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Microencapsulation in Thermo-regulating Fabric : An Overview

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ABSTRACT

Microencapsulation technology was utilized in the early 1980s by the United State National Aeronautics and Space Administration (NASA) with the aim of managing the thermal barrier properties of space suits. The microcapsules have walls less than 1 μm thick and are typically 20 - 40 μm in diameter, with a Phase Change Material (PCM) loading of 80% - 85%. These containers release their core contents under controlled conditions to suit a specific purpose. The microcapsules are produced by depositing a thin polymer coating on small solid particles or liquid droplets, or on dispersions of solids in liquids. The small capsule size provides a relatively large surface area for heat transfer. The rate at which the PCM reacts to an external temperature change is very rapid. PCM microcapsules are being used to develop textile materials with higher thermal mass and PCM slurries with high heat storage and transfer properties. In this paper, Melamine–formaldehyde microcapsules containing eicosane were prepared by *in situ* polymerization. Knit fabrics made of polyester, were treated with phase change-material microcapsules by a pad–dry–cure method with a polyurethane binder. The treated fabrics were evaluated in terms of the thermal properties, moisture vapor permeability, moisture regain, air permeability, with respect to the add-on of microcapsules. The morphology and laundering properties of the treated fabrics were also evaluated.

Keywords: Textile microencapsulation, PCM, Thermo-regulated fabric, Smart textiles

I. INTRODUCTION

In 1987, the microencapsulation technology of Phase Change Materials was developed and incorporated with textile materials [1]. Microencapsulation is defined as a process in which tiny particles or droplets are surrounded by a coating, or embedded in a homogeneous or heterogeneous matrix, to give small capsules with many useful properties [2]. In applications of PCM technology to garments and home furnishing products, PCM microcapsules are incorporated into acrylic fibers or polyurethane foams or are embedded into coating compounds and topically applied to fabrics or foams [3, 4]. 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 [5].

The purpose of this study was to preparation, application and evaluation of microencapsulation applied in thermo-regulating textile materials like fabric. Thermoregulation is the ability of an organism to keep its body temperature within certain boundaries, even when the surrounding temperature is very different [17].

Melamine–formaldehyde microcapsules containing eicosane (chemical formula $C_{20}H_{42}$) were prepared by *in situ* polymerization and were characterized with respect to their structure, morphology, size distribution, thermal properties, and stability. Polyester knit fabrics were treated with the prepared microcapsules with a polyurethane binder by a conventional pad–dry–cure (PDC) process. The treated fabrics were characterized with respect to their morphology and thermal properties, and the laundering durability was evaluated for practical use. The microcapsules were spherical and had melamine–formaldehyde shells containing eicosane. The microcapsules were strong enough to secure capsule stability under stirring in hot water and alkaline solutions. The heat storage capacity increased as the concentration of the microcapsules increased. The thermo-regulating fabrics had heat storage capacities of

0.91– 4.44 J/g, which depended on the concentration of the microcapsules [6].

The treated fabrics retained 54% of their heat storage capacity after five launderings. In addition to the thermal properties, moisture vapor permeability, moisture regain, and the air permeability of materials also influence the heat balance of the body and, consequently, affect clothing comfort [7]. This paper focuses on the effects of the PCM microcapsule add-on level on the thermal properties, moisture regain, moisture vapor permeability, and air permeability of the treated fabrics.

II. METHODS AND MATERIAL

The fabric was a scoured and bleached 100% polyester knit (68×58/in.^{2}) with a weight of 195 g/m^2 and a thickness of 1.47 mm [19]. Melamine and 37% formaldehyde as shell materials, eicosane as a core material, sodium lauryl sulphate as an emulsifier, poly (vinyl alcohol) (PVA; weight average molecular weight = 1500) as a protective colloid, and acetic acid and anhydrous sodium carbonate as pH controllers were used. All the chemicals were reagent-grade. The characteristics of the prepared microcapsule are summarized in Table 1.

