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Innovation in Textiles: Integration of Nanoencapsulation of PCMs in Cotton fabric

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Abstract: *Indian foundry industry stands the world's second largest producer of castings. At the same time, workers who work in uncomfortably hot environment without appropriate clothing for extreme conditions are most likely exposed to higher risks. Their prolonged exposure to high temperature causes thermal discomfort. It is largely concerned with the selection of improper clothing suitable for hot environment. This research is aimed to assess the effects of hot environment near the furnace area in foundry industry and to developing a thermal protection fabric using nanoencapsulated Phase Change Materials (PCMs) or nanocapsules. Polyethylene glycol (PEG) nanocapsules containing PEG as core material and urea-formaldehyde as shell material were successfully developed using in-situ polymerization method. Different characterization techniques were used to analyse the properties of the developed PEG nanocapsules. Scanning electron microscope (SEM) and Transmission Electron Microscope (TEM) study results indicated that the nanocapsules form a regular and spherical shape without agglomeration. The average diameter of the nanocapsules was found to be 161 nm. The size of the PEG nanocapsules was within the range of 100-300 nm. Energy Dispersive Spectroscopy (EDS) test results showed that the nanocapsules contains 38 wt.% carbon, 27 wt.%, oxygen and 35 wt.% nitrogen. Fourier transform infrared (FT-IR) spectroscopy confirmed that the PEG was successfully encapsulated within the urea-formaldehyde shell. Differential scanning calorimetry (DSC) results found that the latent heat energy storage capacity of the PEG nanocapsules was 81.1 J/g. The encapsulation ratio of the nanocapsules was 59.5%. The PEG nanocapsules showed good thermal stability, which makes the coated fabric appropriate for textile application.*

Introduction

According to the relationship between environment temperature and human thermal balance, living environments temperature above 35°C and working environments above 32°C can be considered as hot environments, and environments with relative humidity above 70% can be considered as humid (Zhao et al 2009). Textiles containing PCMs have different thermal properties from conventional textiles. Micro-or nano-capsules can be applied to a wide variety of textile substrates to improve thermo-regulation and insulation properties. Coating, lamination, finishing, melt spinning, bi-component synthetic fibre extrusion, injection moulding, foam techniques are some of the convenient processes for incorporation of PCMs into textile substrate.

Nanoencapsulation is defined as the process of enclosing nano-sized particles within solid or liquid particles. The products obtained by the process are called nanocapsules or nanoparticles. The term nanocapsule is used if the size of the particles is below 1 µm (Sari et al 2009; Ries et al 2006). Recently, nanocapsules have received considerable attention for TES systems because of their high surface area/volume ratio compared to microcapsules which results in a stronger driving force to increase thermodynamic processes. In the literature studied, some research has been carried out into the preparation of nanocapsules for TES. Zhang et al (2004) fabricated both

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microcapsules and nanocapsules containing melamine-formaldehyde and n-octadecane as the shell material and core material using in-situ polymerization. The influence of stirring rate, emulsifier content and amount of cyclohexane on the diameters, morphology, phase change and thermal stability of the capsules were studied. The results indicated that the diameter of the microcapsule decreased to 0.8 μm, forming nanocapsules, with increased stirring rate, emulsifier content and cyclohexane content. According to the DSC results, the onset temperatures and phase change enthalpy remain unchanged with increased stirring rate and emulsifier content, but had little effect on cyclohexane. Wei et al (2007) synthesized micro-and nano-encapsulated PCMs with melamine-formaldehyde as the shell and n-octadecane as the core. The results showed that the core was well encapsulated with added sodium dodecyl sulphate as an emulsifier. The results also indicated that the average diameter of the microcapsules and nanocapsules was 65 μm and 850 nm, respectively. The encapsulation efficiency of the microcapsules and nanocapsules was 87% and 45%, respectively. Fan et al (2005) synthesized nanocapsule containing melamine-formaldehyde as the shell material and, n-octadecane (91.2%) and cyclohexane (8.8%) as the core material. The thermal effects of the nanocapsules after treatment at 120 °C, 140 °C, 160 °C and 180 °C for 30 min were studied. The results showed that pH value has some effect on the stability of emulsion and morphology of nanocapsules. The results also indicated that the size of the nanocapsules ranged between 0.4 and 1.0 μm. It was concluded that the heat treatment increased the thermal stability of nanocapsules initially, then decreased when the temperature exceeded 160 °C.
Li et al (2011) successfully prepared nanocapsules with urea-formaldehyde as the shell material and hexadecane as the core material using the two-step miniemulsion polymerization method. It was observed that the nanocapsules were spherical, and the particle size was 270 nm. It was further reported that the surfactant influenced the particle diameter, coefficient of variation, thickness of the shell and radius of the core. Therefore, this paper aimed to prepare nanocapsules and coated onto the cotton fabric, and to find the thermoprotection properties of cotton fabric.

