Microencapsulated Organic Phase Change Material And Its Functional Finishing On Textiles

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Received: July 10, 2018 Accepted: August 19, 2018

ABSTRACT *Comfort is a state of feeling pleasurable, well-being and satisfaction. Thermal or heat factor affects the wear comfort level. Development of fabrics which can adjust and maintain the thermal energy is necessary. Phase Change Materials (PCM) is the substances that undergo the process of phase change in accordance with temperature. PCM absorb and release thermal energy in order to maintain sustained temperature. Organic PCM's due to their low melting point cannot be coated directly into cotton fabrics and hence PCM's need to be microencapsulated. Microcapsules are Safe, chemically stable and non-reactive. PCM's made of Melamine-formaldehyde is often used because of its good mechanical and thermal stability. Thus in the present study, PCM made of melamine-formaldehyde is prepared and encapsulated into fabrics. Morphology of the microcapsules and microcapsules treated fabrics were observed under Scanning Electron Microscopy (SEM). FTIR analysis of control cotton and PCM coated cotton fabric were compared, thermal property was analyzed using DSC and wash durability of microcapsule treated fabric was evaluated. Most of the microcapsules were observed to be attachedwith the fibre structures. Microcapsules withstanded the wash durability test and the melting point of the obtained PCM calculated using Differential Scanning Calorimeter (DSC) was found to be 113ºC. This indicates that Melamine-formaldehyde microcapsules can act as an effective PCM when treated with fabrics. Thus the developed fabrics encapsulated with Phase change materials (Melamine-Formaldehyde), due to its thermal properties can be considered as a novel product in textile industry.*

Keywords: *Clothing, Phase change material, melamine, Formaldehyde, Encapsulation*

Comfort is a state of feeling pleasurable, well-being and satisfaction. Most of the people are unaware of wear comfort on textiles and clothing. Wear comfort is attained when people feel psychological and physiological satisfaction. Wear comfort is grouped into four main categories: thermo physiological comforts, psychological comfort, garment fit and sensorial comfort (O'Mahony and Braddock, 2002). On account of clothing, there should be an effective exchange of moisture and heat from body to environment. Thermal or heat factor affects the wear comfort level. Therefore, it is necessary to develop fabric which stores/release thermal energy and can adjust and maintain comfort level as circumstances change (Jun et al., 2009).

Phase Change Materials (PCM) is the substances that undergo the process of change of phases in accordance with temperature. Heat is released when they are changed to solid state and heat is absorbed when they are converted to liquid state(Vijayaraaghavan andGopalakrishnan, 2006). In order to maintain a sustained temperature, PCM absorb and release thermal energy. PCM absorbs thermal energy when the mean skin temperature is low (33.3°C). Thermal energy storage (TES) is the temporary storage of high or low temperature which bridges the time gap between energy requirements and energy use. PCM's due to its heat storage abilities used in Cold energy battery, Spacecraft thermal systems, Thermal protection of electronic devices, Computer coolingetc (Hale et al., 1971).

PCM's are of different types and about 500 PCMs are known till date. Organic PCM have different phase change (Tm)and crystallization (Tc) temperatures based on the number of carbon atoms present in their structures. Coating, lamination, finishing, melt spinning and bi-component synthetic fiber extrusion are some of the significant processes for impregnation of PCMs' into the fiber. Organic PCM's due to their low melting point cannot be coated directly into textile fabrics and hence PCM's need to be microencapsulated. Microencapsulation was found be effective due to their stability and durability. Microcapsules are Safe, chemically stable and non-reactive (Shin et al., 2005).

Microencapsulation is the process of impregnation of microcapsules which contains PCM into the fabrics which are durable and safe (Kondo and Valkenburg, 1979). Fabrics containing microcapsules of PCM's must absorb, store, release and maintain the thermal property which should sustain repeated washing.Microcapsules can be produced by two methods: physical and chemical. The use of some techniques has been limited by the high cost of processing,regulatory affairs, and the use of organic solvents, which are a concern for health and theenvironment. Physical methods are mainly spray drying or centrifugal and fluidized bedprocesses, which are inherently incapable of producing microcapsules smaller than 100μm (Mondal, 2008). PCM's made of Melamine-formaldehyde is often used because of its good mechanical and thermal stability. Melamine-formaldehyde microcapsules can be prepared by the in situ polymerization process (Sun, 2001).

