THERMO-REGULATORY PROPERTIES OF WOOL FABRICS BY MICROENCAPSULATED PCM

Mayur Basuk¹ and Girish Kherdekar²

¹,² Assistant Director, Wool Research Association, P.O.Sandoz Baugh, Kolshet Road, Thane- 400607

Abstract— Latent heat storage is one of the most efficient ways of storing thermal energy. There are large numbers of phase change materials that melt and solidify at a wide range of temperatures, making them attractive in a number of applications. In the present paper, Melamine–formaldehyde microcapsules containing eicosane were prepared by in situ polymerization. The characterization of the microcapsules, including the particle size and size distribution, morphology, projection microscopy, thermal properties, was carried out. The prepared microcapsules were added to Indian wool and wool blebbed fabrics by a conventional pad–dry–cure process to develop thermo regulating textile materials. The morphology and thermal properties of the treated fabrics were also investigated. The prepared microcapsules were spherical and had a melamine–formaldehyde resin shell containing Hexadecane, Octadecane, Butyl Stearate & Eicosane. The heat storage capacity increased as the concentration of the microcapsules increased. The thermo regulating fabrics had shown increase in the thermal resistance value

Keywords—Microencapsulation, Phase Change Material, Wool, Blended Fabrics

I. INTRODUCTION

Our human body is a thermo-regulated organism. The body constantly generates heat, CO₂ and H₂O by the metabolism of food and muscle activity. The human body controls the release speed of heat by blood vessel dilatation temperature. It has been shown that the most comfortable skin temperature is 31.4°C. When the temperature of any part of the skin differs by 1.5-3.0°C with respect to ideal temperature, the human body is unaware of the warmth or coolness [1,2]. If the difference is more than ±4.5°C, the human body feels discomfort. In addition, a core body temperature of 36.5°C is required, and a rise or fall of 1.5°C can be fatal. A balance between the rates of heat loss and heat generation must be maintained [3].

Microencapsulation technology was utilised in the early 1980s by the US National Aeronautics and Space Administration (NASA) with the aim of managing the thermal barrier properties of garments, in particular for use in space suits. They encapsulated phase-change materials (PCMs) with the hope of reducing the impact of extreme variations in temperature encountered by astronauts during their missions in space. Ultimately the technology was not taken up within the space programme [4,5].

The basic concept of phase change material Phase change process of PCM from solid to liquid and vice versa. During the complete melting process, the temperature of the PCM as well as its surrounding area remains nearly constant. The same is true for the crystallisation process; during the entire crystallisation process the temperature of the PCM does not change significantly either. The large heat transfer during the melting process as well as the crystallization process without significant temperature change makes PCM interesting as a source of heat storage material in practical applications. When temperature increases, the PCM microcapsules absorbed heat and storing this energy in the liquefied phase change materials. When the temperature falls, the PCM microcapsules release this stored heat energy and consequently PCM solidify [6,7].
II. EXPERIMENTAL

2.1 Materials

Melamine and 37% formaldehyde as shell materials, Butyl Stearete as a core material, Sodium Dodecyl Sulphate (SDS) 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 fabric were scoured and washed. 100% Indian wool, Wool/polyester blended woven fabric as well as polyester knitted fabrics were used for the experiments. The details are given in Table 1.

2.2 Preparation of the microcapsules

Method used

In this method, the Butyl Stearete (PCM) emulsion was prepared first. Distilled water (200 ml) was taken in a beaker & the fine powder of Sodium Dodecyl Sulphate (SDS) was added to this as an emulsifier by proper mixing. To this solution, Butyl Stearate was added slowly over 30 min while stirring the mixture vigorously using a high shear mechanical stirrer at 2200 rpm. The PVA (water soluble form) as a protective colloid was added to the mixture & the stirring continued for an additional 30 min.

The melamine Formaldehyde (MF) pre polymer was prepared separately by adding the calculated amount of formaldehyde & melamine in a beaker containing distilled water. The details of proportion of these materials for making the solutions are given as below. Before heating the solution, the pH of the mixture was adjusted to 8.5 -9.0 using 10% solution of sodium carbonate. The temperature was raised to 70°C while continuously stirring the mixture using a magnetic stirrer. The mixture becomes transparent, indicating the formation of MF pre-polymer. The MF pre polymer, thus obtained, was added slowly into the prepared PCM emulsion & used for further process of encapsulation.