Table 1: Characteristics of eicosane containing microcapsules

Size (μm)	T_m (°C)	T_c (°C)	ΔH_e (J/g)	ΔH_c (J/g)
1.89	36.9	31.7	134.3	132.9

Eicosane ΔH_f= 263.7 J/g, T_m= melting temperature, T_c= crystallization temperature, ΔH_f = heat of fusion, ΔH_c= heat of crystallization

A. Preparation of the microcapsules

At first, 0.1*M* Melamine and 0.3*M* 37% formaldehyde in 100 mL of distilled water were adjusted to pH 8.5–9.0 with a 10% sodium carbonate solution and stirred at 60°C for 1 hour to prepare a melamine-formaldehyde pre-polymer. An oil-in-water emulsion of eicosane (10 g) in 100 mL of a 1% SLS aqueous solution was prepared via stirring at a speed of 6000 r.p.m [19]. The prepared emulsion was added to the pre-polymer to start *in situ* polymerization, and the pH was lowered to 4.0 –5.0 with acetic acid. Subsequently, a 0.001*M* PVA solution was poured into the emulsion/pre-polymer system, and the mixture was stirred at 50°C for 1 h to prevent the agglomeration of emulsion globules. The resultant microcapsules in the slurry state were filtered, washed in distilled water, and dried at room temperature to obtain a microcapsule powder. The yield of the microcapsule powder was 32%.

B. Application of the microcapsules to the fabrics

The fabric samples were impregnated with an aqueous solution composed of a plurality of microcapsules and a polyurethane binder (Snotex P110, Dae Young Chemical Co, Ltd., Seoul, South Korea), were padded up to 300% pickup by the two-dips/two-nips method [4] were dried at 80°C for 8 min, and were cured at 130°C for 10 min. The concentrations of the microcapsules were 12.5, 25, 50, and 100% with respect to the weight of the undiluted microcapsule slurry. The concentration of the binder was 3% (on the weight of both), and liquor ratio was 32:1. The treated fabric samples had been washed and dried for further evaluation.

C. Evaluation of the treated fabrics

The surface of the microcapsule treated fabrics was observed with a scanning electron microscope (JSM-5400, JEOL, Inc., Tokyo, Japan). The heat storage capacity and phase-change temperatures were measured with differential scanning calorimetry. Optical microscopy was performed for the observation of the microcapsules in the slurry state. The mean particle size and size distribution of the microcapsules were determined with an image analyzer (MS100, Malvern Instrument, Malvern, United Kingdom). A differential scanning calorimetry (DSC) instrument (DSC2920, TA Instrument, New Castle, DE) was used to measure some thermal properties. The heating and cooling rate was 2°C/min. up to 50°C under an atmosphere of N_2. The air permeability (Frazier method; ASTM test method D737-96), moisture vapor permeability (ASTM test method E96-95), and moisture regain (ASTM test method D885-98) were measured with standard procedure [8]. The laundering durability of the treated fabrics was evaluated with an LHD-EF launderometer (Atlas Electric Devices Co., Houston, TX) according to AATCC test method 61-1989 [6].

III. RESULTS AND DISCUSSION

A. Morphology of the microcapsule treated fabrics

Fig. 1 shows scanning electron microscope (SEM) photographs of control, binder treated, and microcapsule treated fabric. The control and binder-treated fabric samples had very smooth appearances. The microcapsules in the microcapsule treated fabric samples were located at interstices between the fibers and on the fiber surface. Those microcapsules were heat-resistant and could endure the curing conditions (at 130°C for 10 min).

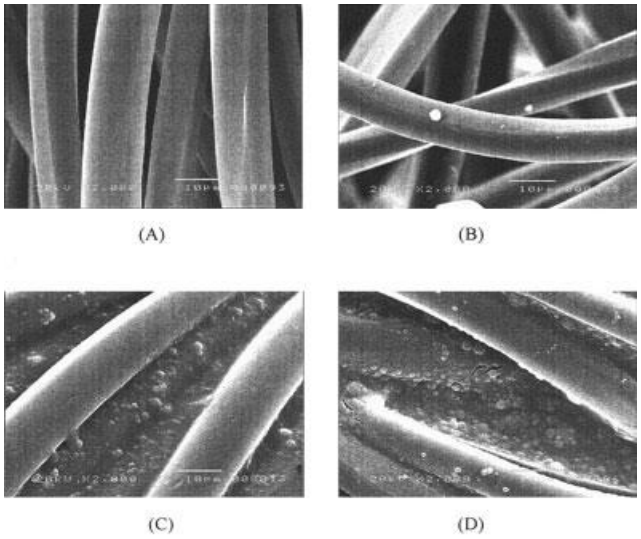

Figure 1: SEM photographs (original magnification = 2000×): (A) control, (B) binder-treated sample, (C) microcapsule treated sample (5% addition), and (D) microcapsule treated sample (23% addition)

Therefore, the microcapsules used in this study are suitable for the finishing process at high curing temperatures. Poly urea microcapsules manufactured by interfacial polymerization were melted at curing temperatures higher than 80°C [9]. On the other hand, more binder was observed in the microcapsule treated samples than in the sample treated with the binder only. Note that, some cracks were observed on the surface of the microcapsule-treated sample with 23% addition, which had the highest addition of the samples.

