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Research Article

FUNCTIONAL FINISHING OF MICROENCAPSULATED INORGANIC PHASE CHANGE MATERIAL ON COTTON TEXTILES

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ABSTRACT

PCM is responsible for the storage and adsorption or release of thermal energy, the encapsulation of them needs a physically and chemically stable shell. The development of an easy, cheap and robust method for the encapsulation of PCMs is so important for textile applications. Inorganic microcapsules (Urea-formaldehyde) were synthesized and capsules were finished onto cotton fabrics to develop into a novel phase change material. Morphology of the microcapsules finished fabrics were analyzed topographically using Scanning Electron Microscopy (SEM). FTIR analysis, thermal properties and wash durability of inorganic PCM capsule finished fabrics were separately characterized. Under Scanning Electron Microscopy, the prepared inorganic PCM microcapsules were found to be spherical shape with smooth surface. The Inorganic PCM microcapsules in the fabrics were found attached on the fiber surface. The melting point of the PCM was found to be 124.9ºC. This confirms that the obtained inorganic PCM can be effective when impregnated with fabrics. 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. Microcapsules retained in the fabric even after 5 washes. The microcapsules were found to be slightly reduced at 5th wash than 2nd wash. As a result of its significant thermal properties, the developed inorganic PCM can be employed in textile materials with medical applications for hot and cold therapies or in high-tech clothes for extreme weather conditions.

Keywords: Inorganic PCM, Urea, formaldehyde, SEM, DSC, FTIR

INTRODUCTION

The microencapsulation of phase change materials (PCMs) to be employed in textile materials with medical applications for hot and cold therapies or in high-tech clothes for extreme weather conditions is a great challenge for technicians and scientists. Up to now, some attempts to develop a cheap and technically easy process for the encapsulation of paraffin waxes have been done. The most common methods used are interfacial polymerization[1], emulsion polymerization[2], in situ polymerization[3], spray drying and coacervation[4]. Although the PCM is responsible for the storage and adsorption or release of thermal energy, the encapsulation of them needs a physically and chemically stable shell. This is the main technical and scientific problem. For that reason, the development of an easy, cheap and robust method for the encapsulation of PCMs is so important for textile applications.

The PCMs have to be materials with high heats of fusion. They can absorb or release the latent heat when the temperature of the material undergoes or overpass the temperature of phase change. A variety of PCMs available are well known for their thermal characteristics; these materials exist in the market (hydrated salts, paraffins or waxes, organic, inorganic and fatty acids), and they can be encapsulated by a polymer cover[5]. The election of the appropriate material depends on the final application the materials that melt below 5°C are used to impart warmth to a user's skin under cold weather conditions, while the materials that melt around 30°C would be suitable to provide some sensation of freshness from the clothes.

Some conventional encapsulation methods like complex coacervation create the "shell" of the microcapsule by precipitating the polymer shell from the continuous phase, in an out-inside process. Another possible method to obtain microcapsules with a polymer cover and a PCM core would be to carry out a suspension-like polymerization process. This process generally involves the dispersion of the monomer, mainly as a liquid in small droplets, into an agitated stabilizing medium usually consisting of water containing small amounts of suspension and dispersion agents. In the present study, one such method was involved to synthesize paraffin wax as inorganic oil phase covered with PVA (polymer) capsules[6].

The synthesized capsules were finished onto cotton fabrics to develop into a novel phase change material. Morphology of the microcapsules finished fabrics were analyzed topographically using Scanning Electron Microscopy (SEM). FTIR analysis, thermal properties and wash durability of inorganic PCM capsule finished fabrics were separately characterized.

MATERIALS AND METHODS

Preparation of the microcapsules[7]

Urea (0.1*M*) and 0.3*M* 37% formaldehyde in 100mL 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 urea–formaldehyde prepolymer. An oil-in-water emulsion of paraffin

wax (10g) in 100mL of a 1% SLS aqueous solution was prepared via stirring at a speed of 6000 rpm (prepolymer). The prepared emulsion was added to the prepolymer 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/ prepolymer system, and the mixture was stirred at 50°C for 1h 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 urea–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 N_2 atmosphere.

Addition of the microcapsules to the fabrics[8]

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 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 SEM after second and fifth wash.

RESULTS AND DISCUSSION

Synthesis of microcapsules

In the present study, inorganic PCM were synthesized as microcapsules using an *in-situ* polymerization process. The process aids in developing paraffin wax as inorganic microcapsules as core material covered with PVA polymers. The method was found to be well in-accordance with the polymerization process reported by Luz Sanchez et al[6] (2007).