Parameters used in microcapsules preparation

- Melamine – 5.46 g (1 Mole), L.R grade
- Formaldehyde – 11.67 g (3.5 Mole), L.R grade
- Sodium Dodecyl Sulphate (SDS) – 7.5 g, L.R grade
- Poly vinyl alcohol (PVA) Cold (Molecular weight 10000) – 1.25 g, L.R grade
- Butyl Stearete PCM – 20 g, L.R grade
- Core to Wall ratio – 2, L.R grade

Microencapsulation process

To facilitate the mechanical stirrer from the reaction emulsion mixture was replaced with a magnetic stirrer. The pH of the system was slowly reduced to 3.0 using 5% solution of sulphuric acid, while the temperature was raised slowly to 70°C. These conditions were maintained for an additional two hours for the formation of capsules. Finally, the Capsules were cooled down to room temperature, filtered, washed with distilled water at room temperature & dried at 40°C in an air oven for 15 hour or at 100°C for 90 min. The Figure 1 shows the experimental set up for microencapsulation of PCM.

![Figure 1. Experimental set up for microencapsulation of PCM](image)
2.3 Characterization of the microcapsules

The mean particle size and size distribution of the microcapsules were determined with particle size analyzer (Beckman Coulter LS model Particle Size Analyzer). Projection Microscope (Heerbruug, Switzerland) images was used to check the formation of capsules. A differential scanning calorimetry (DSC) instrument (DSC3, Mettler Toledo GmbH, Switzerland) was used to measure some thermal properties. The microcapsules were heated and cooled at a rate of 5°C/min in the range of 0–100°C under an N₂ atmosphere.

2.4 Fabric development

Both Woven and knitted fabrics were developed for application of phase change material. the details as as mentioned in the Table1.

2.5 Addition of the microcapsules to the fabrics

The water was taken in the ratio of 10:1 on weight of Fabric. Slow addition of polyquot type TPS GL 502 binder (Obtained from M/s Tanishka Products Ltd.,Mumbai) to water under vigorous stirring was done and mixed properly. Subsequently, pH of water was reduced to 4 to 4.5 by using acetic acid and addition of Micro-Capsule was done to this solution slowly under vigorous stirring and mixed properly. The dosage of Microcapsule was decided based on net pickup of liquor. The solution was mixed thoroughly to ensure that the solution is free from flocculation and the Microcapsule slurry is evenly distributed. The ratio of microcapsules and binder was kept as 1:1.

Application was done using Pad – dry – cure process. 2 dip 2 nip process for better penetration and maintain 70-80% pick up. The concentration of prepared microcapsules was taken 100 gpl, 50 gpl & 30 gpl respectively.

After squeezing the fabric in the padding mangle and ensuring the net pickup of water, dry the fabric at 105° C and curing at 120 ºC in a stenter for 3 – 4 min. Time duration of drying and curing has to be set based on fabric weight and speed through the stenter. The fabric should be completely dry through this process to ensure even distribution and adhesion of capsules to the fabric surface. The treated samples were washed and dried for further evaluation.

Table 1. Details of Samples Developed

| S. no. | Fabric code | GSM  | Thickness (in mm) | Weave         | Warp count Nm | Weft count Nm | EPI | PPI | Blend%        |
|--------|-------------|------|-------------------|---------------|---------------|---------------|-----|-----|---------------|
| 1      | A           | 164.59 | 0.40              | Twill         | 2/47.0        | 2/46.8        | 53  | 41  | 100 % Wool   |
| 2      | B           | 139.26 | 0.30              | Twill         | 2/72.0        | 2/42.5        | 71  | 43  | 100% Wool    |
| 3      | C           | 156.8  | 0.40              | Twill         | 2/48.37       | 2/49.8        | 55  | 40  | 100% Wool    |
| 4      | D           | 121.6  | 0.25              | Twill         | 2/76.0        | 2/44.9        | 65  | 48  | 100% Wool    |
| 5      | E           | 178.6  | 0.45              | Twill         | 2/70.9        | 2/63.7        | 82  | 66  | 45/55 Wool/Polyester |
| 6      | F           | 235.8  | 0.50              | Twill         | 2/56.5        | 2/54.0        | 93  | 70  | 35/65 Wool/Polyester |
| 7      | G           | 254.7  | 0.55              | Twill         | 2/18.8        | 2/19.3        | 34  | 27  | 50/50 Wool/Polyester |
| 8      | H           | 114.0  | 0.30              | Single jersey knitted | 84 D / 48 filament | 14  | 14  | 100% Polyester |}

| 9      | I           | 298.0  | 0.70              | Double jersey knitted | 84 D / 48 filament | 14  | 18  | 100% Polyester |
III. RESULTS AND DISCUSSION

