Preparation of Coated Thermo-Regulating Textiles Using Rubitherm-RT31 Microcapsules

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ABSTRACT: Spherical microcapsules with a 49 wt % of Rubitherm® RT31 were successfully synthesized by means of suspension-like polymerization to be used for textile applications in summer conditions. Microcapsules were fixed into seven fabric substrates for different textile applications by a coating technique without deteriorating original functionalities of the textiles. Thermal performance of different coated textiles with 35 wt % of microcapsules was evaluated by differential scanning calorimetry (DSC) and infrared thermography (IR) techniques and the physical characteristics of textiles with thermo-regulating properties were examined by environmental scanning electron microscopy (ESEM). It was observed that all treated textile substrates allow to obtain thermo-regulating properties with acceptable latent heat storage capacities. Results also indicated that the presence of microcapsules containing Rubitherm® RT31 produces a significant thermal insulation effect during a cold to warm transition (20–45°C). Thus, this kind of microcapsules can be used to obtain textiles with thermal comfort-related properties. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 4809–4818, 2012

Key words: thermo-regulating properties; coating technique; microcapsules; phase change materials

INTRODUCTION

Nowadays, textile industry shows a growing interest into the functionalization of textiles for innovative commercial applications. The emerging technologies based on microencapsulation are able to confer new properties and add value to the textiles that are not possible or cost-effective using other technologies.[1–3]

Microencapsulation has become an active field in textiles as patent applications strongly outnumber the published scientific papers. In textiles, the major interest in microencapsulation is currently in the application of durable fragrances and skin softeners, insect repellents, dyes, vitamins, antimicrobial agents, phase-change materials, and medical applications such as antibiotics, hormones, and other drugs.[4,5]

Microencapsulated phase change materials (PCM) can be incorporated into textile structures to produce fabrics with thermo-regulating properties. A thermo-regulating fabric is a smart material that has the property of offering suitable response to changes in external temperature 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.[6]

Well-known PCM are linear chain hydrocarbons known as paraffin waxes (or *n*-alkanes), hydrated salts, polyethylene glycols (PEGs), fatty acids and mixture or eutectics of organic and nonorganic compounds. PCM materials absorb latent heat as a phase change from solid to liquid takes place during a heating process and release latent heat as a phase change takes place from liquid to solid, during a reverse cooling process.[7]

To apply these PCM microcapsules to fabrics, their thermal activity must correspond with the skin temperature and be harmless to the skin. When the human body is in a neutral thermal stage the single body parts are at certain temperatures. The core of the body, or the abdominal area, and the head normally maintain an average skin temperature higher than that associated with other areas of the body. Generally, the overall average comfortable skin temperature is 33.3°C, and if this cannot be maintained, a person begins to feel uncomfortable.[8]

In this study, the commercial paraffin Rubitherm® RT31 was used as PCM for developing a fabric with thermo-regulating properties. Its melting point is about 31°C, and it allows the PCM to be stabilized in a slushy state below the comfortable skin temperature.

In addition, thermal properties, air permeability, moisture vapor permeability, and moisture regain of

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materials also influence the heat balance of the body and, consequently, affect clothing comfort.[9] The incorporation of PCM microcapsules to textiles can affect other comfort-related properties and hand of the materials adversely, 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.[10]

Several methods of incorporating PCM microcapsules into a fibrous structure have been developed. The microcapsules can be applied by stamping works, exhaustion dyeing, impregnation, spraying and coating or by direct incorporation in the fiber without highly modifying its touch and color.[3,11,12] In previous applications of PCM technology in the textile industry, for garments and home furnishing products, microencapsulated PCM were incorporated into acrylic fibers[6] or polyurethane foams[13] or were embedded into a coating compound and topically applied to a fabric.[14] Shin et al.[10] incorporated melamine-formaldehyde microcapsules containing eicosane on polyester knit fabrics by means of a pad dry cure method with a polyurethane binder. Mengjin et al.[15] developed a new kind of thermoregulating fiber based on PVA and paraffin. Furthermore, Onder et al.[16] studied the microencapsulation of three types of paraffin waxes by complex coacervation to improve thermal performances of woven fabrics. Recently, Koo et al.[17] have attempted to demonstrate the application of PCM microcapsules on waterproof nylon fabrics and to enhance thermal insulation effect with ceramic materials (SiC) by using a dual coating method.

