times and the average value was calculated. DSC analyses of coating textiles from random areas were done.

2.3.4. Infrared thermography (IR)

The infrared thermography allows measuring and observing surface temperatures with precision without having contact. Thermography utilizes the IR spectral band. To perform the temperature distribution analysis, a thermal vision camera was used to detect IR radiation and convert this information into an image, where each

Table 2
Energy storage capacity, final appearance and texture of the coating textile with microcapsules produced using different coating binders.

Samples	Coating binder	Energy storage capacity (J g^{-1})	Physical aspect	Texture
CENTER-30	RESIN CENTER BC®	0.92	Irregular	Rough
PRIMAL-30	PRIMAL E-358-ER EMULSION®	5.23	Regular	Rough
TEXLAK-30	TEXLAKCOLOR SF®	3.32	Irregular	Rough
ECOSOFT-30	TEXPRINT ECOSOFT N10®	4.85	Regular	Smooth
MSTAR-30	MINERSTAR NEUTRO®	4.74	Irregular	Smooth
SUPERMOR-30	WST SUPERMOR®	5.89	Regular	Smooth
CPRINT-30	CENTERPRINT ECO®	3.50	Regular	Rough

pixel corresponded to a temperature value. In these experiments, the temperature range used was from 21 to 37 °C.

To examine the thermoregulatory effect of the thermo-regulating textiles a thermal vision camera Fluke Ti25 was used. This dispositive allows obtaining thermal and visual images in the range of temperatures from −20 to 250 °C with a precision of ±5 °C. Images were downloaded using Fluke SmartView^{TM} software for analysis.

2.3.5. Washing test

To test washing durability the coating textiles were treated on a short time programme in a Gyrowash for 30 min at 40 °C, in accordance with ISO Standard 105-C06 [20]. A solution of ECE detergent type B was prepared with a concentration of 4 g L^{-1} and sodium perborate monohydrate was added in a concentration of 1 g L^{-1}. Then, sample was introduces in the laundry solution bath with a volume of 125 mL using 10 stainless steel balls. When a cycle was finished, samples were washing twice with deionized water for 1 min in two different baths with 100 mL at 40 °C to take out the water excess. After that, sample was dried at 60 °C in air. Finally, after this treatment the thermo-regulating textiles were examined by DSC and OM.

2.3.6. Rubbing test

The rubbing test of thermo-regulating textiles was carried out, in accordance with UNE EN ISO 105-X12 Standard [21], using a standardized Crockmeter. Each sample was rubbed for 10 cycles by a finger covered with a fabric sample at a pressure of 9 N under dry conditions.

2.3.7. Ironing test

The ironing test allows ironing of the coating fabric under dry conditions with a thermal dispositive called Termofix to determine the strength to dry heat of the fabric using UNE EN ISO 105-X11 Standard [22]. All samples were ironed at 150 °C during 15 s at 4 kPa.

3. Results and discussions

3.1. Coating formulation selection for the fixation process

Commercial lacquers and coatings have different properties as chemical nature, affinity with the substrate textile, temperatures and cured times. Those properties could make that certain coating