B. Characterization of the microcapsules

Fig. 2 shows an optical micrograph of the microcapsules in the slurry state and a scanning electron microscope (SEM) photograph of the microcapsules in the powder state. Most of the microcapsules were spheres with a smooth surface morphology. The agglomeration of the microcapsules was not observed in the slurry and powder states.

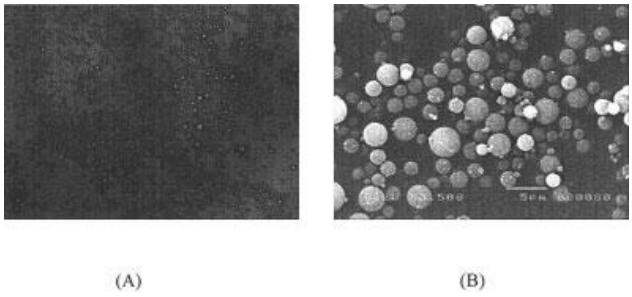

Figure 2: Morphology of the microcapsules: (A) optical micrograph (original magnification = 400×) and (B) SEM photograph (original magnification = 3500×)

Fig. 3 shows the particle size distribution of the microcapsules. The mean particle diameter of the microcapsules was 1.89 μm, and most of the microcapsules had particle sizes of 0.1–10 μm. For the addition of microcapsules to textile materials, the particle size and size uniformity are important factors [10] Pause [3] used PCM microcapsules of 1–60 μm to improve the thermal insulation of textile materials. Colvin and Bryant [11] used microencapsulated PCMs of 30–100 μm for textile fibers, composites, foams, and so forth. They also claimed that much larger particles of 1–3 mm could be placed within clothing layers to improve breathable thermal cooling under high humidity.

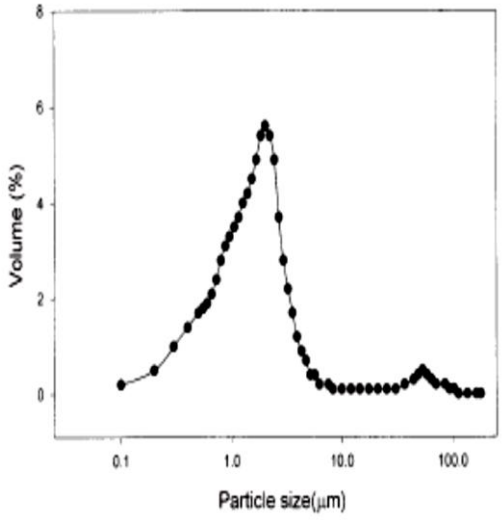

Figure 3: Particle size (μm) to volume (%) relation curve of the microcapsules

C. Stability of the microcapsules

The efficacy of the prepared microcapsules for textile materials, stability testing was performed under common laundering conditions for garments. Microcapsules for textile materials should be stable against mechanical action (e.g., abrasion, shear, and pressure) and chemical [10]. The microcapsules were stirred in neutral (distilled water) and alkaline (pH 11) solutions at 20 and 68°C and at 380 r.p.m for 1 h and 3 h, respectively. As shown in the scanning electron microscope (SEM) pictures of Fig. 4, the microcapsules did not show any significant changes in their morphology and size.

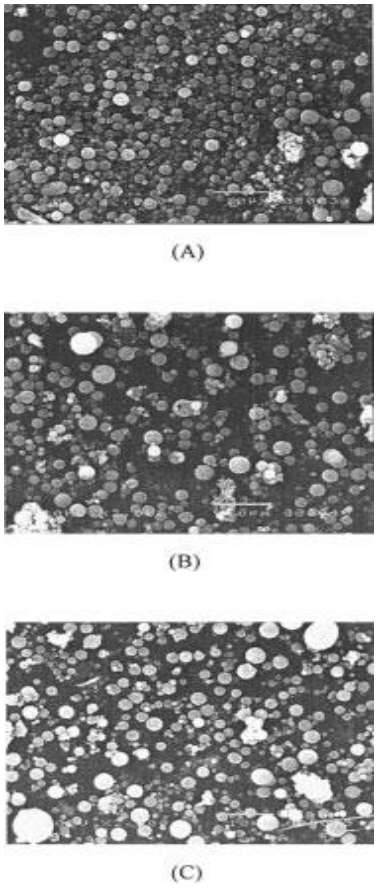

Figure 4: SEM photographs (original magnification = 2000×) of microcapsules treated with stirring under different conditions: (A) 20°C and 3 h; (B) 68°C and 3 h; and (C) pH 10, 20°C, and 3 h.

