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Examination of the Coating Method in Transferring Phase-Changing Materials

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ABSTRACT

In this study, it is intended to identify the characteristics of heat regulation in heat storage microencapsulated fabrics and it is aimed to examine the effect of application method for microcapsules. For this purpose, phase-changing materials (PCM) microcapsules were applied according to the method of impregnation and coating on cotton fabrics. The presence and distribution of microcapsules on the fabric surface were investigated by scanning electron microscopy (SEM). The temperature regulation of the fabrics was examined utilizing the temperature measurement sensor and the data recorder system (Thermal camera). According to the DSC analysis, the melting process in fabrics coated with Mikrathermic P microcapsules occurred between 25.83°C - 31.04°C and the amount of heat energy stored by the cotton fabric during the melting period was measured as 2.70 J/g. Changes in fabric surface temperature due to the presence of microcapsules in the fabric structure were determined in the measurements. When comparing the transfer methods of PCM capsules, the contact angle of impregnated and coated fabric was obtained as 42° and 73°, respectively. As a result of the study, when the analysis results of the microcapsules transferred to the fabric by the impregnation and coating method are evaluated, it is seen that the PCM transferred fabric with the impregnation method performs more efficient temperature regulation. However, the analysis results show that fabrics transferred with PCM by coating also perform heat absorption, although not as much as the impregnation method. Performance evaluation according to the target properties of

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textile material will give the most accurate result for the fabrics which are treated by coating and impregnation method.

Keywords: Phase change material, encapsulation, impregnation, coating, thermal camera

1. INTRODUCTION

The importance of functional processes that add value, create difference and increase market share in the textile sector is increasing day by day with the developing technology. Not only aesthetic features but also functional features determine consumers' wishes. For this purpose, different technologies can be to provide different functional properties to textile materials like plasma, sol-gel or microencapsulation (1).

The microencapsulation process produces small spheres covered with a thin shell film to protect the active substance from outside. Using this technology, it is possible to protect easily perishable substances such as drugs, insecticides, antibacterials, and antioxidants from environmental factors like heat, light, and oxygen. In addition, the wearer is exposed to much lower doses of these substances. Using microcapsules in textile finishing, it is possible to produce resistant-to-wash textile products that are effective even when a less active substance is used. Another area where microcapsules can be used is energy storage (2-6).

In our world, we have so many problems like climate crisis, greenhouse gas emissions, air pollution, usage of finite resources, economic reasons, etc. So we need energy for heating, air conditioning and ventilation. Energy storage plays important role in conserving available energy and improving its utilization since many energy sources are intermittent. Short-term storage of only a few hours is essential in most applications like clothes or curtains, moreover, long-term storage of a few months may be required in some applications like buildings, concrete or space clothes (7-9).

Phase change material or PCM can store and release large amounts of energy. This energy is called latent heat. Latent heat is thermal energy released or absorbed, by a thermodynamic system, during a constant-temperature process — usually a first-order phase transition. Latent heat can be understood as heat energy in a hidden form which is supplied or extracted to change the state of a substance without changing its temperature. PCMs are classified as latent heat storage (LHS) units. Each PCM has a specific melting and crystallization temperature and a specific latent heat storage capacity. Phase change materials (PCM) take advantage of latent heat that can be stored or released from material over a narrow temperature range. These 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. Textiles containing phase change materials react immediately with changes in environmental temperatures and the temperatures in different areas of the body. This system can be used in protective clothing, beds, bedspread, space suits, diving suits, curtains, etc (10-28).

For any phase-changing material to be used in textile products, it must have certain properties. The main ones are; high melting / hydration temperature, high thermal conductivity, high specific heat capacity, minimum volume change during phase transformation, appropriate phase change temperature, repeatability of phase transformation, low corrosion and degradation tendency, non-toxic. Therewithal, the textiles should pass certain FR standards with the PCM material applied. Choosing the appropriate PCM for the protective clothing is crucial for an ideal thermal insulation and regulation effect. Many factors should be taken into consideration while making this choice. What is expected from PCM to be added to a textile product to be used as a garment is to minimize the heat flow between the person and the outside environment by keeping the body temperature constant at a certain value that the person is comfortable with. Suitable materials for textile products in terms of phase change temperatures; hydrate inorganic salts, polyhydric alcohol-water solution, polyethylene glycol (PEG), polytetramethylene glycol (PTGM), aliphatic polyester, linear long chain hydrocarbons, hydrocarbon alcohol, hydrocarbon acid etc. (28-39).