Binders play a crucial role in microcapsule coating formulation for various textile materials, as they are required to fix microcapsules on textile supports permanently. To a large extend, binders determine the quality, durability and washability of textile materials with microencapsulated ingredients. Some of the most frequently used binders in textile are water-soluble polymers, such as starch and modified starches, carboxymethyl cellulose; synthetic latexes, such as styrene-butadiene, polyvinylacetate or acrylate latexes; and aminoaldehyde resins.[18]

In our previous work, the fixation of PCM microcapsules containing paraffin with a melting point around 40°C, into a cotton textile substrate by means of a coating technique were carried out. Furthermore, the influence of different coating formulations and mass ratio of microcapsules to coating formulation were evaluated in order to obtain adequate textiles with thermo-regulating properties.[19] The coating fabric with 35 wt % of microcapsules added related to commercial coated binder (WST SUPERMOR®) showed a latent heat 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 textiles with thermo-regulating properties in comparison with a coated one without PCM microcapsules. The different application areas of textiles with thermo-regulating properties imply the fixation to very different substrates. In this sense, there are few references in the literature studying the influence of the kind of textile on the fixation of microcapsules.[17] In addition, the PCM microcapsules incorporation could degrade the original functionalities of the textile such as soft touch, vapor or moisture permeability and wearing comfort.

The aim of this work was to investigate the production of microcapsules containing Rubitherm® RT31 paraffin in order to prepare textiles with thermo-regulating properties, on the basis of experimental conditions optimized in the previous work, using different types of textile substrates depending on the field of their textile applications (apparel, blankets, insulation, protective clothing). Furthermore, a comparison was made of the thermal insulating effect of the textiles with thermo-regulating properties according to the used textile substrate.

EXPERIMENTAL

Microcapsules synthesis

Styrene (99 wt %) of reagent grade (Merck Chemical) previously purified by washing with sodium hydroxide and dried with calcium chloride was used as the monomer. Benzoyl peroxide (97 wt %) was used as initiator (Fluka Chemical). A commercial grade Rubitherm® RT31 paraffin wax (M_w 268 g mol^{-1}) was used as core material. Polyvinylpyrrolidone (K30, M_w 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. Water was purified by distillation followed by deionization using ion-exchange resins. Nitrogen was of high-purity grade (99.999%).

A suspension-like polymerization process was used for the microcapsules synthesis. A tubular type Shirasu porous glass membrane was used for a better control of microparticle size. Details of the synthesis process were previously described elsewhere.[20]

Preparation of textiles with thermo-regulating properties

Microcapsules were fixed into seven fabrics by means of a coating technique, using a motorized film applicator from Elcometer model 4340 according with ASTM D-823.[21] WST SUPERMOR® (supplied by

TABLE I
Textile Substrates Characterization and Their Textile Uses

Sample	Composition	Area weight ($g\ m^{-2}$)	Thickness (mm)	Uses
A	82% Polyester 18% Polyurethane	296	1.50	Soft-Shell fabric with an intermediate polyurethane membrane for cold protection
B	11% Elastane; 35% Polyamide; 54% Polyester	270	1.34	Soft-Shell fabric with an intermediate polyurethane membrane for cold protection
C	100% Polyamide	202	0.48	Green fabric for military uses
D	100% Polyester coating with 100% PVC	121	0.27	Yellow fabric for garments of high visibility
E	40% Polyester; 60% Cotton	185	0.35	Openwork fabric in blue tone for medical garments uses
F	100% Polyamide	328	0.61	Military printed fabric for military uses
G	100% Cotton	158	0.30	Fabric used for upholstery, sheets, curtains and garments

Minerva Color) were used as commercial coating binder. In a previous study,[19] this binder was selected in order to allow an efficient fixation of the PCM microcapsules on the fabrics. Seven fabrics substrates for different textile applications were used. Their description and properties are shown in Table I. Samples were named A to G, according to the textile substrates employed. Every sample had 200 mm of wide and 290 mm of length due to requirements of the motorized film applicator.

The coated formulation consisted of WST SUPERMOR® commercial binder and Rubitherm® RT31 microcapsules (35 wt % of the coating mixture).

The textile substrate was set on the motorized film applicator surface assuring the fabric with clips. In this study, the thickness selection of the coating layer was 0.1 mm to obtain a high thermal storage. 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 coated fabric was cured at 95°C for 11 min.

Characterization

Differential scanning calorimetry

Measurements of melting point and latent heat storage capacities of different materials 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 carried out in the temperature range from −30°C to 80°C with heating and cooling rate of 10°C min^{-1}.

Various samples of each experiment were analyzed at least three times and the average value was recorded. Differential scanning calorimetry (DSC) analyses of coating textiles from random areas were carried out.

Furthermore, the encapsulation ratio of Rubitherm® RT31 in the microcapsule was calculated with the following equation based on enthalpy values:

$$\%\ \text{PCM content by weight} = (\Delta H_m/\Delta H_{pcm}) \times 100\% \qquad (1)$$

where ΔH_m is the enthalpy for the analyzed microcapsules ($J\ g^{-1}$) and ΔH_{pcm} is the enthalpy of pure Rubitherm® RT31 (154.3 $J\ g^{-1}$).

To determine the thermal stability of the reversible phenomena of phase change, the coated textiles were subjected to repeated cycles of melting and crystallization (three cycles).