Thus the main objective of the present study is to prepare an organic PCM made of melamine-formaldehyde and encapsulated into fabrics. Morphology of the microcapsules and microcapsules treated fabrics were observed under Scanning Electron Microscopy (SEM). FTIR analysis, thermal properties and wash durability of microcapsule treated fabric was evaluated.

Method

Preparation of the microcapsules

This method was carried out as described by Shin and Dong (2008). Melamine (0.1*M*) 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 h to prepare a melamine–formaldehyde prepolymer. About 0.001 *M PVA solutions*were poured into the prepolymer solution, 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%.

Characterization of the microcapsules

Infrared spectra of melamine–formaldehyde prepolymer, and the microcapsules were obtained with a Fourier transform infrared spectrophotometer. Scanning electron microscopy (SEM) was performed with a platinum coating. A differential scanning calorimetry (DSC) instrument was used to measure some thermal properties. The microcapsules were heated and cooled at a rate of 2°C/min in the range of 10–50°C under an N2 atmosphere.

Addition of the microcapsules to the fabrics

The fabric samples were impregnated with an aqueoussolution 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 method10 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 samples were washed and dried for further evaluation.

Evaluation of the microcapsule-treated fabrics

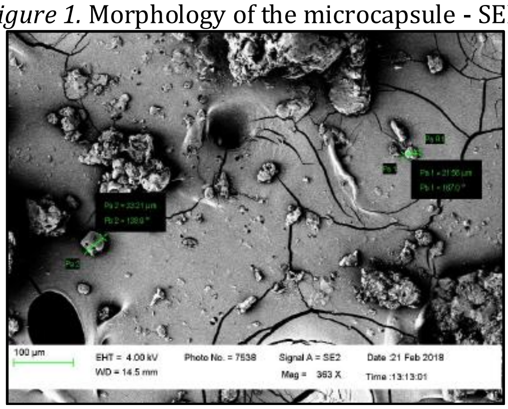
SEM and DSC analysis were performed on microcapsule treated fabrics. The melting point of PCM is calculated by Differential Scanning Calorimeter (DSC). The wash durability of the treated fabrics is evaluated by observation of capsules under SEMafter second and fifth wash.

Results and Discussion

Morphology of the microcapsule

The prepared microcapsules were observed under Scanning Electron Microscopy. Most of the microcapsules were spheres with a smooth surface morphology. Figure 1 illustrates the observation of microcapsules under SEM.

Figure 1. Morphology of the microcapsule - SEM



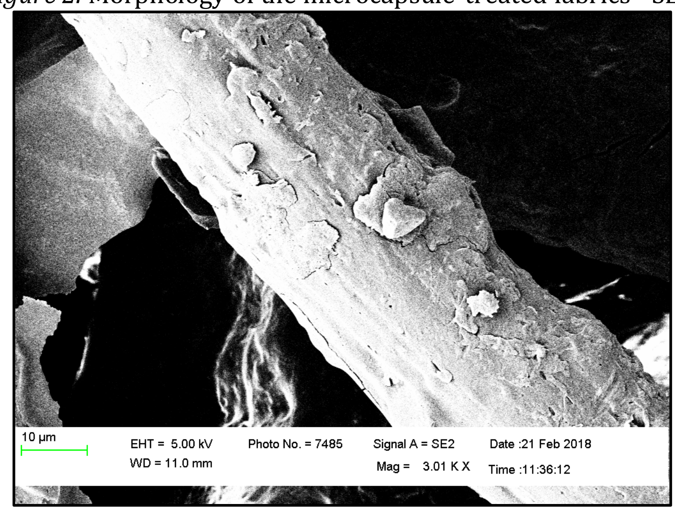
The variation in structure of microcapsules under different pH was examined by Wang and Zhao(2017). At the pH of 4.6 most of the microcapsules of regular spherical shape and smooth surface were obtained and that the microcapsules had a diameter of about 4 mm. At the pH of 5.1 only a few globular microcapsules was observed and most of the products were broken hemispheric fragments with a little polymer piled between them.

Effect of the shearing rate during the emulsion on the particle size and its distribution was investigated by Salaun and Devaux (2005). The increase in speed of emulsification resulted in decrease of smooth surface. The surface of the particles from the 1000 rpm experiment seemed smooth and less permeable, whereas the surfaces of the others were coarse with dimples, probably linked by the contracting volume of the crystallized PCMs during the microcapsules recovering.

