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A STUDY ON DEVELOPING THERMO-REGULATING DENIM FABRIC BY INTEGRATING OF MICROENCAPSULATED PHASE CHANGE MATERIAL USING EXHAUSTION METHOD

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Abstract: *A blend of octadecane and organic coconut oil were used as phase change material in core and melamine formaldehyde was used in shell to produce microencapsulated thermal storing material. A melamine resin containing methylol was used as crosslinker for the polymer shell. The microencapsulation was processed using in-situ polymerization method. The synthesized microcapsules (microPCM) were applied to a stretch denim fabric with composition of 98 % cotton and 2% elastane, which is widely used in daily life as jeanswear, by using exhaustion method in different concentrations. The core material, the microcapsule and the treated fabrics were analyzed by Fourier Transform Infrared Spectroscopy (FT-IR), Differential Scanning Calorimetry (DSC) and Scanning Electron Microscopy (SEM). FT-IR results revealed that the microcapsules were fabricated successfully. SEM images indicated that the microcapsules were yielded in spherical form with a particle size of 1-3 µm. and the particles were achieved to be exhausted in the fabric texture, comparing to untreated fabric. DSC results showed that the microcapsules stored latent heat of melting 111 J/g at a peak temperature of 28.1ºC and the treated fabrics possessed latent heat of melting 0.7-0.8 J/g at peak temperatures of 25.2-25.5 ºC which are in human comfort temperature range. It was calculated that the microcapsules were obtained with a 70.3% encapsulation yield in 63.4 % core content.*

Key words: *Denim, thermo-regulation, phase change material, microencapsulation, textile.*

1. INTRODUCTION

Functional smart textiles have been drawing attention increasingly. Phase change materials (PCMs) are thermal energy storing materials which have been using for functional textiles as well as in building materials, solar cells and panels, electronic equipment, etc. for thermal regulation purpose. PCMs can store or release high amount of latent heat during phase change. PCMs with melting point in between 15-35 ºC, which are close to human skin temperature are the most effective ones to use in textiles [1]. Linear long chain hydrocarbon paraffin waxes, polyethylene glycols and fatty acids are the most usable PCMs for textiles considering phase change temperatures. PCMs used for textiles are mainly microencapsulated within a polymer shell to prevent the leakage of the material during solid to liquid phase change and to provide a better thermal conductivity. PCMs are usually integrated to fabric with padding, coating or lamination as fabric treatment [2,3].

In this study, a blend of organic coconut oil and octadecane was investigated to be used as phase change material. Coconut oil consists of high amount of low molecular weight of saturated fatty acids which mainly come from lauric oil. It has high latent heat capacity to be used for thermal energy storage. Wi et al. prepared shape stabilized PCMs using palm oil and coconut oil as phase change materials, impregnated into exfoliated graphite nanoplatelets to use in buildings for saving energy [4]. Silalahi et al. examined the temperature of organic coconut oil vs. time by T history method during phase change from liquid to solid and revealed that phase change took place gradually depending on the oil composition of different kinds of fatty acids [5]. The main idea in this study was to obtain a thermal storing material in a wider phase change temperature interval to give comfort to the wearer in a wider temperature scale. In addition, the use of this blend also provided an economic advantage for the cost of the core material. Furthermore, exhaustion method was processed to give the microPCMs to the fabric texture, which was not widely studied according to the literature. The microencapsulation was processed by in situ polymerization which was one of the main encapsulation methods [6].

2. EXPERIMENTAL

2.1 Material

100% Organic Coconut Oil supplied by the LifeCo Company, Turkey and 98% crystalline mass/melt n-octadecane from Alfa Aesar were used as PCM core materials. Melamine (99% pure) from Aldrich, formaldehyde (37% aqueous solution by weight) from Merck were used as shell materials. Sodium dodecyl sulphate (SDS) from Neofroxx Gmbh was used as emulsifier surfactant. Melateks 700 containing methylol from Melamin Kemicna was used as a crosslinking agent for the melamine formaldehyde polymer shell, ammonium chloride (extra pure) from Riedel-de Haen was used as nucleating agent in encapsulation and triethanolamine (99% pure) from Merck was used for adjusting the pH in reactions. Stretch denim fabric with composition of 98% Cotton 2% Elastane, 280 g/m^2 was supplied from Çalık Denim, Turkey. Orgaresin HC 77 was used as acrylic binder, Orgafix DX New was used for fixing agent, Dispersant 850 K was used as dispersing agent which were supplied from Organik Kimya San. ve Tic. A.S., Turkey.