Environmental scanning electron microscopy

Environmental scanning electron microscopy (ESEM) was used to analyze the morphological structure of the microcapsules and the fixation and integrity of PCM microcapsules into the coated textile substrates. Textile samples were observed by using XL30 (LFD) ESEM with a wolfram filament operating at a working potential of 20 kV.

Calculation of number-average diameter and volume-average diameter

Particle size and particle size distribution (PSD) of the PCM microcapsules were determined on a Malvern Mastersizer Hydro 2000 SM light scattering apparatus with dilute dispersions of the particles in methanol.

Infrared thermography

The temperature distributions of the coated textiles with thermo-regulating properties were evaluated by means of an infrared and visible camera Fluke Ti25. This dispositive allows to obtain thermal and visual images in the temperature range from −20°C to 250°C with a precision of ±2°C. The screen was

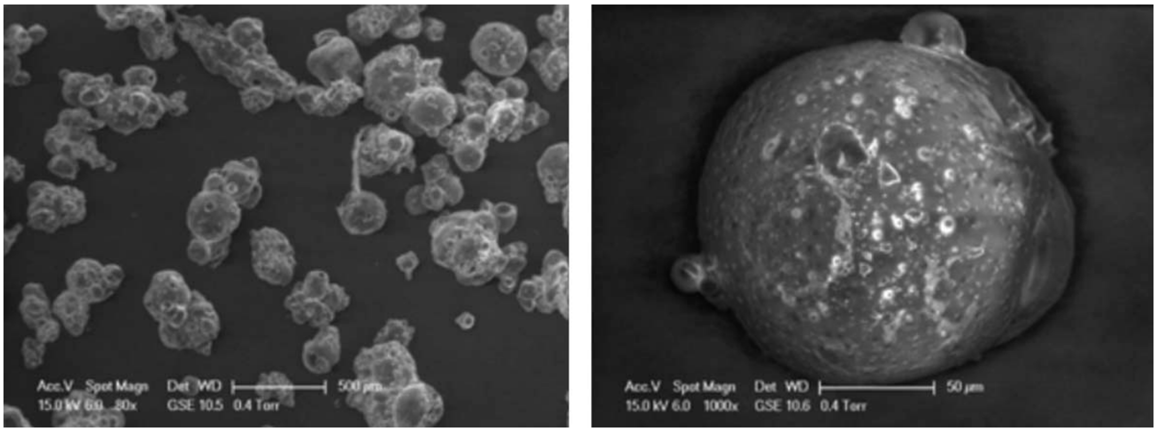

Figure 1 ESEM micrographs of microcapsules containing Rubitherm® RT31.

observed from a distance of 30 cm at 24°C. Images of textiles were taken at different times from 60°C to room temperature. Fluke SmartView™ software was used for downloaded of images.

Thermal human comfort in summer conditions was tested, recording images from 25°C to outside temperature (35°C), comparing a reference textile with a prototype textile with thermo-regulating properties in contact with the body (shoulders in this specific case).

Thermoregulating test

There is a no standard method for measuring the heat absorption effect of the textiles. An experimental equipment was designed to test the thermal performance of textiles with thermo-regulating properties. The experimental set up consists of a hollow metallic box of aluminium, through which is constantly flowing demineralized water by means of a peristaltic pump from a thermostatic bath at the desired temperature. This water allowed to fix the temperature on the aluminium cell. Dimensions of the aluminium cell were $10 \times 6 \times 3$ cm^3 and a thickness of walls of 0.5 cm. The cell was incorporated in an internal diffuser plate to improve the liquid distribution avoiding the formation of preferential flow paths. The liquid flow was high enough to ensure the same temperature on the whole aluminium plate surface, regardless of the flow direction. Details of the test apparatus were previously described elsewhere.[22]

To minimize the experimental error of this apparatus, four layers of textile substrate with 1.5 mm of thickness were used. The sample was placed on the upper surface of the aluminium cell and further insulated with cork boards of 2 cm in thickness. Six thermocouples of K-type were used to measure

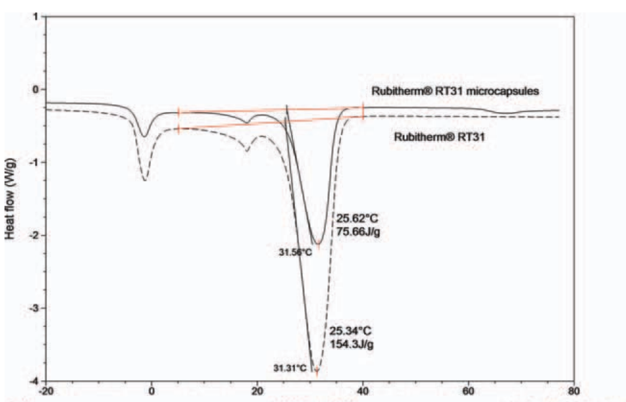

Figure 2 DSC thermograms of microcapsules containing Rubitherm® RT31 and the pure Rubitherm® RT31. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