Morphology of the microcapsule-treated fabrics

Figure 2 represents morphology of microcapsule-treated fabric. The microcapsules in the microcapsule treated samples were found attached on the fiber surface. Those microcapsules were heat-resistant and could endure the curing conditions (at 130°C for 10 min). Thus these microcapsules can be used in finishing process at high temperatures.

Figure 2. Morphology of the microcapsule-treated fabrics - SEM

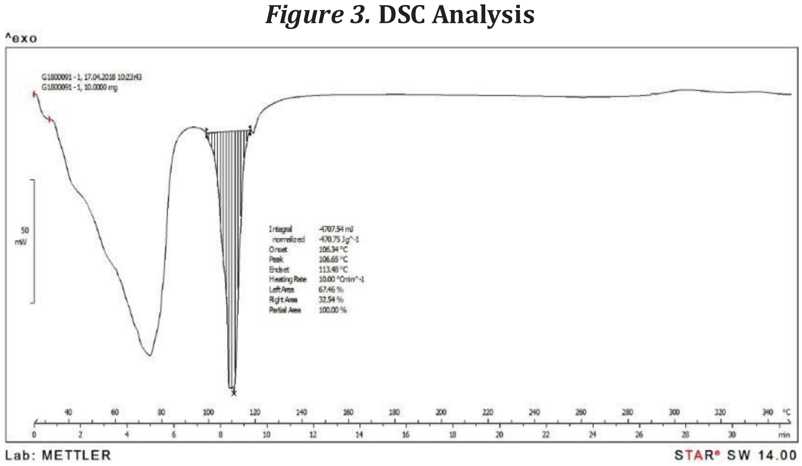


Polyurea microcapsules manufactured by interfacial polymerization were melted at curing temperatures higher than 80°C (Kim and Cho, 2002). On the other hand, more binder was observed in the microcapsuletreated samples than in the sample treated with the binder only. Some cracks were observed on the surface of the microcapsule-treated sample with 23% addition, which had the highest addition of the samples.

Thermal properties of the microcapsule-treated Fabrics

The melting point of the obtained PCM calculated using Differential Scanning Calorimeter (DSC) was found to be 113ºC. This indicates that Melamine-Formaldehyde microcapsules can act as an effective PCM when impregnated with fabrics. Figure 3 presents a DSC thermogram of the microcapsules.

Figure 3. **DSC Analysis**



Bhatkhande (2011)experimented in the situ polymerization of urea-formaldehyde resins encapsulating the core PEG material. The phase change temperature (Tm) of the microcapsules was found to be around 21ºC. The heat storage capacity of the microcapsule was 12.78J/g. Near the Recrystallization of the PCM material was found 12ºC, thus releasing heat to the fabric.

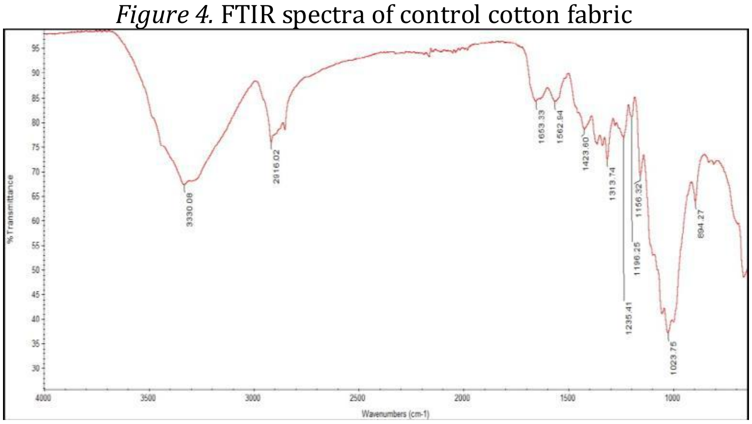
Similarly, Melamine–formaldehyde microcapsules containing eicosane were prepared by Shinet al. (2005). They observed the thermal properties i.e. heat storage capacity, phase change temperature (Tm) and crystallization temperature (Tc). The phase-change temperatures, *Tm* and *Tc*, of the microcapsules were 36.9 and 31.7°C, respectively. The heat storage capacity of the microcapsules was 134.3 J/g. The treated fabrics retained about 40% of their heat storage capacity after five launderings.