In general, the impregnation and exhaustion method can be used to transfer microcapsules in the textile industry. In the impregnation method, it is prepared in a liquor and the capsules are mixed into this liquor at a certain rate. Afterwards, the fabric is absorbed into this float and passed through the foulard machine and the process is completed with the pressure of the cylinders. To date, tiny research has been done on possible applications of microcapsules in functional coating processes. In the coating method, a coating paste is prepared and added the capsules in this paste at a certain rate, too. One of the most important problem of PCM is low thermal conductivity For example, paraffin has 0.22 W/(m·K) thermal conductivity when compared with >3000 W/(m·K) for multiwall carbon nanotubes (MWCNTs). Moreover, microencapsulated PCMs have a polymeric shell, which not only prevents the content from leaking but also resists heat transition at the same time. In the case where the capsules are transferred to the fabrics with the coating, another viscous coating layer is added on the shell material of the capsule. It is thought that this feature will increase in cases where PCM capsules are transferred by the coating method compared to the ones transferred by the impregnation method (40-

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43). Within the scope of this study, it is thought that the coating application can be applied especially in black out curtains. In this study, PCM microcapsules were used to develop thermoregulating textile materials and it is aimed to examine the effect of application method for microcapsules, too. In this research, Mikrathermic P PCM microcapsules were transferred to 100% cotton woven fabrics by the method of impregnation and coating. The thermal regulation properties of the fabrics were analyzed by differential scanning calorimetry (DSC) and the surface morphological properties by scanning electron microscopy (SEM). In addition, the thermal properties of the fabrics were obtained with a thermal camera. Contact angles and water vapor permeability of coated and impregnated fabrics were investigated.

2. MATERIAL and METHOD

2.1. Material

In this research, desized, 100% cotton fabrics (warp/weft yarn density of 34/17 yarns/cm) were used. Mikrathermic P PCM capsules were provided by Devan Chemicals, Belgium. For the coating process, Mikracat B as a cross linker and L Mikrasoftener as a softener were supplied from Devan Cehmicals. Rucocoat PU 1110 polyurethane coating material was used for coating process and supplied from Rudolf Duraner, Turkey. Edolan MR polyurethane binder were used for the impregnation method and provided by Tanatex, Switzerland to bond the microcapsules to the fabric. All other auxiliary chemicals used in the study were of laboratory-reagent grade.

2.2. Application of the Microcapsules to the Cotton Fabrics

The application of the capsule to the cotton fabrics was carried out with impregnation and coating method. Fabrics were conditioned in accordance with EN ISO 139 at standard atmospheric conditions (20 °C±2 and 65% RH±4) during 24 hours. Capsule transfer prescription were made according to the Table 1 and 2 which was given below and in the same ratio to compare the application processes. Polyurethane was selected as binder and each experiment was repeated 3 times.

Table 1. Capsule Transfer Prescription for Impregnation Method

Mikrathermic P - Capsule (g/l)	***Edolan MR - PUR Binder (g/l)***	***Pick-Up Ratio (%)***	***Drying***		***Fixing***	
			Temperature (ºC)	Time (min)	Temperature (ºC)	Time (min)
125	30	90	80	10	140	3

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The capsule was impregnated in a solution bath containing capsules (125 g/L) and binding agent (30 g/L), and then squeezed between rollers to 90% wet pick-up. Achieving long lasting effect, the fabric was exposed to drying for 10 min at 80°C and fixation process for 3 min at 140°C in a laboratory Stenter (Table 1).