The heat storage capacities of the microcapsules before and after stability testing are presented in Table 2. On the basis of the heat storage capacity (134.3 J/g) before the testing, more than 90% of the heat storage capacity of the microcapsules was retained after the testing, regardless of the test conditions. The results confirmed that the microcapsules were durable enough to secure capsule stability during stirring in hot water and alkaline solutions.

Table 2: Effect of the water temperature and pH on the heat storage capacity of the microcapsules

Solution		Retention of heat storage capacity (%)	
Temperature (°C)	pH	1 h	3 h
20	Neutral[a]	99	89
68	Neutral[a]	100	99
20	Alkalin[b]	98	96

[a] Distilled water.
[b] pH 11.

D. Thermal properties of the treated fabrics

Table 3 shows the melting temperature and heat storage capacity versus the add-on of the microcapsules. As the add-on increases, the heat storage capacity of the treated fabric increases. Therefore, during the phase-change process, the rate of the temperature rise of the treated fabrics with a higher microcapsule add-on level is expected to be lower than that with fewer microcapsules [12]. The conventional PDC method used in this study seems to have limitations in loading microcapsules for high levels of thermal storage capacity. Other application methods being used currently also have some limitations in loading microcapsules. For example, microcapsules incorporated into the spinning dope of acrylic fibers have an upper loading limit of 5–10% microcapsules because the physical properties of the fibers begin to suffer above that limit, and the finest fiber available is about 2.2 dtex [10]. Although the loading can be as high as 60 wt % for coated fabrics, properties such as drape, breathability, softness, and tensile strength can be affected adversely as the loading increases [13]. PCM microcapsules into textiles should be selected according to the performance properties and end use of the finished products. The treated fabric with 22.9% add-on is capable of absorbing 4.44 J/g of heat if the microcapsules on the fabric undergo a melting process. The heat of absorption by the microcapsules

delays the microclimate temperature increase of clothing and results in a decrease of the sweat release from skin [14]. This leads to enhanced thermo physiological comfort and prevents heat stress.

Table 3: Heat capacity (ΔH_f) and melting temperature (T_m) of the treated fabrics depending on the add-on

Add-on (%)	T_m (°C)	ΔH_f (J/g)
5	35.3	0.91
11	34.9	2.15
18	35.3	4.10
23	34.9	4.44

T_m: melting temperature; ΔH_f: heat storage capacity.

The selection of a PCM should take into account the end use of the textile material. For example, if the textiles are used for underwear, a PCM with a phase change occurring in the range of skin temperatures should be selected. On the other hand, for the lining material of a ski suit, a PCM needs to phase-change at a much lower temperature [15]. The melting temperature of the treated fabric, 35°C, is slightly lower than that of the microcapsules. It can be speculated that a trace amount of impurities, possibly from the shells of microcapsules, may transfer into eicosane when the treated fabrics are cured at 150°C. This would cause the melting temperature of the microcapsule-treated fabrics to decrease. Because of the melting temperature of the fabrics treated with the microcapsules, the treated fabrics would be appropriate for outwear use in a warm environment.

E. Scanning electron microscopy (SEM) observation of the treated fabrics

Fig. 5 shows the micrographs of the surfaces of treated fabrics obtained from SEM observations. With 5.35% add-on, microcapsules with a binder fill up some of the interstices between fibers. As the add-on increases, more and more interstices are filled, and the microcapsule–binder layer covers most of the fabric surface at 22.9% add-on. Small cracks can be observed on the layer at 22.9% add-on. The surface morphology of the fabric is extensively changed by the microcapsule treatment, and this change affects the overall properties of the fabric.