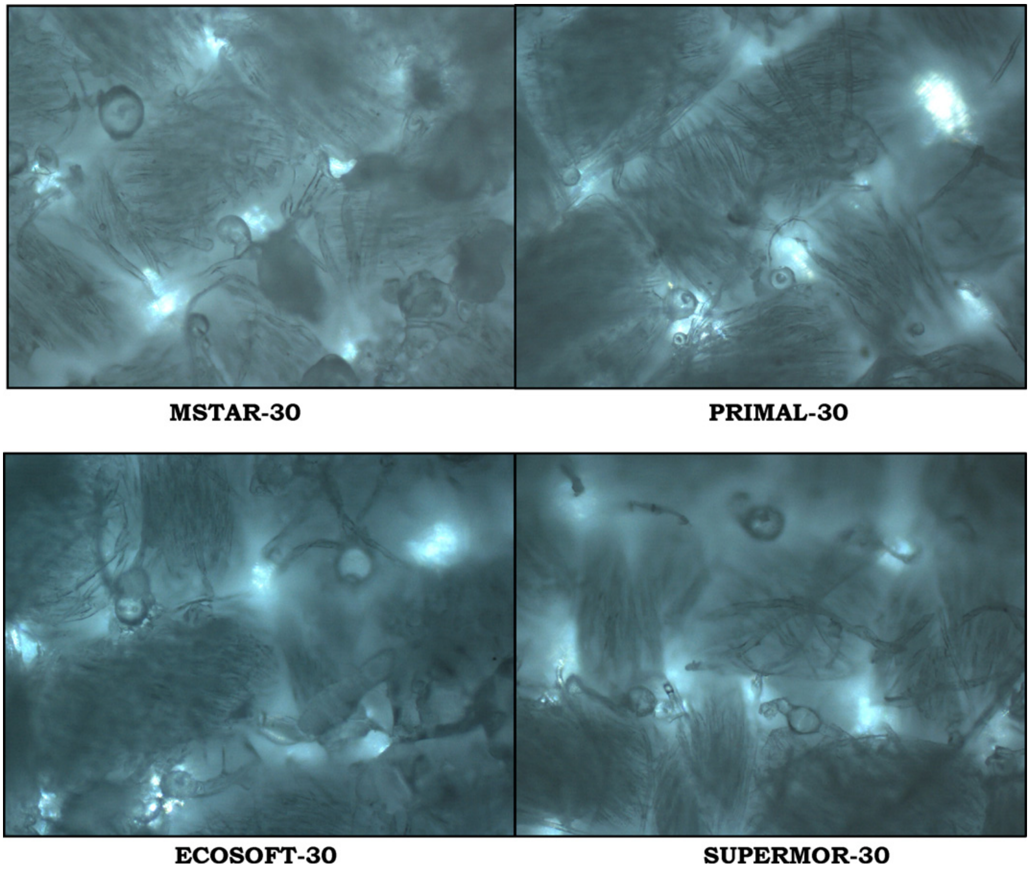

Fig. 1. OM micrographs of thermo-regulating textiles with different commercial binders (10×).

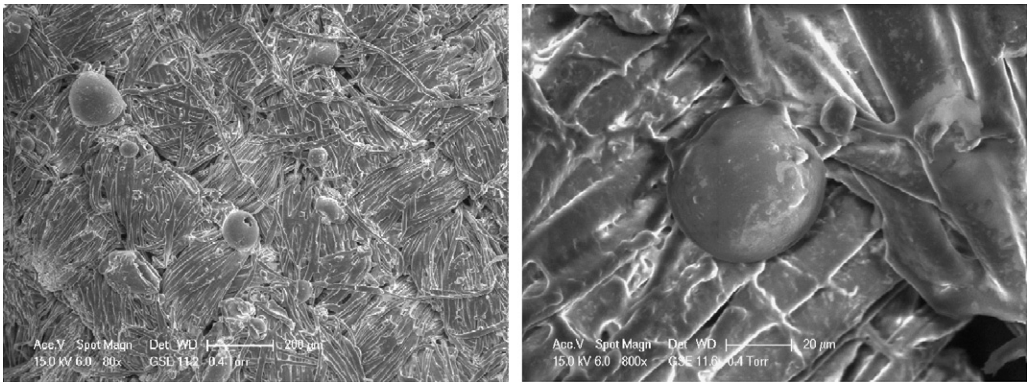

Fig. 2. ESEM micrographs of ECOSOFT-30.

formulation will be more convenient for the microcapsules fixation on the substrate textile.

In order to select the polymeric binder more appropriate for the fixation process different commercial binders were employed. For this purpose, all experiments were carried out with a constant amount of 30 wt.% of PCM microcapsules related to the coating binder.

The cured conditions were fixed at 95 °C for 11 min. The most important characteristics of the coating textiles obtained with different coating formulations for this procedure are reported in Table 2.

It can be seen that all the coating binders allow to obtain thermo-regulating textiles with acceptable thermal storage capacities except CENTER-30. On the other hand, the final appearance and texture of ECOSOFT-30 and SUPERMOR-30 could be considered as the best coating formulations due to their smooth texture and regular physical aspect.

In Fig. 1 optical micrographs of textiles obtained by means of the coating technique using different binders are shown. It can be observed that the textiles produced using MINERSTAR NEUTRO®, PRIMAL E-358-ER EMULSION®, TEXPRINT ECOSOFT N10® and WST SUPERMOR® showed high energy storage capacities and better fixation of PCM microcapsules on the textile substrate. Fig. 2 shows ESEM micrographs of the thermo-regulating fabric obtained using TEXPRINT ECOSOFT N10® binder. As it can be seen, the successful fixation between textile substrate and PCM microcapsules and the microcapsule stability after coating process were verified.