Table 2. Capsule Transfer Prescription for Coating Method

Content	PU Paste
Mikrathermic P Capsule (g)	125
Rucocoat PU 1110 (g)	770
Mikracat B cross-linking agent (g)	100
L Mikrasoftener (g)	5

Viscosities of the coating pastes were measured by using Brookfield viscometer and the viscosity of the coating paste was determined to be 9000 cps. Cotton base fabrics were coated with the above mentioned coating pastes by using laboratory type blade coating machine, as two layers of coating. It was subjected to intermediate drying at 100 ºC for 2 minutes between two layers. Coated samples were cured at 140°C for 3 minutes.

2.3. Evaluation of Treated Fabrics

SEM images were taken to obtain the existence of capsules on the textile surface from both coated and impregnated samples. Samples were gold-coated (15 mA, 2 min) to assure electrical conductivity. The measurements were taken at 2 kV accelerating voltage. The images were taken at 250 and 1000×magnifcation.

Thermal properties of the fabrics, such as melting and crystallizing temperatures and enthalpies, were measured by differential scanning calorimetry. DSC was performed using a Perkin-Elmer diamond differential scanning calorimeter to distinguishing the capsules on the fabric with the help of characteristic endothermic or exothermic peaks, too. The samples were cooled down at -20^{o}C and then heated up to 40^{o}C at a constant rate of 10^{o}C/min under a nitrogen flow rate of 60 ml/min.

In order to examine the efficiency of the transferred capsules, the temperature of the fabric surface was measured at a certain time interval for raw fabric samples containing PCM by thermal camera as Figure 1. Measurements in the system were made in an insulated box. Before measurement, the inner temperature of the box was heated to a constant temperature of 40°C and the test was carried out at this temperature. The inner temperature of the can was kept constant by means of a thermostat. Also, before

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measurement, the fabrics were conditioned for 12 hours and placed in the box as quickly as possible. Once the fabric was placed in the box, the surface temperature was measured from a fixed point for 15 minutes. Thermal camera (Fluke Ti100 Thermal Imager, emission value 0.94) was used in the measurements and the temperature was recorded every 30 seconds.

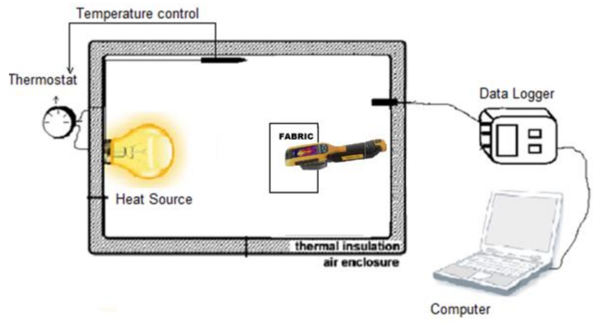

Figure 1. Thermal camera system (18)

When an interface exists between a liquid and a solid, the angle between the surface of the liquid and the outline of the contact surface is described as the contact angle θ (lower case theta). The contact angle (wetting angle) is a measure of the wettability of a solid by a liquid. In order to examine the hydrophility properties of the fabrics, contact angle analysis was examined. The measurements were carried out at 25°C using the Biolin Scientific Brand Theta Lite T 101 model contact angle device. The image of approximately 5 μL of water droplet dropped onto the surface to be m easured was recorded for 10 seconds by a camera of the device. Using the software of the device, an average of 200 data was recorded for 10 seconds for each sample and the arithmetic mean was taken.

Water vapour permeability is related to breathability of fabrics. Water vapour permeability of samples was determined by using SDL Atlas International M261 model water vapour permeability tester, according to BS 3424-34: 1992-Method 37 (44). The amount of water vapour passed through the samples was determined after 24 h and permeability values were calculated. Test was repeated 3 times for each sample type.

3. RESULTS and DISCUSSION

After the capsules containing PCM were transferred to cotton fabrics by impregnation and coating methods, analyzes were carried out on the fabrics.

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3.1. Scanning Electron Microscope (SEM)

SEM images of the Mikrathermic P PCM capsule was indicated in Figure 2. Mikrathermic P was around 3 μm and had a spherical shape as expected.