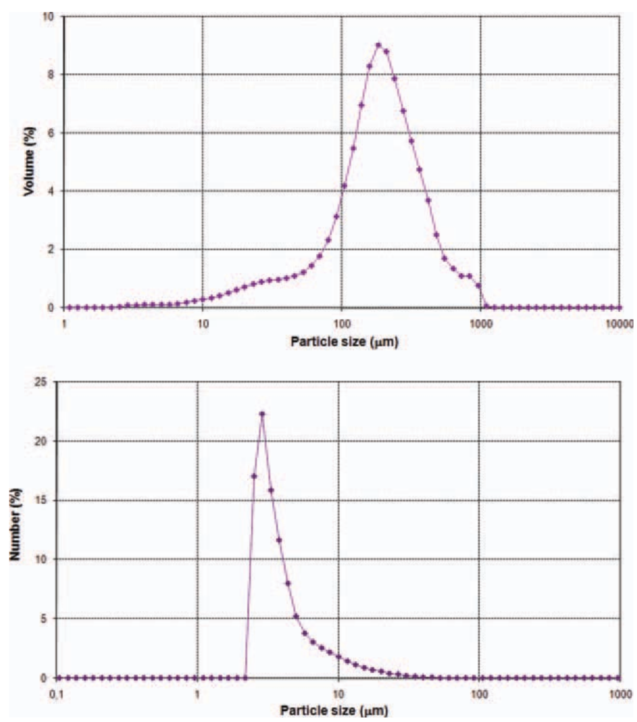

Figure 3 Particle size distribution for microcapsules containing Rubitherm® RT31: (a) in volume and (b) in number. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

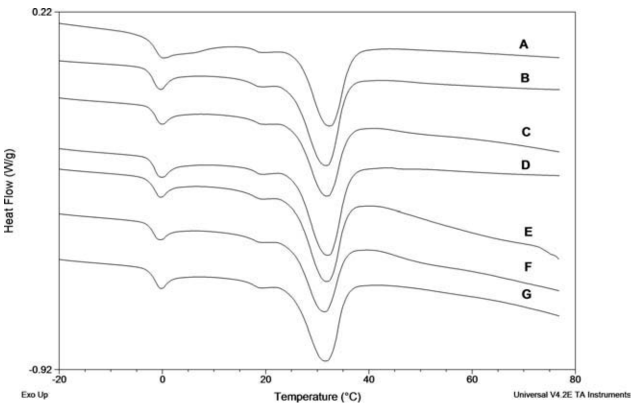

Figure 4 DSC thermograms of the different coated textiles with 35 wt % of microcapsules containing Rubitherm® RT31.

temperatures: two were put on the external surface of the textile, other two on the surface of the aluminium cell, one more on the insulating cork and the other were used to measure the room temperature. All these signals were registered continuously using the NOKEVAL program and recorded by means of a computer. The thermal absorption study of the textile was carried out by changing the thermostatic bath set-point from 20 to 45 ± 0.1°C.

RESULTS AND DISCUSSION

Characterization of Rubitherm® RT31 microcapsules

In Figure 1, the PCM microcapsules morphologies determined by environmental scanning electron microscopy (ESEM) are shown. It can be seen that the PCM microcapsules were successfully synthesized by means of suspension like polymerization using Rubitherm® RT31 as core. Most of the microcapsules obtained had a spherical shape with a smooth surface and a relatively homogeneous external appearance without broken particles or incomplete spheres.

The thermal properties of Rubitherm® RT31 microcapsules were evaluated using DSC analysis (Fig. 2). The melting phase change temperature of obtained PCM microcapsules was 31.56°C, slightly higher than the exhibited by the pure core material, Rubitherm® RT31 (31.31°C). This fact can be correlated to the low thermal conductivity of the shell material, which affects the latent heat transfer rate from the outside to the PCM inside the polystyrene shell, and therefore influenced the phase change temperature of the microencapsuled PCM.[23] The heat storage capacity of the microcapsules containing Rubitherm® RT31 was 75.66 J g^{-1}. This value is quite similar to that obtained previously when PRS® paraffin was used, 104.70 J g^{-1},[20] and represents a percentage of 49 wt % of Rubitherm® RT31 encapsulated. Therefore, the high encapsulation efficiency and the melting temperature range of these PCM microcapsules are quite satisfactory to be used in thermal comfort requirements in textile field.