The researchers reported that the process generally involves the dispersion of the monomer, mainly as a liquid in small droplets, into an agitated stabilizing medium usually consisting of water containing small amounts of suspension and dispersion agents. The initiator is dissolved in the monomer–PCM mixture, and it could be chosen to control the site of the free radical generation as desired, i.e. within the droplet or in the water phase external to the droplet. Similarly, in the present study, a prepolymer solution was prepared using urea and formaldehyde.

An oil-in-water emulsion of paraffin wax was prepared with aqueous SLS solution. The prepared emulsion was added to the prepolymer to start *in situ* polymerization using the polymer PVA. PVA was poured into the emulsion/prepolymer system for the development of paraffin inside the polymer covering layer. Thus, formed PCM microcapsules, were found to own a polarity in such a way that the site of free radical generation will be the interface between water and the oil droplet, maintaining the paraffin inside the polymer.

Other researchers were also reported to develop PCM microcapsules using different core and wall materials. Ma *et al*[9]. (2003) developed a suspension-like polymerization process to encapsulate a PCM. They carried out a microencapsulation method to retain hexadecane (HD) inside a shell formed by a mixture of N, N-dimethylaminoethyl methacrylate and styrene as monomers. Jonsson *et al*[10]. (2006) prepared by suspension polymerization acrylonitrile–methacrylonitrile copolymer particles with a core/shell structure. Sundberg *et al*[11]. (1990) studied other water/polymer/polymer systems and observed that the thermodynamic properties of the particles were independent of particle size and the method of emulsion processing.

Morphology of the microcapsule

Under Scanning Electron Microscopy, the prepared inorganic PCM microcapsules were found to be spherical shape with smooth surface. The surface morphology of Inorganic PCM materials was presented in Figure 1.

Similar study was conducted by Luz *et al*[6]., 2007. The first visual appreciation in an optical microscope and after that in the ESEM (Fig. 2) indicated that spherical microspheres of PCM material were obtained with a relatively homogeneous external appearance and a wide distribution of sizes.

Morphology of the microcapsule-treated fabrics

The microcapsule treated fabric was observed under Scanning Electron Microscope (SEM). The Inorganic PCM microcapsules in the fabrics 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. Some cracks were observed on the surface of the microcapsule-treated sample (Figure 2).

Micro capsules containing eicosane were manufactured by in-situ polymerization[8]. The manufactured microcapsules mixed with a polyurethane binder were applied topically to the polyester knit fabric with a pad–dry– cure method. 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.

Thermal properties of the microcapsule-treated Fabrics

The thermal properties of Inorganic PCM were evaluated by Differential Scanning Calorimeter (DSC) and the melting point of the PCM was found to be 124.9ºC. This confirms that the obtained inorganic PCM can be effective when impregnated with fabrics. The DSC thermogram of the microcapsules was presented in Figure 3.

In a study conducted by Gao and Deng[12]., 2013, The DSC curve of PCM-B was measured by a differential scanning calorimeter at the scanning rate of 5ºC/min in a nitrogen atmosphere from 30º to 80ºC. It shows that the melting temperature of PCM-B is 8.2ºC, and the latent heat value of phase change is 154.8kJ/kg. Compared with the physical parameters of single magnesium nitrate hexahydrate, the melting point and the latent heat of phase change of PCM-B are lower than that of single magnesium nitrate hexahydrate, and the reduction amplitude of PCM-B are 40.8ºC and 8.0 kJ/kg, respectively. From the DSC thermal analysis, it can be clearly indicated that ammonium nitrate has remarkable effect on the phase transition temperature of magnesium nitrate hexahydrate and slight influence on the value of the latent heat.

FTIR analysis

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 3330.90cm^{-1} and 1636.49cm^{-1} attained –N-H stretching. Strong band appeared at 1027.64cm^{-1} denoted C-O stretching respectively. Such bands confirm presence of microcapsules which acts as a shell and core materials. FTIR spectra of PCM coated cotton fabric was presented in Figure 4.

Figure 5 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}.

Morphology of the washed fabrics - SEM

The fabric finished with PCM was subjected to washing and the durability was observed. The SEM observation was done for 2nd and 5th wash. Microcapsules retained in the fabric even after 5 washes. The microcapsules were found to be slightly reduced at 5th wash than 2nd wash (Figure 6). The mild attachment of microcapsules on fibre resulted in lowering of microcapsules after repeated washing. The usage of binder in finishing can increase the retention of microcapsules on fibres[13].

PCM undergo phase transition from solid to liquid state when heated. A temporary cooling effect is produced during the transition. Similarly heat energy will be released when PCM is cooled i.e. when they change from liquid to solid state, thus developing a temporary warming effect. This principle of PCM when applied in fabrics increases comfortness during summer and winter days.