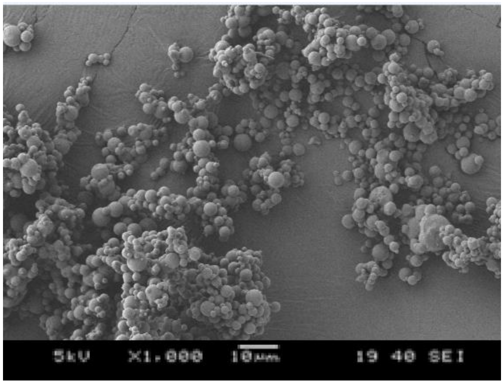
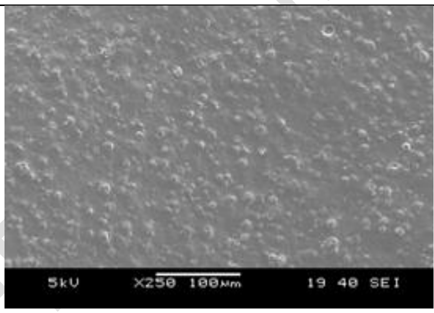
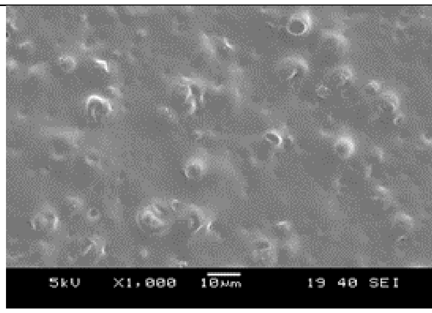
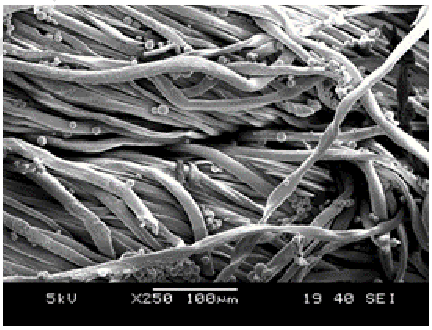
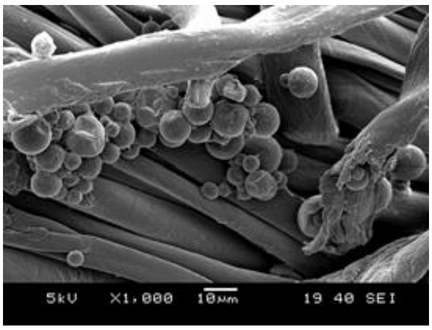


Figure 2. SEM images of Mikrathermic P capsules(1000x)

SEM images of the PCM capsules transferred to cotton fabrics by coating and impregnation method, enlarged 250 and 1000 times, are given in Table 3.

Table 3. SEM photomicrographs of fabrics treated with PCM capsules

	250x	1000x
Coated Fabric		
Impregnated Fabric		

When the images were examined morphologically, it was observed that the capsules transferred by the impregnation method preserved their spherical form. It was seen that PCMs transferred by coating remain under the coating polymer and were homogeneously distributed over the entire surface. These images showed that capsule application were

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succeeded for both method which was impregnation and coating. It was observed that capsules were covered with the binder and fixed onto the textile surface for cotton fabrics.

3.2. Differential Scanning Calorimeter (DSC) Analysis

The DSC diagrams of coated and impregnated fabrics are given in Figure 3.