3. Methodology

The research methodology adopted in this work comprises initially assessment of hot environment, synthesis of nanocapsules containing different core material such as PEG and paraffin wax using in-situ polymerization method.

4. Development of the PEG nanocapsules

PEG nanocapsules were prepared using in-situ polymerization method. This method involves two processes: preparation of emulsifier and pre-polymer. In preparation of emulsified PEG, 16 g of PEG, 2.9 g L^{-1} of SDS, and 100 mL water were emulsified mechanically at 80°C with a stirring rate of 2500 rpm for 45 min. Subsequently, 1.8 g L^{-1} of PVA was added to the mixture to stabilize the emulsion. In preparation of pre-polymer, 24 g of urea and 14.8% formaldehyde were added to water. The mixture was stirred and adjusted to pH 8.5- 9 with an aqueous solution of 10% sodium hydroxide. Then, the mixture was continuously stirred at 70-75° for 1 h to prepare pre-polymer solution. Finally, droplets of the pre-polymer solution were added to the emulsion where the emulsion mixture was stirred at a rate of 1000 rpm at 80°C. The pH was reduced to the range of 5 -5.5 by adding dilute hydrochloric acid into the mixture. Then, the mixture was agitated continuously at a stirring rate of 600 rpm for 1 h, and the temperature was slowly reduced to 35°C. The resultant nanocapsules were filtered, washed and dried in an oven at 70°C for 8 h to remove water.

5.1 Coating the nanocapsules onto the cotton fabric

For developing thermoprotected cotton fabric, the prepared PEG and paraffin wax nanocapsules using polyurethane binding agent were applied on-to cotton fabric using pad-dry-cure method. The details of coating composition for different ratios of nanocapsules to polyurethane binding agent are given in Table 1. The four different ratios related to PEG and polyurethane binding agent was prepared for this study. Correspondingly, two different ratios related to paraffin wax and polyurethane binding agent was selected.

Table 1 Details of the prepared coating compositions

S.No.	Sample code	Nanocapsules to binder ratio (in mass ratio)
1.	Untreated fabric	-
Polyethylene glycol		
1.	$PEGS_{131}$	3:1
2.	$PEGS_{132}$	3:2
3.	$PEGS_{133}$	3:3
4.	$PEGS_{141}$	4:1
Paraffin Wax		
1.	PWS_{115}	1:5
2.	PWS_{225}	2:5

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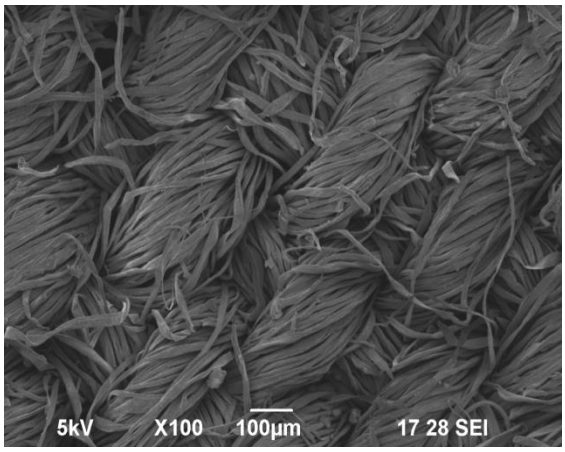
5.2 Development of Thermoprotected cotton fabric containing PEG nanocapsules