3.1 Particle Size Analysis

Figure 2 shows the distribution of particle size of Micro encapsulated phase change material. It can be seen that, maximum particles lie 0.1 µm to 50 µm and the average particle size of m-encapsulated Hexadecane, Octadecane, Butyl Stearate & Eicosane determined by Gaussian Intensity was found to be 6.181 µm, 6.042 µm, 10.81 µm & 30.32 µm respectively. The standard deviation was also found towards lower side indicating the uniform particle size of the particles.

![Particle Size Analysis](image1.png)

3.2 Projection Microscope Test

All the micro encapsulated PCMs were tested for microscope analysis. Projection Microscope (Heerbruug, Switzerland) images were captured at 500x magnification. All the images clearly shows the core and sheath part of the capsules and this confirms the formation of micro capsules.

![Microscope Images](image2.png)
Figure 3. (A) Microscopic view of m-encapsulated HEXADECANE, (D) Microscopic view of m-encapsulated OCTADECANE, (F) Microscopic view of m-encapsulated Butyl Stearate, (H) Microscopic view of m-encapsulated Eiscosane

3.3 Differential Scanning Calorimeter

Differential Scanning Calorimeter (DSC) analysis is used to measure the latent heat storage capacity of the materials. The prepared microcapsules were also tested for DSC analysis and it was found that latent heat of microcapsules containing Hexadecane, Octadecane, Butyl Stearate & Eicosane was 80.54 J/g at 19.08 °C, 120.72 J/g at 28.88 °C, 38.37 J/g at 19.53 °C and 217.26 J/g at 37.58 °C respectively. Hence, microcapsules of eicosane give maximum latent heat for 37.58 °C temperature. Figure 4 shows the graph of the prepared microcapsules tested on Differential Scanning Calorimeter (DSC).

![DSC Graphs](A B C)
3.5 Test results of Scanning Electron Microscope

The FeSEM test was carried out for prepared microcapsules; The SEM images were captured at different magnification of 10.0 KX, 25.0 KX, 50.0 KX and 100.0 KX.

In the Figure 5 of SEM images of prepared microcapsules shows an optical micrograph of the dried microcapsules of slurry state that means an 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.
3.5 Thermal Resistance Test

The test was performed according to test Standard ASTM D 1518-14 using dry guarded hot plate (MTNW Coroporation, USA). The test covers the measurement of the thermal resistance under steady state conditions.

The thermal resistance of the untreated fabric lower as compared to the tested fabrics in all the cases. This implies that the thermal resistance of the fabric was more when the guarded hot plate temperature was increased above the melting temperature of the phase change material. The test results are as mentioned in the Table 2, 3 & 4. The tables illustrates the changes due to the increase of thermal resistance values in the treated fabric as compared to the untreated fabrics.

Table 2. Thermal Resistance  at 100 gpl microcapsules concentration

| S. no. | Fabric code | TOG UT | TOG m – Eicosane | TOG m – Hexadecane | TOG m – Butyl Stearate | TOG m – Octadecane |
|--------|-------------|--------|------------------|---------------------|------------------------|-------------------|
| 1      | A           | 0.311  | 0.562            | 0.574               | 0.535                  | 0.579             |
| 2      | B           | 0.262  | 0.515            | 0.515               | 0.445                  | 0.452             |
| 3      | C           | 0.311  | 0.542            | 0.569               | 0.522                  | 0.561             |
| 4      | D           | 0.275  | 0.511            | 0.495               | 0.435                  | 0.458             |
| 5      | E           | 0.162  | 0.306            | 0.273               | 0.350                  | 0.447             |
| 6      | F           | 0.270  | 0.396            | 0.359               | 0.421                  | 0.251             |
| 7      | G           | 0.454  | 0.605            | 0.602               | 0.698                  | 0.654             |
| 8      | H           | 0.251  | 0.351            | 0.302               | 0.256                  | 0.295             |
| 9      | I           | 0.290  | 0.383            | 0.342               | 0.364                  | 0.388             |