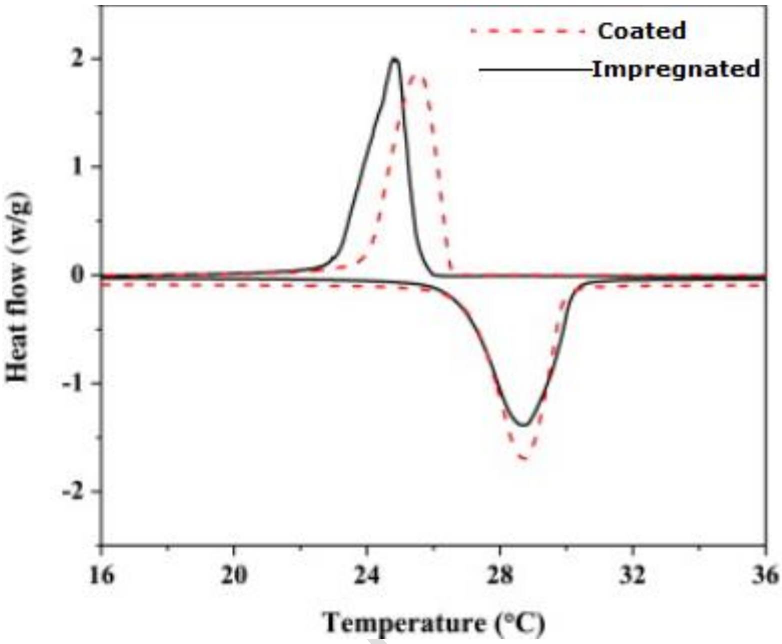

Figure 3. DSC diagrams of coated and impregnated fabrics with PCM capsules

The heat storage capacity of the Mikrathermic P PCM microcapsule was 140 J/g according to the literature (45-47). From the DSC curve is given in the Figure 3 and from the Table 4, the amount of heat stored and emitted by the fabrics from the area under the endothermic and exothermic melting and solidification peaks and the temperatures at which heat storage and emission begins can be seen. According to the DSC analysis, the values were obtained similar for coated and impregnated fabrics. On the other hand, the values were examined in Table 4, in detail.

Table 4.Thermal properties of coated and impregnated fabrics

Fabric	Melting point (°C)	Melting enthalpy (J/g)	Crystallization point (°C)	Crystallization enthalpy (J/g)
Coated	25.83	2.70	25.70	-1.45
Impregnated	25.72	2.64	25.61	-1.39

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The melting process in fabrics coated with Mikrathermic P microcapsules occurred between 25.83°C - 31.04°C and the amount of heat energy stored by the cotton fabric during the melting period was measured as 2.70 J/g. For the Mikrathermic P microcapsule, the crystallization process occurred in the range of 25.70°C – 23.45°C and the cotton fabric released -1.45 J/g heat during crystallization. Impregnated fabric, which absorbed 2.70 J/g at 25.72°C during melting, and released –1.39 J/g at 25.61°C during crystallization.

Thermal conductivity measures the capacity of temperature exchange between heat and cold passing through a material mass. Decreased thermal conductivity allows for a faster rate of heat transfer in phase change material, increasing the time required for the PCM to undergo a complete charge or discharge. The major shortcoming of PCM is its limited ability to exchange heat efectively due to low thermal conductivity. This suppresses the amount of thermal heat that can be exchanged during melting processes and a lower thermal conductivity of the solidification will occurred at low temperatures. Increasing the efective thermal conductivity of PCM can be achieved by many mechanisms such as inserting fins and dispersion high thermal conductivity nanoparticles (48, 49).

Although the process temperatures are very close to each other, coated fabrics have changed state at higher temperatures compared to impregnated fabrics. The case of shifting of the process peaks to higher temperatures has been explained in the literature as the lower thermal conductivity of the fabric (50). This situation was interpreted as the lower thermal conductivity value of coated fabrics compared to impregnated fabrics resulting in melting and solidification at higher temperatures. However, considering that these data are very close to each other, it was thought that the capsules can be transferred to the fabrics by the coating method. Encapsulated PCMs which were transferred with coating and impregnateion, lead to lower thermal conductivity and increased heat capacity of a textile structure. They improve the thermal performance of textile material and therefore may save energy.

3.3. Thermal Camera

Depending on the change in ambient temperature, the fabric surface temperature change caused by PCM capsules was measured. For this purpose; thermal camera was used to determine the heat regulation properties of fabrics that can store heat. Two measurements were taken from two different points in the fabric samples and their averages are shown in the Figure 4.