When the PCM microcapsules are heated, they absorb energy and go from a solid state to a liquid state. This phase change produces a temporary cooling effect in the clothing layer. If the PCM microcapsules are cooled down below the freezing point of PCM material, the material will change back to solid state from the liquid state, releasing heat and thus developing a temporary warming effect. This phenomenon is helpful to the wearer of the fabric in winter and summer because it provides him body comfort.

FTIR analysis

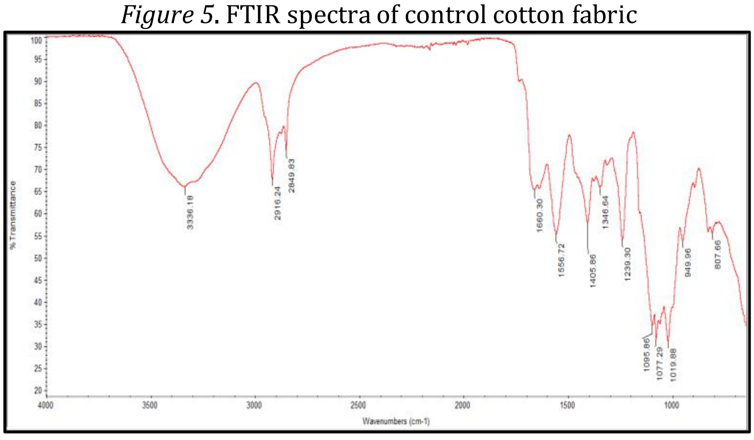
Figure 4 shows FTIR spectra of the control cotton fabric. The alteration in the functional groups of plain cotton and PCM coated cotton fabric were determined chemically using FTIR spectroscopy. FTIR spectra of cotton showed absorption bands denoting various functional groups. Absorption band at 2916cm^{-1} and 1423cm^{-1} denotes aliphatic -CH_2 group and band at 1653cm^{-1} denotes absorbed water and hydrogen bond. A strong band appeared at 1313cm^{-1}attained –OH in plan bending. C-O-C- asymmetric bridge stretching was observed at 1156cm^{-1} and another band representing C-O stretching was found to be 1023cm^{-1}.

Figure 4. FTIR spectra of control cotton fabric



PCM coated cotton fabrics were compared with control cotton fabric using FTIR spectra. The obtained FTIR spectra for PCM coated cotton fabric showed various absorption bands denoting the same functional groups attained on control cotton except few functional groups which are significantly corresponding to the PCM's. Absorption bands at 3336cm^{-1} and 1556cm^{-1}attained –N-H stretching. Strong band appeared at 1095cm^{-1} denoted C-O stretching respectively. Such bands confirmpresence of microcapsules which acts as a shell and core materials. FTIR spectra of PCM coated cotton fabric was presented in Figure 5.

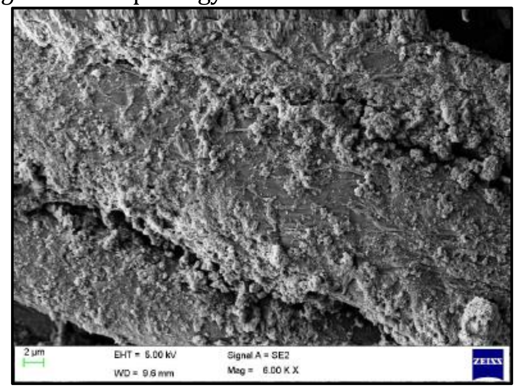
Figure 5. FTIR spectra of control cotton fabric



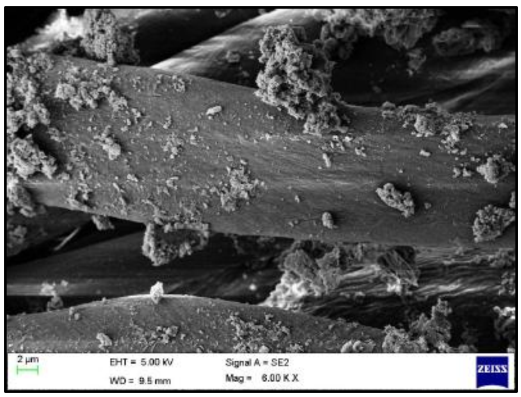
Morphology of the washed fabrics - SEM

Figure 6 shows SEM images of the samples before and after 2nd and 5th wash. Microcapsules were found to be attached to the surface of the fibre. The microcapsules were found to be slightly reduced in 5th wash on comparing with 2nd wash. This was due to the loose attachment of microcapsules on the fibre. Kim and Cho (2002) 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 ontreated fabrics. Also, mild washing conditions would be helpful for better maintenance of microcapsuletreated materials.