The Figure 3 shows the particle size distribution of the microcapsules synthesized. It can be observed that the particle size distribution seems to be quite uniform. The average particle sizes in volume and in number are 203.9 μm and 4.1 μm, respectively. This particle size is small enough in order to apply the microcapsules into the textiles without a significant loss on the properties of the untreated textile.[24,25]

Coated thermo-regulating textiles

DSC measurements

Thermal performance of different coated textiles with thermo-regulating properties with 35 wt % of PCM microcapsules as a function of the kind of textile substrate was evaluated by DSC analyses (Fig. 4). It can be seen that all treated textile substrates allows to obtain thermo-regulating properties with acceptable latent heat storage capacities. The same result was observed by other authors.[23,26] On the other hand, no significant differences on the phase change transition temperature of coated samples and the Rubitherm® RT31 microcapsules were

TABLE II
Thermal Properties of the Thermo-Regulating Textiles

Sample	ΔH (J g^{-1})	Duration of the heat release from 33 to 25°C (s)	Latent heat accumulated in 1 m^2 of fabric substrate (kJ m^{-2})	PCM microcapsules added on the textile (wt %)
A	19.4	79	5.7	25.6
B	18.1	62	4.9	23.9
C	13.5	65	2.7	17.8
D	16.3	28	4.4	21.5
E	14.3	42	2.6	18.9
F	11.1	67	3.6	14.7
G	14.4	48	2.3	19.0

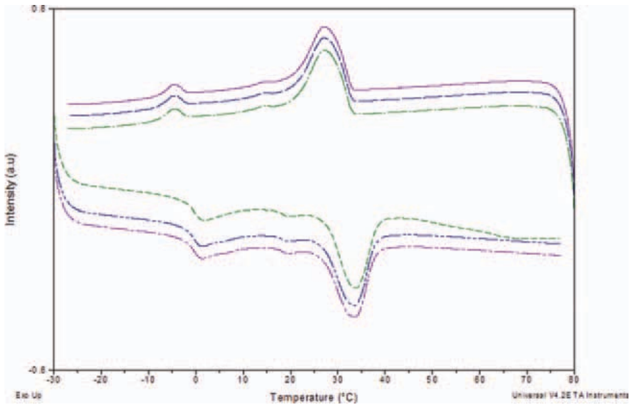

Figure 5 DSC curves of a textile with thermo-regulating properties (Sample A), triple scan. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

observed. It was found that the melting transition points in the coated fabrics from A to G changed 0.72, 0.01, 0.13, 0.37, 0.13, −0.35, and 0.01°C, respectively. This indicated that the kind of textile substrate have not significant effect on the microcapsules melting effect. In this sense, Koo et al.[17] observed changes up to 0.58°C by using a wet coating method.

In our previous paper, textile substrate G (100% cotton) was used to obtain the textiles with thermoregulating properties.[19] In the present work the employed experimental conditions were the same except for the thickness of the coating layer. A thermal storage capacity of 14.4 J g^{-1} was achieved using cotton textile substrate and a coating thickness of 0.1 mm. Comparing this result with the value of 7.6 J g^{-1} obtained in the previous work with 0.01 mm of thickness, it is observed that the latent heat storage capacity of the treated fabric increased as the thickness of the coating layer increased.

Table II summarizes the latent heat storage capacity, the necessary time to decrease the temperature of coated textiles from 33 to 25°C, the latent heat accumulated in 1 m^2 of fabric substrate associated with each sample and the amount of PCM microcapsules added on each textile substrate. There are not important differences in the latent heat storage capacity and the amount of retained PCM microcapsules depending on the kind of used substrate textile. Coated textiles A and B exhibit the highest latent heat and a long thermoregulatory effect, due to the soft shell characteristics and the large thickness of these fabrics that allow to accommodate a high amount of PCM microcapsules and improve the resistance of heat transfer, respectively. Sample D with 16.3 J g^{-1} shows a short time of the heat release, this may be related to the small thickness of the used textile substrate (0.27 mm). For samples E and G the latent heat storage capacity, the amount of PCM microcapsules added on the textiles and the thickness of these textiles are similar, thus the duration of the thermal buffer effect was quite similar. Furthermore, the lowest latent heat storage capacity for samples having polyamide as textile substrate (Samples F and C) suggests that this composition does not allow the incorporation of a large amount of PCM microcapsules into the fabric. Nevertheless, small differences of latent heat storage capacity were obtained. According to these results, the heat transfer through fabric depends on the quantity of PCM microcapsules added on the coating binder but also the textile composition and structure. Consequently, numerous factors must be considered in the evaluation of the heat transfer properties of fabrics.[17]

Reliability tests to study the thermal performance of a textile with thermo-regulating properties (Sample A) during thermal cycling were carried out using DSC analysis (Fig. 5). It can be observed that the latent heat storage of the sample does not change when heating/cooling cycle is repeated (less than 2% of latent heat variation). Furthermore, melting and freezing transition points of the coated fabrics