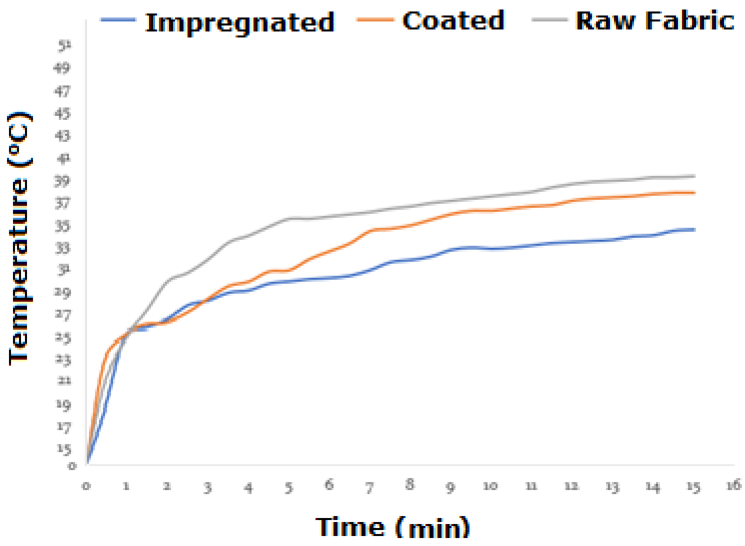

Figure 4. Thermal camera results of the fabric

The temperature-time curves of the measurement results are given in the Figure 4. When the graph was examined, it was seen that the fabrics which brought from the cold environment (4°C±2) to the warm environment (40°C±2) were warmed and the temperatures measured on their surfaces were increased. On the other hand, it is observed that the heating time of the fabrics in a hot environment and the maximum temperature they were reached were not equal. According to both measurement results, it was seen that the fabric that heats up the fastest was the raw fabric. Similarly, it was seen that the maximum surface temperature of the raw fabric was higher than the fabrics containing PCM. The raw fabric warmed to almost maximum temperature (about 42°C) in about 5 minutes. For fabrics containing PCM, the maximum temperature was recorded lower at the end of the measurement period. This value was recorded as 37°C for the fabric in which the PCM capsules were impregnated and 40-41°C for the fabric transferred with the coating. Thermal camera analysis was performed for 15 minutes. It was determined that the temperature of the fabrics remained at the last point which they reached, in the extended period. During the measurement period, it was determined that the temperature measured on the surface of the fabric to which the PCM capsules were impregnated was 3°C to 5°C lower than the raw fabric surface temperature. It was determined that the surface temperature of the fabric to which the PCM capsules were transferred with the coating was 1-3.5°C lower than the raw fabric.

When the analysis results were evaluated, it was seen that the fabric transferred to PCM with the impregnation method makes more effective temperature regulation. Because the impregnated fabric, which has the lowest temperature, was absorbed more heat in the cold environment when the PCM structure was applied. On the other hand, it also appears

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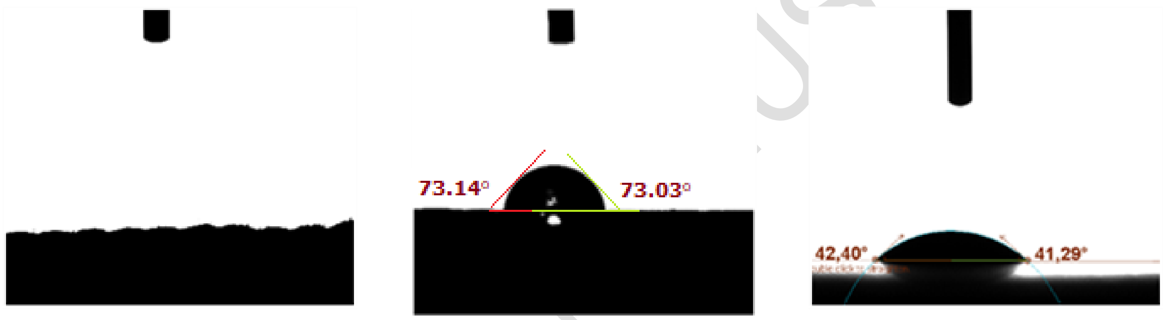
that there was not a big difference between coating and impregnation methods at the thermal camera analysis like DSC. In that point, thermal camera method demonstrates the heat regulation ability of fabrics, but does not provide information about their performance in end-use areas. Therefore, for fabrics treated with coating and impregnation method, performance evaluation according to the area of use will give the most accurate result. This shows that PCM capsules can also be transferred by the coating method according to the usage areas.