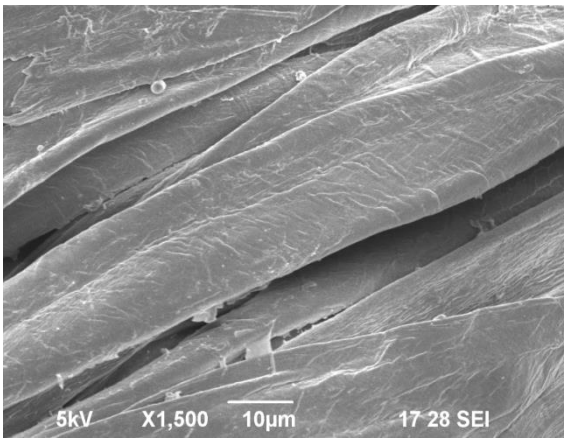
Thermoprotected cotton fabric is prepared by embedding the prepared PEG nanocapsules on-to the cotton fabric based on the four compositions is given in Table 1. The surface morphologies of the PEG nanocapsules coated fabric were investigated to understand the performance of the nanocapsules and binding agent affinity towards the cotton fabric. The physical testing such as tensile strength of the treated fabrics was carried out to evaluate the related changes before and after coating. The durability of the PEG nanocapsules related to binding agent adhered to the cotton fabric was identified with respect to abrasion resistance. According to heat and water absorption management by the clothing during furnace operations in foundry division, water absorbency and latent heat absorption of the treated fabric are crucial, and so they are evaluated in this study.

5.2.1 Morphology of the PEG Nanocapsule Treated Fabrics

SEM analysis was performed to study the morphological changes after treatment with PEG nanocapsules. Figure 9 shows the morphology of the untreated cotton fabrics. Figure 1 (a) illustrates that there are some protruding fibres can be observed on the surface of the untreated cotton fabric. Figure 1 (b) shows the grooves, and fibrils can be observed on the surface of the untreated cotton fabric.

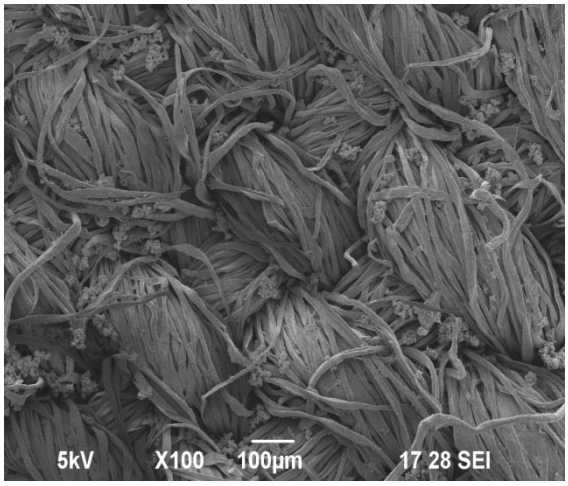


(a)

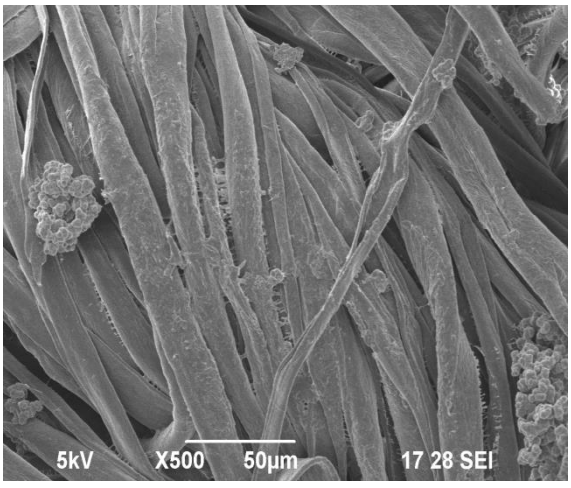


(b)

Figure 1 SEM Images of untreated fabric at different magnifications: (a) x100; (b) x1500 In Figure 2 (a), SEM image shows nanocapsules distributed over different locations on cotton fabric. The coated nanocapsules have a smooth and regular surface on the coated surface of the fabric, as observed in Figure 2 (b). It is also illustrated that the PEG nanocapsules are successfully coated on-to the cotton fabric without breaking or any deformation. Therefore, an even distribution of the PEG nanocapsules on the surface of the cotton fabric was achieved.