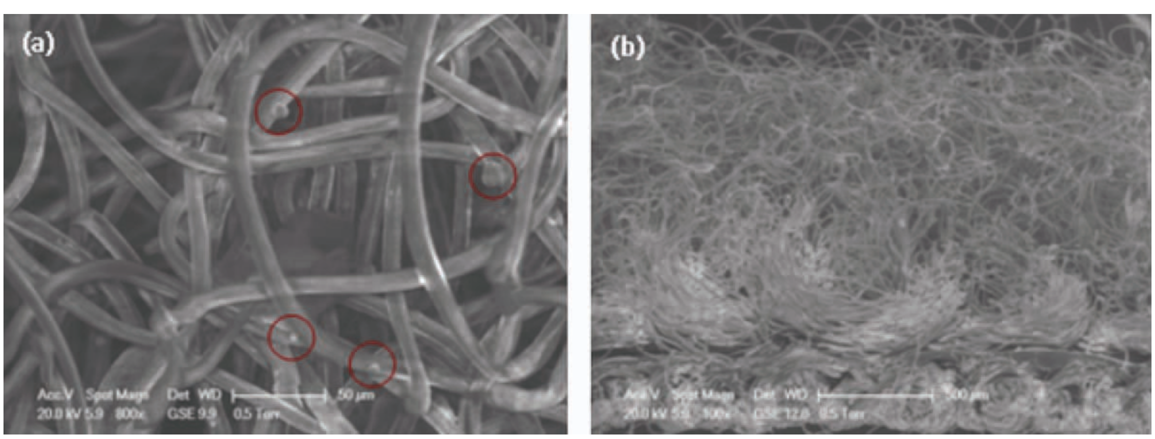

Figure 6 ESEM micrographs of a thermo-regulating textile (Sample A): (a) surface and (b) cross section. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

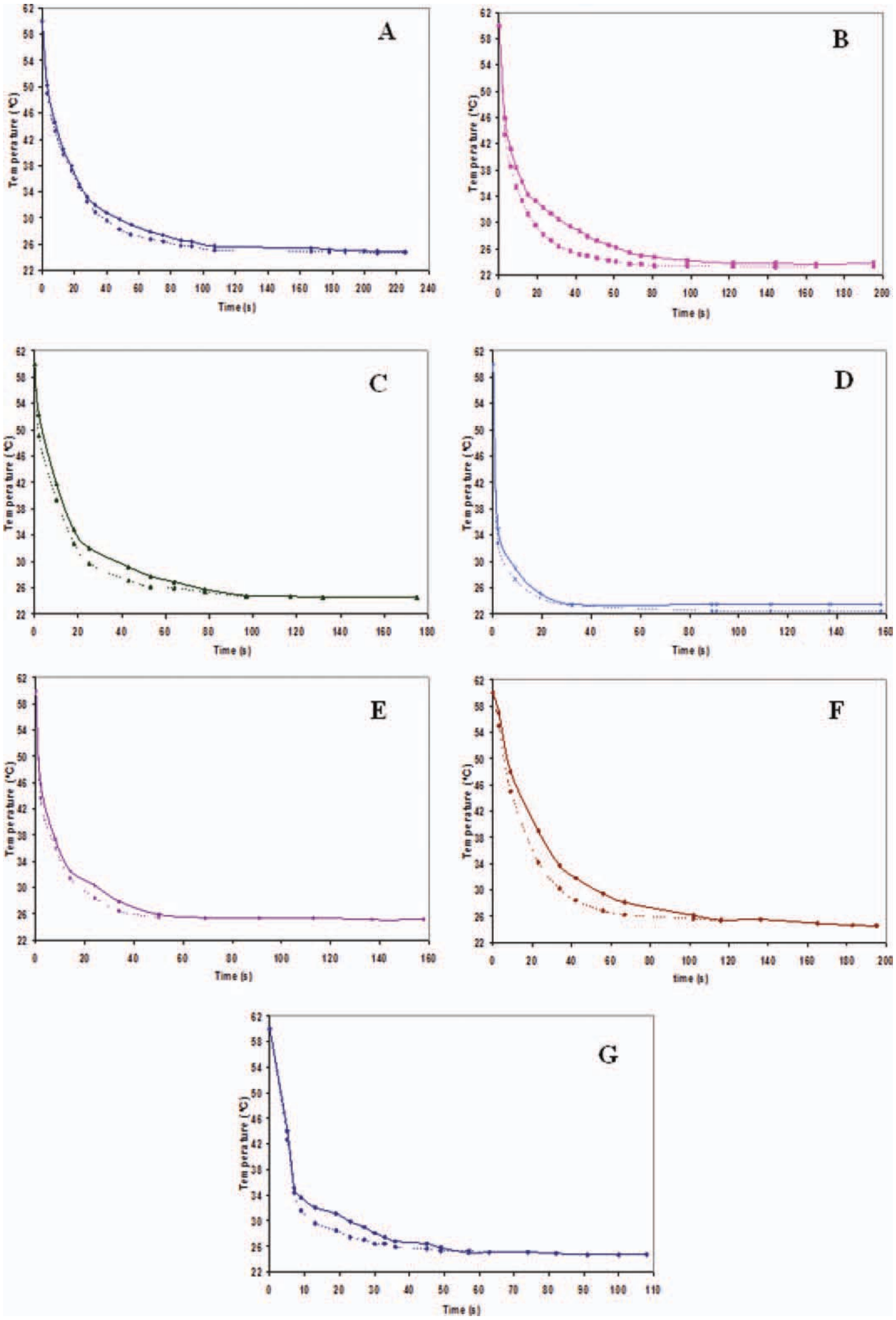

Figure 7 Temperature distribution as a function of time for different coated textiles by thermal vision camera: ----- Textile without PCM microcapsules Textile with PCM microcapsules. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

shift to higher temperature than microcapsules containing Rubitherm® RT31. This fact could be attributed to the influence of the polymeric binder on the thermal response of textile.[23]