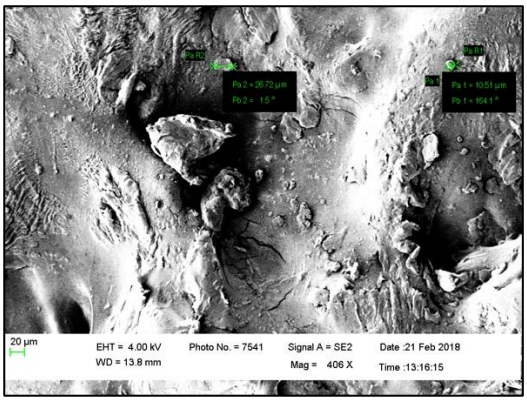


Figure 1: Morphology of the microcapsule - SEM

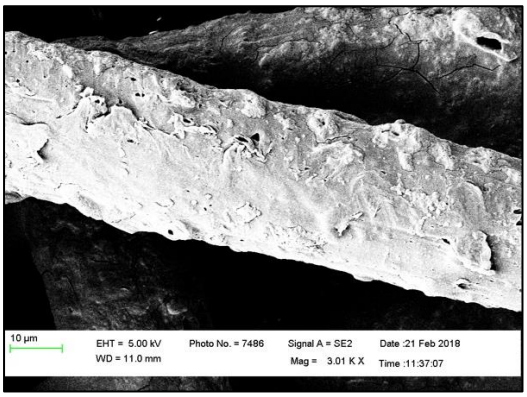


Figure 2: Morphology of the microcapsule-treated fabrics - SEM

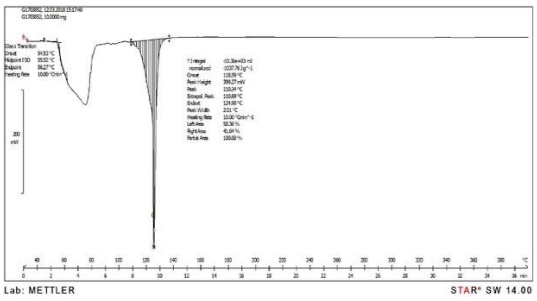


Figure 3: DSC Analysis

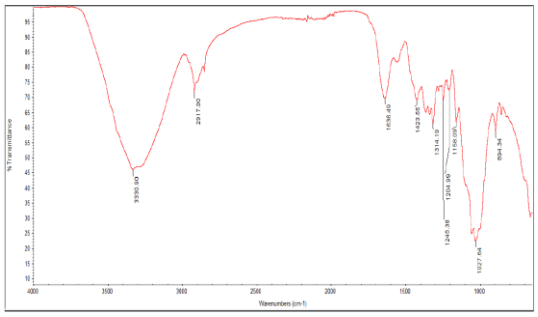


Figure 4: FTIR spectra of cotton fabric (inorganic PCM)

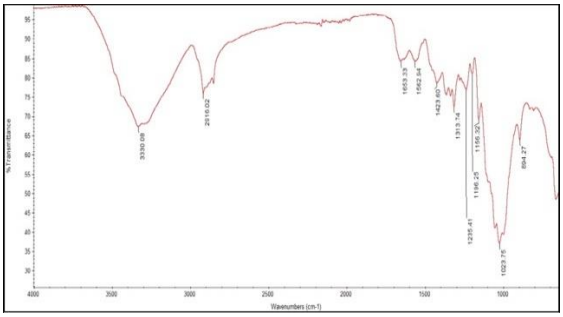
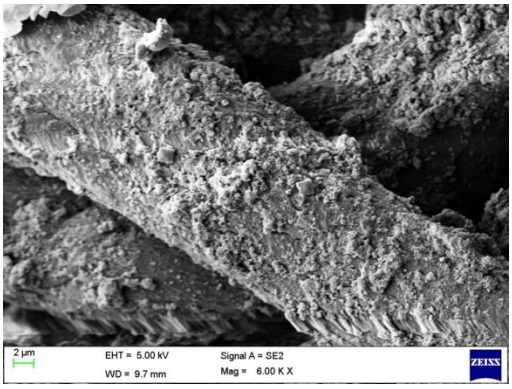
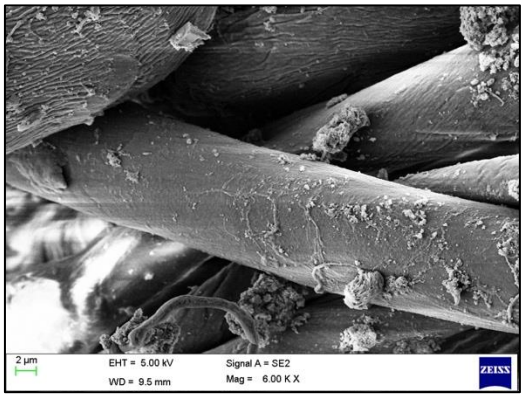