2.2. Microencapsulation of microPCM

A blend of 70% octadecane 30% organic coconut oil was used in core and encapsulated with melamine formaldehyde shell by in situ polymerization. A pre-polymer solution of melamine formaldehyde was prepared by dissolving 6 g. of melamine and 30 mL of formaldehyde in 30 ml of distilled water by magnetic stirring at 70 ºC. The pH of the solution was adjusted to 8.5-9 using triethanolamine. After the solution became transparent, 1 g of melamine and 10 mL of distilled water were added to the solution. The emulsion solution was prepared by emulsifying of 28 g octadecane and 12 g. organic coconut oil in 300 mL distilled water by mechanical stirring at 2500 rpm for 3 hours at 50 ºC, with the addition of 4 g of SDS as emulsifier and 1.5 g of Melateks 700 as crosslinker. The pH of the emulsion solution was adjusted to 3.5-4.0 with the addition of acetic acid. The pre-polymer solution was added to the emulsion solution while stirring at 600 rpm. The encapsulation reaction occurred at 60 ºC during 90 minutes. 2 g of ammonium chloride was used as nucleating agent. The reaction was ended by adjusting the pH of the solution to 8-8.5. The microcapsules were collected by filtering and washed repeatedly with 30% ethanol solution and then dried at 50 ºC during 24 hours.

2.3. Exhaustion of microPCM into fabric texture

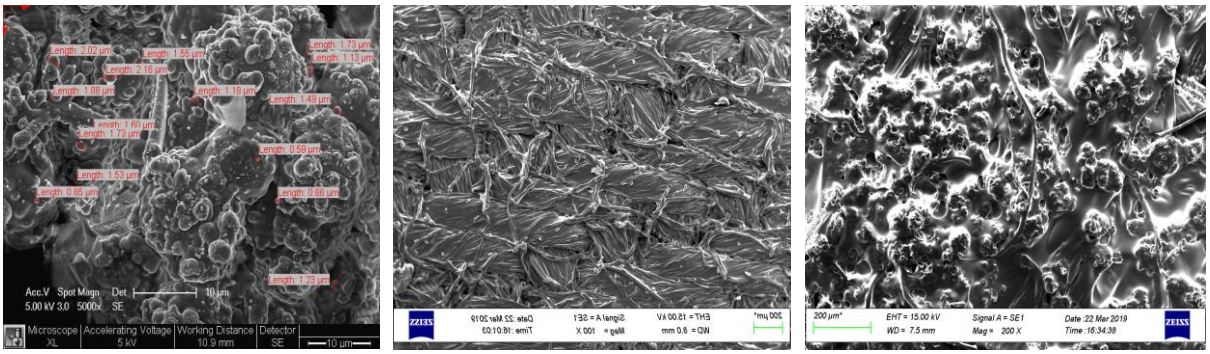
Two pieces of 4 cm x10 cm fabric were immersed in separate three tubes of Gyrowash machine and the recipes given in Table 1 were applied to 2.50 g of dry fabric samples in average. One fabric sample was treated with the solution without microPCM, the others were treated with different concentrations of microPCM as given in Table 1. The solutions were prepared with a well dispersing of microPCM with dispersing agent and distilled water by magnetic stirring. The exhaustion process was applied at 50 ºC during 1 hour.