Table 3. Thermal Resistance  at 50 gpl microcapsules concentration

| S. no. | Fabric code | TOG UT | TOG m – Eicosane | TOG m – Hexadecane | TOG m – Butyl Stearate | TOG m – Octadecane |
|--------|-------------|--------|------------------|---------------------|------------------------|-------------------|
| 1      | A           | 0.311  | 0.438            | 0.448               | 0.417                  | 0.452             |
| 2      | B           | 0.262  | 0.402            | 0.402               | 0.347                  | 0.353             |
| 3      | C           | 0.311  | 0.423            | 0.444               | 0.407                  | 0.438             |
| 4      | D           | 0.275  | 0.399            | 0.386               | 0.339                  | 0.357             |
| 5      | E           | 0.162  | 0.239            | 0.213               | 0.273                  | 0.349             |
| 6      | F           | 0.270  | 0.309            | 0.280               | 0.328                  | 0.196             |
| 7      | G           | 0.454  | 0.472            | 0.470               | 0.544                  | 0.510             |
| 8      | H           | 0.251  | 0.316            | 0.336               | 0.300                  | 0.330             |
| 9      | I           | 0.290  | 0.329            | 0.367               | 0.384                  | 0.323             |
Table 4. Thermal Resistance 30 gpl microcaps concentration

| S. no. | Fabric code | TOG UT | TOG m – Eicosane | TOG m – Hexadecane | TOG m – Butyl Stearate | TOG m – Octadecane |
|--------|-------------|--------|------------------|-------------------|------------------------|-------------------|
| 1      | A           | 0.311  | 0.326            | 0.333             | 0.320                  | 0.336             |
| 2      | B           | 0.262  | 0.299            | 0.279             | 0.258                  | 0.282             |
| 3      | C           | 0.311  | 0.324            | 0.350             | 0.323                  | 0.355             |
| 4      | D           | 0.275  | 0.296            | 0.287             | 0.252                  | 0.266             |
| 5      | E           | 0.162  | 0.177            | 0.158             | 0.203                  | 0.259             |
| 6      | F           | 0.270  | 0.230            | 0.208             | 0.244                  | 0.146             |
| 7      | G           | 0.454  | 0.351            | 0.349             | 0.405                  | 0.379             |
| 8      | H           | 0.251  | 0.275            | 0.295             | 0.268                  | 0.271             |
| 9      | I           | 0.290  | 0.299            | 0.298             | 0.309                  | 0.315             |

IV. CONCLUSION

The prepared microcapsules were spherical and had a melamine–formaldehyde resin shell containing Hexadecane, Octadecane, Butyl Stearate & Eicosane. All the prepared microcapsules were showing excellent amount of the latent heat during their transition temperature. The treated fabrics shows significant improvement in thermal resistance values as compared to the untreated fabrics. These fabric have shown thermal regulating properties above and below the transition temperature.

REFERENCES

[1] Xiaoming Tao, “Smart fibers. Fabrics and clothing”, Wood head Publishing Limited, Cambridge, 2001, 34-57
[2] Dr Heikki Mattila, “Intelligent textiles and clothing”, Wood head Publishing Limited, Cambridge, 2006, 19-82
[3] M. Parthiban, S. Riswanth Kumar, K. Santhosh Kumar, K. Senthil Kumar, PCM – Manufacture and Applications in the Field of Textiles, http://www.fibre2fashion.com
[4] Gokhan Erkan; Enhancing The Thermal Properties of Textiles With Phase Change Materials; RJTA Vol. 8 No. 2 2004, 57-64.
[5] Sánchez, Paula, et al. “Development of thermo-regulating textiles using paraffin wax microcapsules.” Thermochimica Acta 498.1 (2010): 16-21.
[6] Gordon Nelson, Application of microencapsulation in textiles, International Journal of Pharmaceutics 242 (2002) 55–62.
[7] S. Mondal, “Phase changing materials for smart textiles”, Applied Thermal Engineering, August 2007, p.no.1536 – 1550.