(a)



(b)

Figure 2 SEM images of PEG nanoc eated fabric at different magnifications (a) x100; (b) x500

5.2.2 Testing of Tensile Strength of PEG Nanocapsule Treated Fabrics

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The tensile strength measurements of the PEG nanocapsules treated fabric and untreated fabric are given in Table 3. The fabrics were tested in both warp and weft directions. The maximum load and elongation of the fabric at break were observed and recorded, as also given in Table 3.

Table *2* Tensile strength of untreated and PEG nanocapsule treated fabrics

S.No.	Sample Code	Tensile strength (N)		Elongation at maximum load (mm)
		Warp	Weft	
1.	**Untreated fabric**	283.46±0.49	111.6±0.36	22.36±0.05
2.	**$PEGS_{131}$**	236.8±0.1	124.2±0.2	22.46±0.17
3.	**$PEGS_{132}$**	253.7±0.15	132.23±0.15	22.7±0.26
4.	**$PEGS_{133}$**	258.26±0.68	128.56±1.04	22.5±0.34
5.	**$PEGS_{141}$**	268.23±0.23	114.33±0.35	22.06±0.49

From Table 2, it is clear that the tensile strength of the PEG nanocapsules treated fabrics decreased compared to that of the untreated fabric. With the exception of treated fabric $PEGS_{141}$, the tensile strength of the treated fabrics ($PEGS_{131}$ to $PEGS_{133}$) increased with increasing binder agent. The treated fabric $PEGS_{141}$ had higher tensile strength on the warp side of the fabric compared to other treated fabrics due to the lower amount of binder agent in the chemical finish. In general, the addition of the chemical finishes to the cotton fabric results in deterioration of the tensile strength of the fabric. Similar to that, all of the treated fabrics were found to have a lower tensile strength in the warp direction. On the contrary, the tensile strength of the treated fabric on the weft side was higher compared to that of the untreated fabric. This result may be due to the binder accumulation on the surface of the weft side of the treated fabric. In addition, the tensile strength of treated fabric $PEGS_{141}$ in the weft direction was lower than that of the other treated fabrics. This finding may be due to the tendency of more chemical finish to accumulate on the weft side of the fabric. It was observed that the tensile strength of the treated fabrics ($PEGS_{131}$, $PEGS_{132}$ and $PEGS_{131}$) increased due to the increased content of the binder agent in both the warp and weft directions. The elongation at break did not show any considerable differences after treatment.

5.2.3 Testing of Water Absorbency of PEG Nanocapsule Treated Fabrics

The water absorbency measurements of the treated and untreated fabrics are shown in Figure 3. From the figure, it is evident that the untreated fabric took more time to absorb water because it had more protruding fibres on the surface of the fabric. The treated fabric $PEGS_{131}$ took the least amount of time for water absorption compared to all other treated fabrics due to coating materials and reduced number of protruding fibres during the drying and curing processes.