As it was reported in Table 2, the presence of the microcapsules in the textiles was confirmed by DSC analysis. In Fig. 3 DSC curves of the coating fabrics with TEXPRINT ECOSOFT N10® (ECOSOFT) and WST SUPERMOR® (SUPERMOR) binders and the same fabrics with these binders and PCM microcapsules (ECOSOFT-30 and SUPERMOR-30) are shown. Endothermic peaks at 40 °C of 4.8 J g^{-1} and at 40 °C of 5.8 J g^{-1} were obtained for ECOSOFT-30 and SUPERMOR-30, respectively, due to the phase change of fusion of the paraffin wax encapsulated inside the microcapsules added on the textile (as shown in the inset graph).

Finally, the results obtained indicate that TEXPRINT ECOSOFT N10® and WST SUPERMOR® commercial binders allowed an efficient fixation of the PCM microcapsules on the fabric. PCM microcapsules were very stable to the thermal and mechanical treatments used for the fixation into the textile.

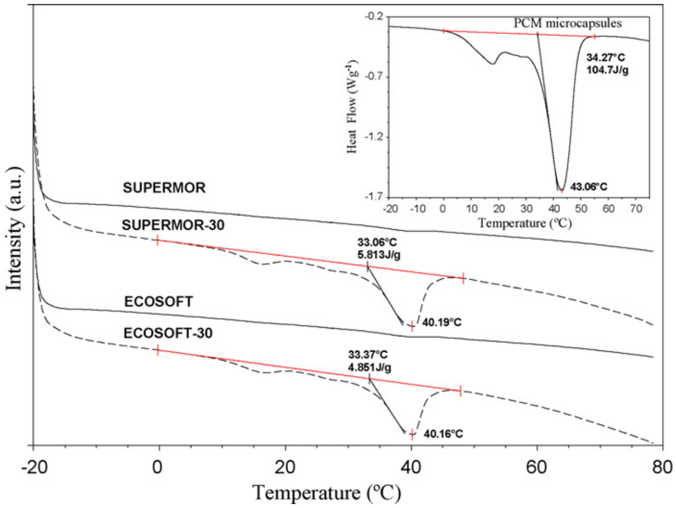

Fig. 3. DSC thermograms of the coating textiles with 30 wt.% of PCM microcapsules and without them with TEXPRINT ECOSOFT N10® and WST SUPERMOR® binders. The inset graph shows DSC thermogram of the paraffin wax microcapsules used.

3.2. *Influence of the amount of microcapsules in the coating formulation*

Thermoregulatory characteristics of the textiles with microcapsules of PRS® paraffin would depend directly on the amount of them into the textile substrate. The influence of the increase of microcapsules amount into the coating formulation was studied in order to obtain PCM fabrics with a high energy storage capacity. The experiments were carried out using a 15, 20, 25, 30, 35, 40 and 45 wt.% of microcapsules with respect to the weight of WST SUPERMOR® and TEXPRINT ECOSOFT N10®, respectively.

Fig. 4 shows the energy storage capacity of the coating textiles with different amount of microcapsules related to the wt.%

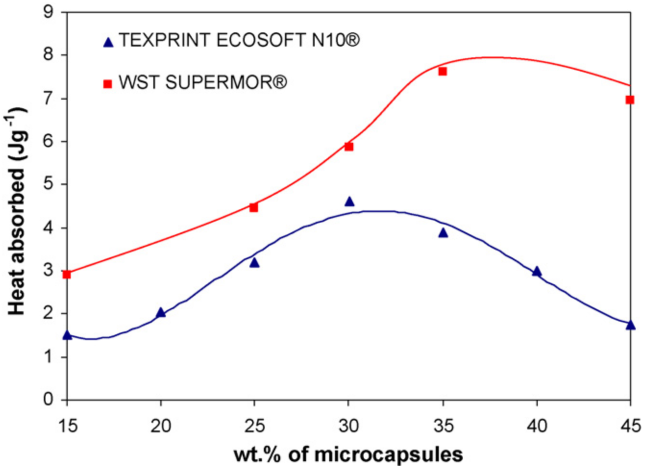

Fig. 4. Heat absorbed of coating textiles with different wt.% of microcapsules added.