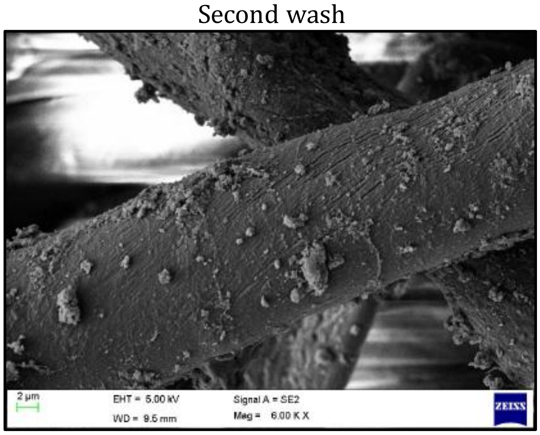
Figure 6. Morphology of the washed fabrics - SEM



Before wash



Second wash



Fifth wash

Conclusion

The prepared microcapsules were spherical and had a melamine–formaldehyde were coated on cotton fabrics. Microcapsules withstanded the wash durability test and the thermo regulating fabrics developed in this study showed significant thermal properties. The melting point of the obtained PCM calculated using Differential Scanning Calorimeter (DSC) was found to be 113ºC. This indicates that Melamine – formaldehyde microcapsules can act as an effective PCM when impregnated with fabrics. Thus the developed fabrics encapsulated with Phase change materials (Melamine-Formaldehyde), due to its thermal properties can be considered as a novel product in textile industry.

References

1. Bhatkhande, Prasad S(2011). Development of Thermo-Regulating Fabric Using Phase Change Material (PCM).Master's Theses and Doctoral Dissertations, 361.
2. Fabien Salaün1 and Eric Devaux (2005). Development of Phase Change Materials in Clothing Part I: Formulation of Microencapsulated Phase Change. Textile Research Journal,80(3), 195–205.
3. Hale DV, Hoover MJ, O'Neill MJ. Phase Change Materials Handbook, NASA Contractor Report NASA CR-61365, NASA Marshall Space Flight Center, Alabama, 1971.
4. Jun Y, Park C, Shim H, Kang T (2009). Thermal comfort properties of wearing caps from various textiles. Tex Res J, 79(2), 179-189.
5. Kim, J.; Cho, G (2002).Textile Research Journal, 72, 1093.
6. Kondo A, Valkenburg JWV (1979). Microcapsule Processing and Technology,Marcel Dekker, Inc., NY.
7. Mondal S (2008). Phase change materials for smart textiles – An overview. AppliedThermal Engineering, 28, 1536–1550.
8. O'Mahony, M and Braddock, S (2002).Sportstech : revolutionary fabrics, fashion and design. London: Thames & Hudson.
9. Shin Y, Yoo D, Son K (2005). Development of thermoregulating textile materials with microencapsulated phase change materials (pcm). iv. performance properties and hand of fabrics treated with pcm microcapsules. Journal of Applied Polymer Science, 97: 910-915.
10. Sun G, Zhang Z (2001). Mechanical properties of melamineformaldehyde microcapsules. Journal of Microencapsulation, 18, 593–602.
11. Vijayaraaghavan N, Gopalakrishnan D (2006). Phase change materials for application in textiles. Synthetic Fiber, 11-16.
12. Xianfeng Wang and Tao Zhao (2017). Effects of parameters of the shell formation process on the performance of microencapsulated phase change materials based on melamine-formaldehyde. Textile Research Journal, 87(15), 1848–1859.
13. Younsook Shin, Dong-Il Yoo, Kyunghee Son (2010). Development of Thermoregulating Textile Materials withMicroencapsulated Phase Change Materials (PCM). II.Preparation and Application of PCM Microcapsules. Journal of Applied Polymer Science2005; 96.