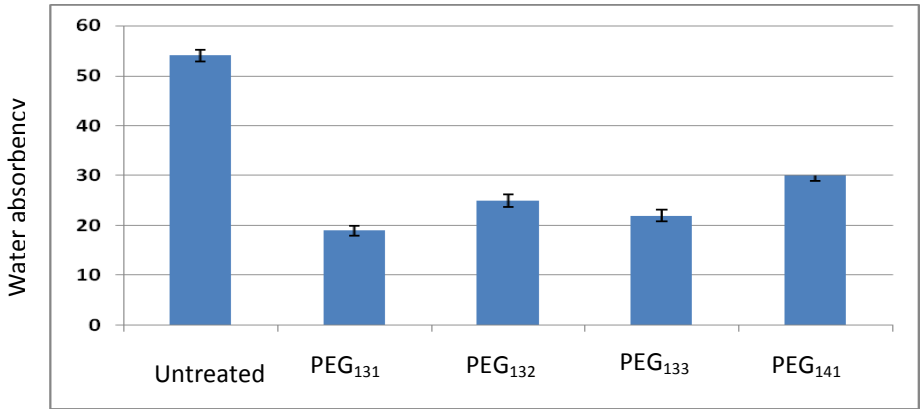

Figure 3 Water absorbency of untreated and PEG nanocapsule treated fabrics

The time taken for water absorption for treated fabric $PEGS_{132}$ was longer compared to that of the treated fabric $PEGS_{131}$. This result is due to higher amount of coating material added to the surface of the fabric, which reduces the number of pores on the surface of the fabric. In the case of treated fabric $PEGS_{133}$, larger amount of the coating materials on the surface of the fabric resulted in shorter time taken for water absorption is shorter compared to treated fabric $PEGS_{132}$ but longer than $PEGS_{131}$. This is due to the polar OH groups present in the cellulosic cotton that make the untreated fabric more hydrophilic than the fabrics treated with hydrophobic nanocapsules. For treated fabric $PEGS_{141}$, the fabric took longer time to absorb water than all other treated fabrics because of an insufficient amount of binder agent relative to the amount of nanocapsules. Hence, it is clearly indicates that the water absorbency rate is based on the deposition of amount of coating materials and treatment method.

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5.2.4 Testing of Abrasion Resistance of PEG Nanocapsule Treated Fabrics

The abrasion resistance measurements of the tested samples are shown in Figure 4. The test was carried out until the fabric threads on the abraded surface broke down.

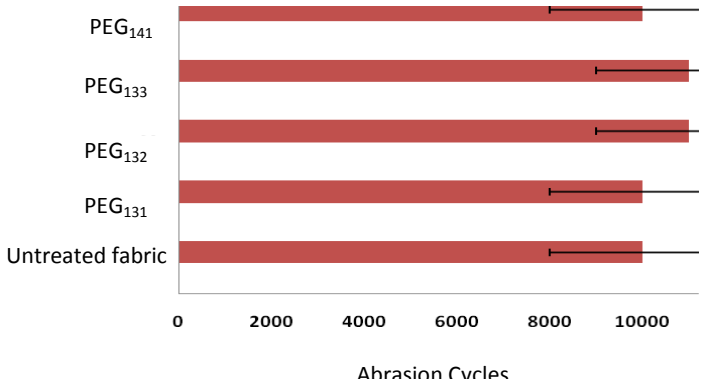

Figure 4 Abrasion resistance of untreated and PEG nanocapsule treated fabric

From the figure, it was found that the treated fabrics $PEGS_{131}$, $PEGS_{141}$ and the untreated fabric were abraded after 10,000 cycles, whereas the treated fabrics $PEGS_{132}$ and $PEGS_{133}$ were abraded after 11,000 cycles. The results of the reduced abrasion resistance of the treated fabrics may be due to the poor adhesion of the binding agent that added PEG nanocapsules on the surface of coated fabrics. Over-all, the treated fabrics $PEGS_{132}$ and $PEGS_{133}$ showed better durability, which was the result of the higher amount of binding agent added in the coating composition and increased the inter-molecular interactions between the PEG nanocapsules and textile surface.

5.2.5 Testing of Thermal Properties of the PEG Nanocapsule Treated Fabrics using DSC

The DSC curves of the PEG nanocapsule treated cotton fabrics are shown in Figure 5. Thermal properties of the treated fabrics, including melting temperature (Tm) and latent heat energy storage (ΔHm) are also illustrated in the figure. The figure also shows that the treated fabrics were capable of absorbing 1.98, 2.13, 3.19 and 2.03 J/g latent heat energy for the nanocapsules/binder agent ratio of 3:1, 3:2 3:3 and 4:1, respectively. Further, it is found that the melting temperature of the treated fabric $PEG_{132}$is higher than that of the other treated samples. This result shows that higher amount of PEG nanocapsules and binding agent may be present, according to the weight per unit area of the treated fabric PEG_{132} compared to the other treated fabrics.