2.4. Characterization

The core PCM material and the fabricated microcapsule were examined by Fourier transform infra red spectroscopy (Perkin Elmer Spectrum 100 FT-IR) to analyze the chemical composition in the frequency range of 380 cm^{-1} and 4000 cm^{-1} .The thermal storage capacities and the phase change temperatures during melting and crystallization of the core PCM, the microPCM and the microPCM treated fabric samples were measured by differential scanning calorimeter (Perkin Elmer Diamond DSC) at a heating or cooling rate of 10 ºC min^{-1}, in the temperature range of -10 ºC and 50 ºC, within nitrogen atmosphere 25 mL min^{-1}. The encapsulation ratio (R) which is the core PCM content of the microcapsules [7,8] and the encapsulation yield (EY%) which is the ratio between the mass of dry microcapsules (m_{MPCM}) and the total mass of the polymer shell components and the core (m_t) [9] were calculated using the equations of 1 and 2, respectively. The surface morphology of the microPCM treated nonwoven fabric layers were analyzed by scanning electron microscopy (Zeiss EVO-MA 10).

3. FIGURES AND TABLES

3.1. Figures



(a) ***(b)*** ***(c)***

Fig. 1*: SEM images of (a)microPCM, (b) untreated fabric, (c) treated fabric*

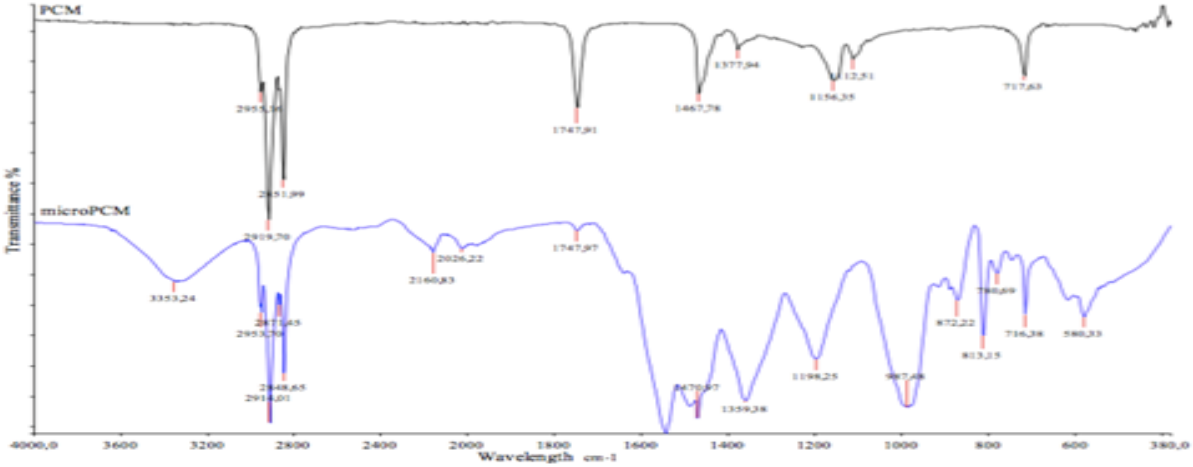
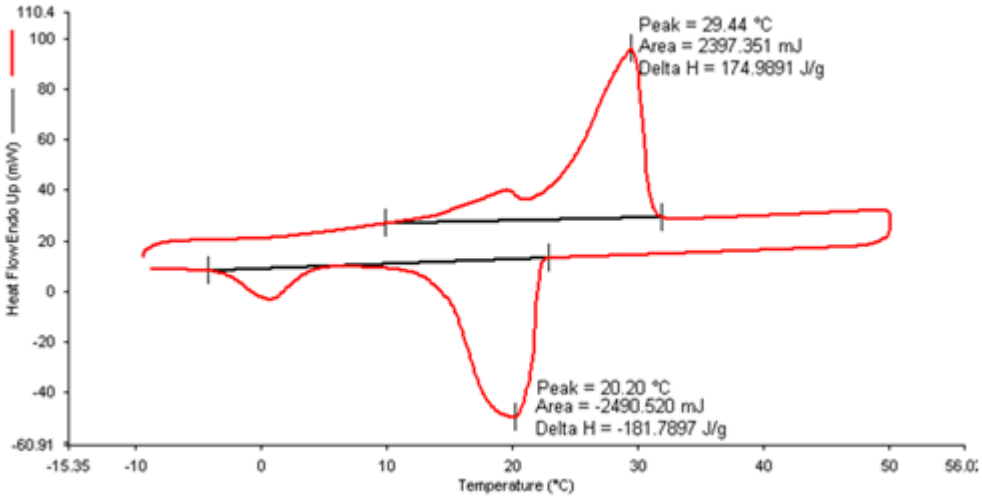
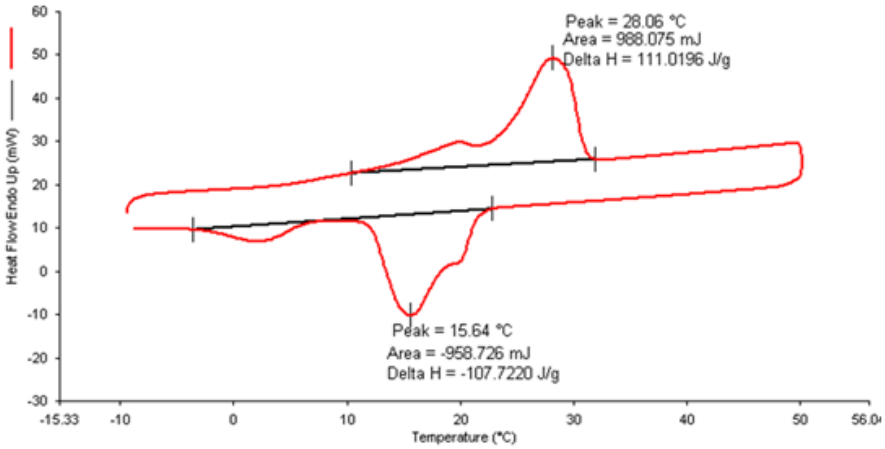