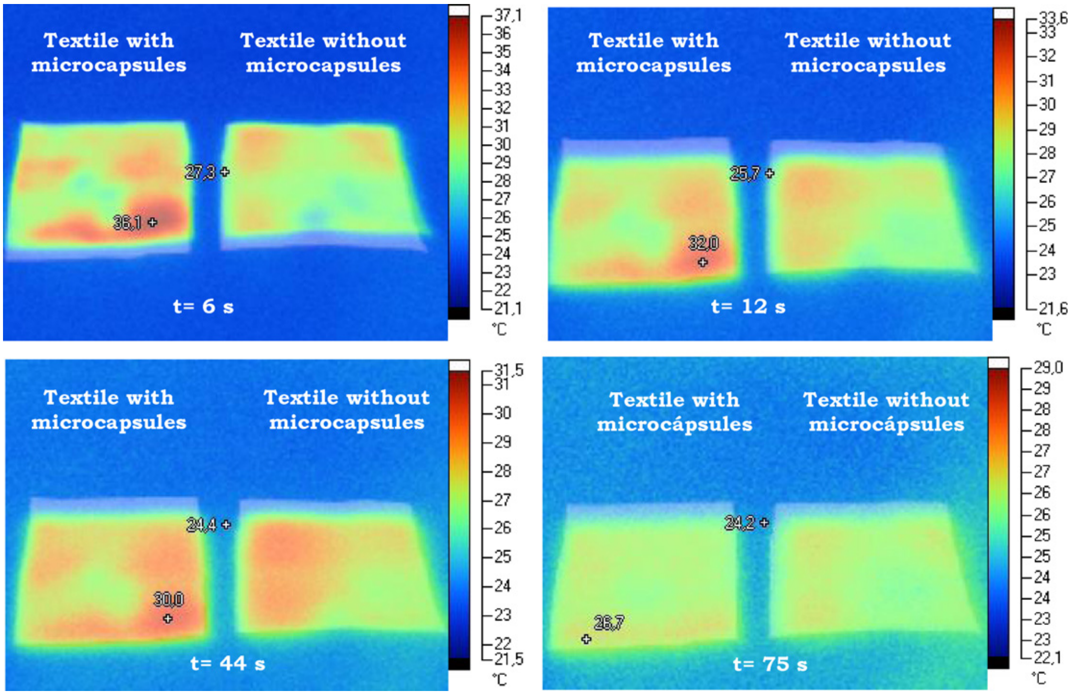

Fig. 5. Comparison of thermal images obtained for SUPERMOR-35 and a normal textile preheated at different times.

of microcapsules added on the coating binder for both polymeric binders. As the amount of PCM microcapsule added into the binder increases, the heat storage capacity of the thermo-regulating fabric increases passing through a maximum and then decreases again for both polymeric binders. These results showed that, with the increase of the amount of microcapsules into polymeric binder until 30–35 wt.%, the proportion of the PCM microcapsule fixed increases. However, when the amount of microcapsules exceeds these values the coating paste become thick and sticky and the blade of the motorized film applicator produces heterogeneous and irregular films. Consequently, the coatings applied contain lower amount of microcapsules.

As can be seen in Fig. 4, a maximum of the heat absorbed for SUPERMOR-35 of 7.6 J g^{-1} was obtained. However, using TEXPRINT ECOSOFT N10® as binder a maximum of 4.7 J g^{-1} was achieved with a 30 wt.% of microcapsules. These results indicate that these polymeric binders allowed the incorporation of a large amount of PCM microcapsules into the fabric. In this sense, Shin et al. [7] attained thermo-regulating textiles with 4.44 J g^{-1} using a 23 wt.% of melamine-formaldehyde microcapsules containing eicosane (34.9 °C of phase change temperature) to incorporate into polyester knit fabrics by a conventional pad–dry–cure process. Therefore, WST SUPERMOR® coating binder would be the best choice to produce an effective fixation process according to the results obtained (76.8 g of PCM microcapsules per m^2 of fabric).

From technical, commercial and clothing industry point of view ECOSOFT-30 and SUPERMOR-35 offer excellent thermal storage capacities without affecting the look and feel of the textile.

3.3. *Study of the thermoregulatory effect of the PCM fabrics*

The efficiency of the active thermal insulation effect of the textiles with incorporated microcapsules has been evaluated using an infrared thermography camera. Intensity and duration of the active thermal insulation effect depend mainly on the heat storage capacity of the PCM microcapsules and their applied quantity. Infrared thermography analyses were carried out to study the thermoregulatory effect of SUPERMOR-35. For that, textiles with PCM microcapsules and without them were heated until a temperature higher than the phase change of the PCM (45 °C) encapsulated. The aim was to compare the buffer effect that thermo-regulating textile provides respect to the plain textile after cooling until environmental temperature by infrared and visible vision camera.