Therefore, these results demonstrated that textile substrates with different characteristics and applications are suitable for application of the microcapsules by means of a coating method. Similar results of latent heat storage capacity using natural leather and 40 wt % of PCM microcapsules were obtained by Izzo Renxi et al.[26]

ESEM observations

Figure 6 shows ESEM micrographs of the surface and the cross-sectional morphology of a representatively sample (coated textile A) in which PCM microcapsules were used as the thermal insulation material. It can be seen, the successful fixation between textile substrate and the microcapsules containing Rubitherm® RT31. The PCM microcapsules in the treated samples were mainly located at the spaces between fibers and the fiber surface. As shown in Figure 6(b), the textile substrate A is remarkable thick which leads to a large amount of PCM microcapsules added on to the textile substrate according to DSC analysis obtained.

Evaluation of thermo-regulating properties

During the last years several testing methods have been developed for measuring the temperature-regulating ability of PCM in fabrics.[14–17] Onder et al.[16] evaluated the active thermal insulation effect of the textiles with incorporated microcapsules by means of a thermal vision camera. Figure 7 shows a comparison of the maxima temperature reached on the surface temperature distribution for the seven samples with the time. In this study, the coated textiles with and without PCM microcapsules were cooled from 60°C to room temperature. In all the cases, Rubitherm® RT31 buffer effect is observed. Therefore, during the cooling process there is a temperature range when the temperature of coated textile with PCM microcapsules is higher than noncoated one. This indicates that the stored energy of PCM is transferred to the environment in the phase change range during the reverse cooling process.[23] As it was previously mentioned, coated textile D exhibits the highest rate of temperature decrease and Samples A and B have a significant effect on the thermal insulation.

Furthermore, tests of the thermoregulating response of coated fabric containing PCM microcapsules in the designed experimental set-up, described on *Thermoregulating test* section, were carried out during a cold to warm transition (20–45°C). The difference between inlet and outlet heat flux of the coated textile with PCM microcapsules (coated Sample A) and the reference (coated Sample A without PCM microcapsules) are graphed in Figure 8. There is a significant difference of heat flux change between the reference and Sample A due to the presence of microcapsules containing Rubitherm® RT31. As the phase change materials absorb or release heat during the processes of phase change,

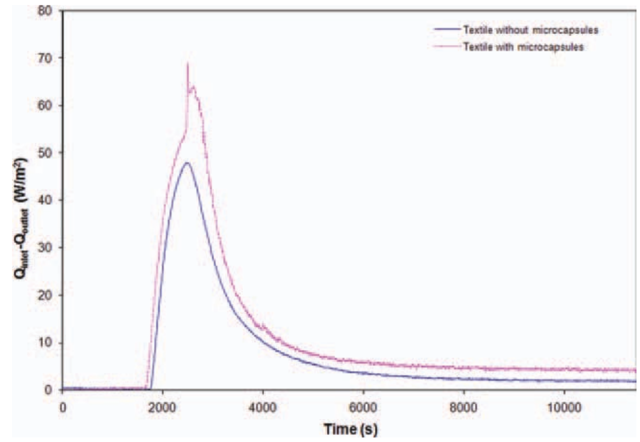

Figure 8 Comparison of heat flux change of coated textile A with PCM microcapsules and without them. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the heat flux change for the coated fabric is influenced by the PCM microcapsules containing in the textile. This behavior was also observed by other authors.[17,26,27]

With the aim to test the thermal comfort in summer conditions, the effect of a reference textile and a prototype of a textile with thermo-regulating properties in contact with the body (shoulders in this specific case) was visualized by IR thermography images (Fig. 9). Images when the individual went out to the building, from 25°C (inside temperature) to 35°C (outside temperature), were taken. Thermal imagines show that textiles with thermo-regulating properties undergoes a heating effect less marked than the textile without PCM microcapsules, as a consequence of the melting process of encapsulated Rubitherm® RT31. Thermoregulatory effect observed for textile with PCM microcapsules with respect to the normal fabric was 1, 0.9, 0.8, and 0.7°C after 10, 30, 45, and 75 s.

Concluding, the thermal performance of textiles with thermo-regulating properties depends on the latent heat storage capacity, PCM microcapsules content, textile substrate structure, and the correspondence between the phased change temperature and the application temperature range. Consequently, thermal effects can be improved according to the final application request using an appropriate combination of all these parameters.