***Fig. 2**: FT-IR Spectra of PCM core and microPCM*



(a) *DSC graph of PCM core*



(b) *DSC graph of microPCM*

***Fig.3**: DSC graphs of PCM core and microPCM*

3.2. Tables

***Table 1:** Recipes for the exhaustion process*

Chemicals	R1	R2	R3
Distilled water	50 mL	50 mL	50 mL
Dispersant K 850	3 g	3 g	3 g
Orgal HC 77	45 g	45 g	45 g
Orgafix DX New	4 g	4 g	4 g
MicroPCM	---	2 g	3 g

***Table 2:** DSC results of PCM core, microPCM and microPCM treated fabrics*

Sample Name	Melting Enthalpy ΔH_m (J/g)	Melting Peak Temp. T_m (°C)	Crystallization Enthalpy ΔH_c (J/g)	Crystallization Peak Temp. T_c (°C)
PCM core	175.0	29.4	-181.8	20.2
microPCM	111.0	28.1	-107.7	15.6
microPCM treated fabric with R2	0.68	25.2	-0.96	18.1
mcroPCM treated fabric with R3	0.80	25.5	-1.01	17.6

4. EQUATIONS

$$R = (\Delta H_{m,MPCM} / \Delta H_{m,PCM}) \times 100 \quad \textbf{(1)}$$

$$EY\% = (m_{MPCM} / m_t) \times 100 \quad \textbf{(2)}$$

5. CONCLUSIONS

In this study, organic coconut oil and octadecane were blended and used as phase change material to obtain a thermal energy storing material which would store or release heat in a wider

temperature interval for thermal regulation in an optimum peak temperature for human comfort. Besides, exhaustion technique was experimented to integrate the microPCMs in the fabric texture, which was not majorly studied according to the literature. FT-IR spectra of PCM core and the microcapsule confirmed that the core material was encapsulated successfully. In spectra of the microcapsule, the absorption peak at 3353 cm^{-1} was attributed to O-H stretching vibration coming from the methylol content of the melamine resin crosslinked to melamine formaldehyde shell polymer. Calculations made by equations 1 and 2 showed that the microcapsules were obtained with an encapsulation yield of 70.3% in a core content of 63.4%. DSC results indicated that, the blend PCM core and the fabricated microcapsules stored latent heat of melting 175 J/g and 111 J/g at a peak melting temperature of 29.4 °C and 28.1 °C, respectively, which are remarkably high values. DSC results also showed that microPCM treated fabrics possessed latent heat of melting 0,7-0.8 J/g. which would be improved and optimized in future studies. SEM images revealed that the microPCMs were exhausted on the fabric surface and embedded in the binder material with a dense and uniform distribution.

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