Fig. 5 shows thermal images of the surface temperature distribution of textiles with PCM microcapsules and without them obtained for different times by infrared thermography. The zero time was taken when the textiles were taken out of the heater. Thermal images for SUPERMOR-35 show that this thermo-regulating textile undergoes a cooling effect less marked than the textile without PCM microcapsules. This fact is due to the high amount of heat absorbed by the paraffin wax in the melting process (7.6 J g^{-1}) is released into the surrounding area in a cooling process starting at the PCM crystallization temperature. Thermoregulatory effect observed for SUPERMOR-35 with respect to the normal fabric was 8.8, 6.3, 5.6 and 2.5 °C after 6, 12, 44 and 75 s, respectively. Therefore, infrared thermography analysis confirms the thermoregulatory effect of the coating textile with incorporated microcapsules of PRS® paraffin and the effectiveness of the fixation technique selected.

After comparing the thermal performance of both textiles, the thermoregulatory effect of the paraffin wax microcapsules results a substantial improvement of the thermal comfort.

3.4. *Effect of washing, rubbing and ironing tests on the treated fabric*

The lifetime and the aging of the thermo-regulating textiles obtained were evaluated for the practical use. The selection of the appropriate binder and application method may result in a higher retention of the energy storage capacity of the thermo-regulating fabrics [7]. For this purpose, ECOSOFT-30 and SUPERMOR-35 were selected. Several normalized tests have been applied to reproduce the usage of garments. In particular, washing, rubbing and ironing tests were carried out applying the corresponding international rule.DSC analyses were used to verify the durability and mechanical stability after washing, rubbing and ironing tests due to the

Table 3
Energy storage capacity of the thermo-regulating textiles after washing, rubbing and ironing tests.

	Initial	Washing	Rubbing	Ironing
SUPERMOR-35				
Energy storage capacity ($J\,g^{-1}$)	7.6	3.6	4.8	5.9
ECOSOFT-30				
Energy storage capacity ($J\,g^{-1}$)	4.7	0.6	3.4	2.8

heat storage capacity of the fabric depends on the presence of PCM microcapsules (Table 3).

As can be seen in Table 3, after durability tests the thermo-regulating textiles undergo a decrease of microcapsules content. The loss of microcapsules was more prominent after washing treatments for both fabrics. Furthermore, a decrease of the thermoregulatory capacity of 80% for ECOSOFT-30 was observed. Therefore, mild washing conditions would be helpful for better maintenance of PCM microcapsules [7].

On the other hand, after rubbing and ironing tests a lower deterioration of the microcapsules was obtained, maintaining 75% of the initial energy storage capacity. These results are higher than the results found in the literature [23]. In addition, the textiles with microcapsules fixated with WST SUPERMOR® binder have more resistance to durability tests.

To sump up, SUPERMOR-35 showed a low alteration of the thermoregulatory properties of fabrics after washing, rubbing and ironing treatments.

4. Conclusion

The fixation of PCM microcapsules into textile substrate by means coating technique offers successfully results on the production of thermo-regulating textiles. ESEM micrographs and OM analysis have shown good adhesion between microcapsules and textile substrate, and also confirmed the integrity of microcapsules into the textile. TEXPRINT ECOSOFT N10® and WST SUPERMOR® commercial binders allowed an efficient fixation of the PCM microcapsules on the fabric. The final appearance and texture of both binders could be considered as the best coating formulations due to their smooth texture and regular physical aspect.

The coating fabric with 35% of microcapsules related to coating formulation (WST SUPERMOR®) is capable of absorbing 7.6 $J\,g^{-1}$ of heat if the microcapsules on the fabric undergo a melting process without altering the initial properties of the fabrics.

The WST SUPERMOR® binder showed the fixation of a greater amount of microcapsules into the textile substrate (76.8 $g\,m^{-2}$) coating the textile with 35 wt.% of microcapsules respect to the coating formulation.

A thermoregulatory effect of 8.8, 6.3, 5.6 and 2.5 °C after 6, 12, 44 and 75 s, respectively, was observed by means of infrared thermography.

Textiles with microcapsules fixated with WST SUPERMOR® binder had more resistance to washing, rubbing and ironing treatments.

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