CONCLUSIONS

Polystyrene microcapsules containing Rubitherm® RT31 with a latent heat storage capacity of 98.72 J g^{-1} and an average particle size in number of 4.1 μm were produced by suspension like polymerization

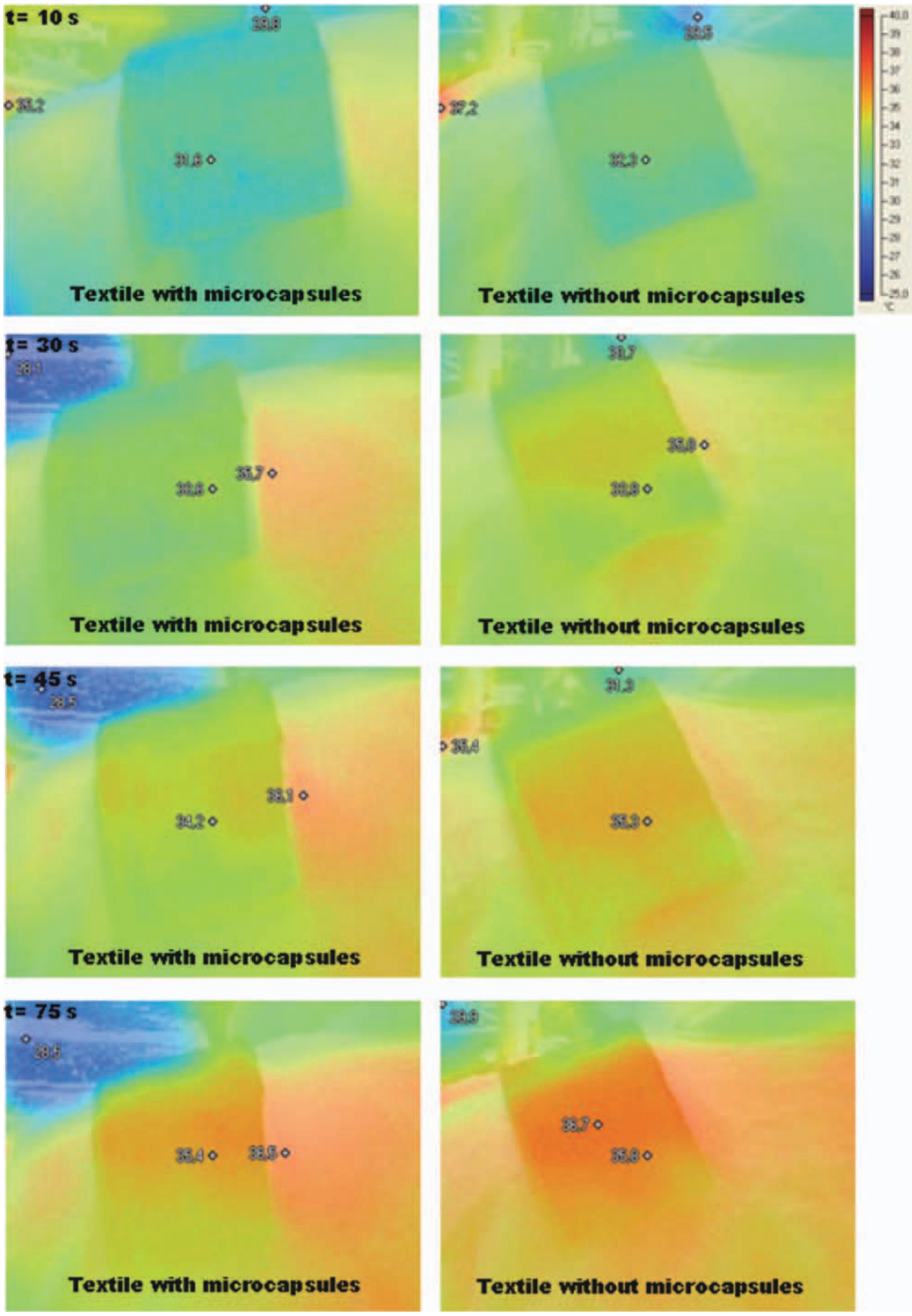

Figure 9 Infrared camera images of coated textile A with and without PCM microcapsules at different times. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

technique. ESEM micrographs analyses have shown that most of the microcapsules had a spherical shape with a smooth surface. Seven types of textile substrates were used in order to prepare textiles with thermo-regulating properties by means of a coating technique.

Fabrics with thermo-regulating properties had latent heat storage capacities of 11.1–19.4 J g^{-1}, depending on the textile substrate. The DSC and the thermal temperature distribution analyses indicated that all the coated textiles with 35 wt % of PCM microcapsules showed high latent heat storage capacities and significant thermoregulatory effects. Furthermore, DSC curves demonstrated that the latent heat storage of the textiles with thermo-regulating properties does not change when heating/cooling cycle was repeated. A thermoregulatory effect in contact with the body of 0.9°C after 45 s was observed by means of IR thermography images.

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