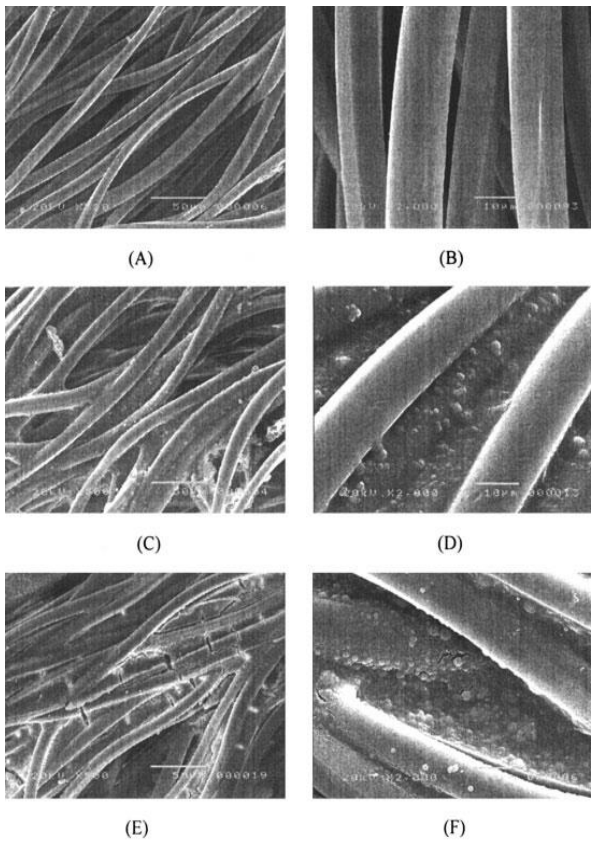

Figure 5: SEM photographs of an untreated sample at (A) 500× and (B) 2000×, a sample with 5.3% add-on at (C) 500× and (D) 2000×, and (c) a sample with 22.9% add-on at (E) 500× and (F) 2000×.

F. Air permeability and hygroscopic properties

Fig. 6–8 shows the moisture vapor permeability, moisture regain, and air permeability as functions of the microcapsule add-on, respectively. As the add-on increases, moisture vapor permeability and the air permeability decrease. The moisture vapor permeability and air permeability decrease by 20% and 28% at 22.9% add-on, respectively, in comparison with those of the untreated sample. There are some factors affecting the moisture vapor permeability and air permeability of the fabric, such as the fabric structure, thickness, and surface characteristics (pore size and porosity) [7].

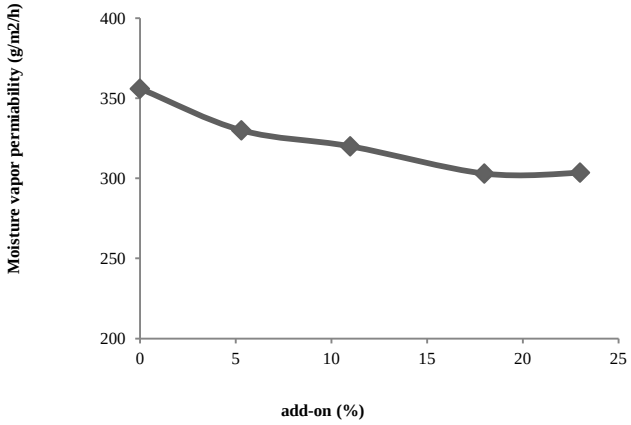

Figure 6: Moisture vapor permeability of the treated fabrics versus the microcapsule add-on.

As shown in the SEM pictures of Fig. 5, the microcapsules and binder fill up pores of the treated fabric, consequently, change the surface morphology, and increase the thickness of the fabric. These changes lead to a decrease in the moisture vapor permeability and air permeability. The moisture vapor permeability determines heat released by means of evaporative heat reflux [15] and affects the formation of condensation in a garment system [7].

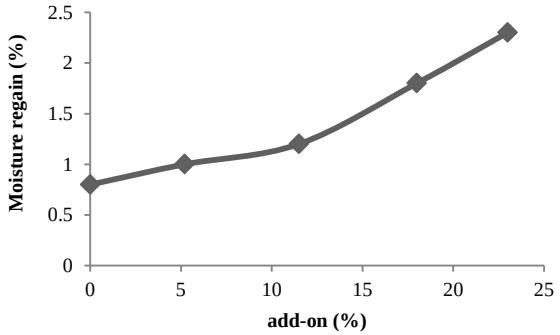

Figure 7: Moisture regain of the treated fabrics versus the microcapsule add-on

Therefore, the reduction of the moisture vapor permeability affects the thermal comfort of a garment adversely. On the other hand, the treated fabrics become more hygroscopic with increasing add-on. The moisture regain of the treated fabric with 22.9% add-on increases to 220% - 228% in comparison with that of the untreated sample. More hygroscopic material removes sweat more effectively from the skin or adjacent environment and helps with more effective wet heat loss through evaporation, leading to a pleasant microclimate in clothing [16]. It has been speculated that the hydrophilicity of the treated fabrics increases because of methylol groups in the shell material (melamine–formaldehyde) of the microcapsules [18] and hydrophilic binder.