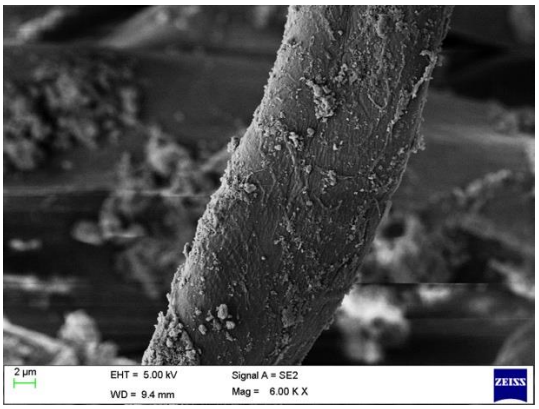
Figure 5: FTIR spectra of control cotton fabric



Before wash



Second wash



Fifth wash

Figure 6: Morphology of the washed fabrics - SEM

CONCLUSION

Inorganic PCM microcapsules (urea-formaldehyde) were prepared using an *in-situ* polymerization process and finished onto fabric. Under Scanning Electron Microscopy, the prepared inorganic PCM microcapsules were found to be spherical shape with smooth surface. The Inorganic PCM microcapsules in the fabrics were found attached on the fiber surface. The melting point of the PCM was found to be 124.9ºC. This confirms that the obtained inorganic PCM can be effective when impregnated with fabrics. 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. Microcapsules retained in the fabric even after 5 washes. The microcapsules were found to be slightly reduced at 5th wash than 2nd wash. As a result of its significant thermal properties, the developed inorganic PCM can be employed in textile materials with medical applications for hot and cold therapies or in high-tech clothes for extreme weather conditions.

REFERENCES

1. Chu L, Xie R, Zhu J, Chen W, Yamaguchi T, Nakao S (2003). 'Study of SPG membrane emulsification processes for the preparation of monodisperse core-shell microcapsules. J Colloid Interface Sci 265:187–196
2. McDonald CJ, Devon MJ (2002). Hollow latex particles: synthesis and applications. Adv Colloid Interface Sci 99:181–213
3. Yang R, Xu H, Zhang Y (2003). Preparation, physical property and thermal physical property of phase change microcapsule slurry and phase change emulsion, Sol Energy Mater Sol Cells 80:405–416

4. Hawlader MNA, Uddin MS, Khin M (2003). Microencapsulated PCM Thermal-Energy Storage System Appl Energy 74:195–202
5. Maruoka N, Akiyama T (2003). Thermal Stress Analysis of PCM Encapsulation for Heat Recovery of High Temperature Waste Heat. J Chem Eng Jpn 36:794–798.
6. Sánchez-Silva, Luz & sánchez, Paula & de Lucas, Antonio & Carmona, Manuel & Rodríguez, Juan. (2007). Microencapsulation of PCMs with a polystyrene shell. Colloid and Polymer Science. 285. 1377-1385. 10.1007/s00396-007-1696-7.
7. Rochmadi, A., A. Prasetya and W. Hasokowati, "Mechanism of Microencapsulation with Urea-Formaldehyde Polymer," *American Journal of Applied Sciences*, Vol. 7, No. 6, 2010, pp. 739-745.
8. 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.
9. Ma GH, Su ZG, Omi S, Sundberg D, Stubb J (2003). Microencapsulation of oil with poly (styrene-N, N-dimethylaminoethyl methacrylate）by SPG emulsification technique: Effects of conversion and composition of oil phase. J Colloid Interface Sci 266:282–294
10. Jonsson M, Nordin O, Malmström E, Hammer C (2006). Suspension polymerization of thermally expandable core/shell particles. Polymer 47:3315–3324.
11. Sundberg D, Casassa AP, Pantazopoulos J, Muscato MR (1990). Morphology development of polymeric microparticles in aqueous dispersions. I. Thermodynamic considerations. J Appl Polym Sci 41:1425–1442.
12. Daolin Gao and Tianlong Deng. Energy storage: Preparations and physicochemical properties of solid liquid. Phase change materials for thermal energy storage. 2013. 32- 44.
13. Kim, J.; Cho, G (2002). Thermal Storage/Release, Durability, and Temperature Sensing Properties of Thermostatic Fabrics Treated with Octadecane-Containing Microcapsules. *Textile Research Journal*, 72, 1093.

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