3.4. Contact Angle Measurement

In order to evaluate the hydrophilicity properties of raw fabric and PCM-transferred fabrics with different methods, contact angle measurement was made and the following figures were obtained and shown in Figure 5.



Raw Fabric | Coated Fabric | Impragnated Fabric

Figure 5. Contact angle images of fabrics

The angle between the surface of the liquid and the outline of the contact surface is described as the contact angle θ. The contact angle is a measure of the wettability of a solid by a liquid. In the case of complete wetting, the contact angle is 0°. Between 0° and 90°, the solid is wettable and above 90° it is not wettable. When the analysis results were examined, water were completely absorbed by raw fabric in 5 seconds and this indicates that the fabric is hydrophilic. When comparing the transfer methods of PCM capsules, contact angle of impregnated and coated fabric was obtained as 42° and 73°, respectively. In general, the coating paste has a more viscous structure and this structure causes a thick layer to form on the fabric. Due to this structure, the surface energy of the fabric decreases and it gains water repellency. In the impregnation method, since a viscous structure is not obtained and a layer is not formed on the fabric surface, the contact angle becomes lower and it causes that the textile material be more hydrophilic than the coated one. This result, as expected, was indicated that the coated fabric was more hydrophobic than the impregnated fabric.

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3.5. Water vapour permeability

Water vapor permeability analysis was carried out to examine the comfort properties of the fabrics obtained. Water vapour permeability of samples are tabulated in Table 5.

Table 5. Water vapour permeability results of fabrics

Fabric	Water vapour permeability (g/m²·24 h)
Raw Fabric	625.44
Impregnated Fabric	619.02
Coated Fabric	352.18

Highest water vapour permeability was obtained from raw fabric with 625 g/m2/24h permeability value. It was determined that the fabrics transferred to PCM by the impregnation process gave a similar result to the raw fabric. On the other hand, water vapour permeability of coated samples reduced to approximately 50% raw base fabric in parallel with the contact angle results. This was due to additional PU coating layer which contributed mass transfer limitation through the fabric. Even the most breathable coating polymer was applied to the samples; it would add a resistance to the vapour flow by closing the pores and creating an additional layer (51). The water vapor permeability of a material plays an important part in evaluating the physiological wearing comfort of clothing systems or determining the performance characteristics of textile materials used in technical applications. Therefore, it is important to choose the transfer method of PCM capsules considering the area where the fabric will be used.

CONCLUSION

Within the scope of this study, PCM capsules were applied to the textile materials with coating and impregnation methods, successfully. As a result of the study, it was observed that the capsules transferred by the impregnation method preserved their spherical form according to the SEM images. It was seen that PCMs transferred by coating remain under the coating polymer and were homogeneously distributed over the entire surface. When thermal properties of coated and impregnated fabrics were examined with DSC analyses, it was seen that thermal behaviours of impregnation and coating method were similar. According to the results of the thermal camera, it was seen that the PCM transferred fabric with the impregnation method performs more effective temperature regulation. The fabric transferred to PCM with the impregnation method makes more effective temperature regulation. Because the impregnated fabric, which has the lowest

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temperature, was absorbed more heat in the cold environment when the PCM structure was applied. Impregnation method was showed better results according to the thermal camera although it was close to each other with coating method. As predicted, it has been found that the contact angle of the coated fabric was higher and the air permeability was lower than the impregnated fabric. However, the thermal results obtained show that PCM capsules can also be transferred by the coating method. This situation makes the end use area of the fabric to be used important. There are lots of clothing comfort properties of textiles such as heat transfer, thermal protection, air permeability, moisture permeability, water repellence and etc. While it may be preferred to use the impregnation method at the points where comfort features are important, it was thought that PCM capsules can be transferred by the coating method if the comfort features are not important. Performance evaluation according to the target properties of textile material will give the most accurate result for the fabrics which are treated by coating and impregnation method. On the other hand, it was considered that the coating method may be an alternative to the impregnation method. According to the results, it eas thought that the fabrics in which the capsules were transferred by coating can be used in black out curtains. Besides, it was foreseen that the fabrics to which the capsules were transferred by impregnation method can be used in bedding fabrics or clothes considering their comfort properties.

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