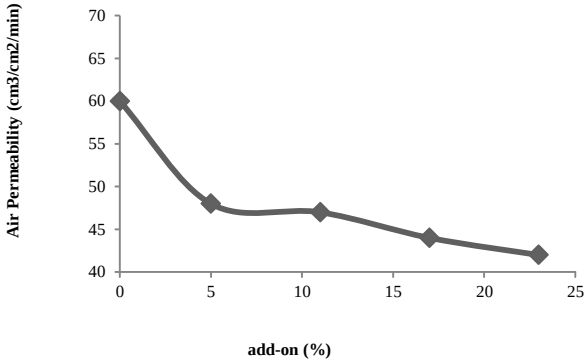

Figure 8: Air permeability of the treated fabrics versus the microcapsule add-on.

G. Laundering durability of the microcapsule treated fabrics

Fig. 9 shows SEM photographs of the samples after 1, 5, 10, and 20 launderings. The sample with 23% addition was used for the laundering durability test. The microcapsules were observed on the fiber surface and at the interstices between the fibers in the samples after one and five launderings.

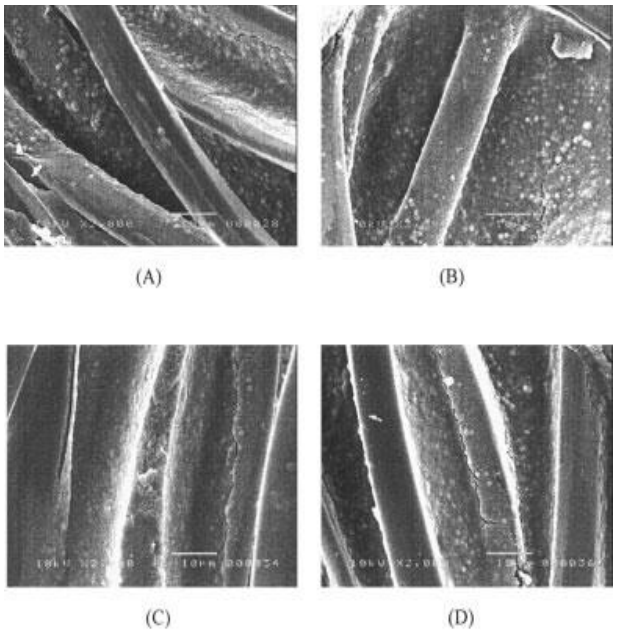

Figure 9: SEM photographs (original magnification 1000×) of microcapsule-treated samples after laundering: (A) 1 cycle, (B) 5 cycles, (C) 10 cycles, and (D) 20 cycles

This result indicated that the microcapsules on the fiber surface tended to come off more easily than those at interstices during laundering.

Table 4: Heat storage capacity of the polyester knit fabrics laundering effect

Laundering (cycle)	ΔH_f (J/g)	Retention (%)
0	4.44	100
1	3.21	72
5	2.42	54
10	1.80	40
20	1.74	39

Table 4 shows the effect of the laundering on the heat storage capacity (ΔH_f) of the treated fabrics. The heat storage capacity decreased as the laundering cycles increased. With respect to the heat storage capacity of the sample before laundering, 72% or 54% of the heat storage capacity was retained after one or five launderings, respectively. The largest decrease occurred after the first laundering. Microcapsules loosely attached to the fabric fell off during repeated laundering, and so the heat storage capacity of the treated samples decreased continuously up to 10 launderings; however, no more reduction in the heat storage capacity occurred thereafter. Kim and Cho [9] used an acrylic binder for coating PCM microcapsules onto fabrics and obtained 52–70% retention of the heat of fusion after 10 launderings. The selections of the appropriate binder and application method may result in a higher retention of the heat storage capacity of treated fabrics. Also, mild washing conditions would be helpful for better maintenance of microcapsule treated materials.

IV. CONCLUSION

The thermo-regulating polyester knit fabrics developed in this study showed heat storage capacities of 0.91–4.44 J/g. The treated polyester fabrics retained about 54% of their heat storage capacity after five launderings. A treated fabric with 22.9% add-on is capable of absorbing 4.44 J/g of heat if the microcapsules on the fabric undergo a melting process. The air permeability and moisture vapor permeability decrease by 28 and 20%, respectively, at 22.9% add-on. The moisture regains of the treated fabrics increases progressively 220% - 228% in comparison with that of the control fabric.

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