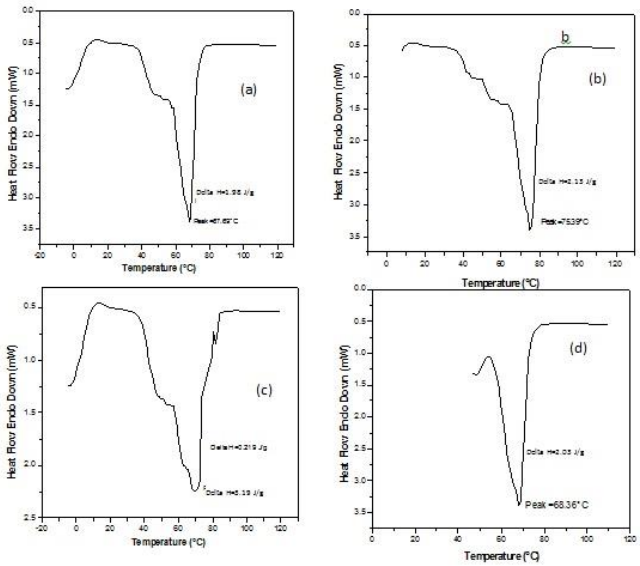

Figure 5 DSC analysis of PEG nanocapsule treated fabrics: (a) $PEGS_{131}$; (b) $PEGS_{132}$; (c) $PEGS_{133}$; (d) $PEGS_{141}$

5.2.6 Testing of Thermal Stability of the PEG Nanocapsule Treated Fabrics using TG Analyzer

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A thermogravimetry analysis experiments was used to assess the thermal stability of the treated and untreated fabric samples. Figure 6 shows the TGA curves observed and demonstrate that the weight loss occurs between 20 to 900 °C. The thermal degradation interval data obtained from the TGA curves are given in Table 6. The table shows that all the PEG nanocapsule treated fabrics show two-step weight loss thermal degradation, but there is only single-step weight loss degradation for the untreated fabric. From Figure 6, it is observed that changes in the weight loss occurring at temperatures below 250 °C were due to the removal of physically adsorbed water in all cases. The untreated cotton fabric containing cellulose was found to lose 70% of its weight between the temperatures of 300 and 400 °C. At this stage, the rapid weight loss is due to the dehydration of cellulose. At temperatures above 476 °C, char formation occurs and produces CO_2, carbonyl and carboxyl products.

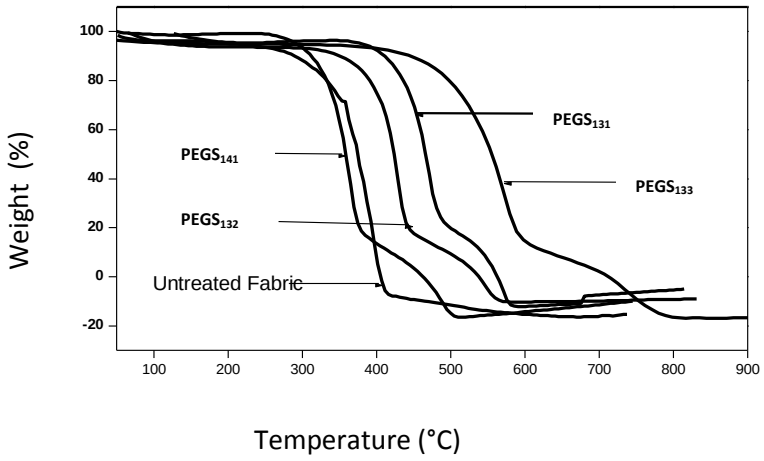

Figure 6 TGA of untreated fabric and PEG nanocapsule treated fabrics: $PEGS_{131}$; $PEGS_{132}$; $PEGS_{133}$; $PEGS_{141}$

The weight loss of the treated fabrics $PEGS_{132}$ and $PEGS_{141}$ between 300 and 400 °C was higher than that of the untreated fabric. The treated fabrics containing PEG nanocapsules/ binding agent ratio of 3:1 and 3:3 had a higher decomposition temperature in the first stage than the other treated fabrics. In the second stage, the decomposition temperature of treated fabric $PEGS_{133}$ (i.e. nanocapsules/binding agent ratio of 3:3) was higher than that of the other treated fabrics due to the cross-linking of the binding agent with cellulose in the cotton fabric. With the exception of treated fabric $PEGS_{141}$, the amount of final residue was lower in the treated samples than that of the untreated fabric. As a result, the treated fabric $PEGS_{133}$ containing PEG nanocapsules/binding agent with ratio of 3:3 reveals that the fabric has good thermal stability. Further, it is also confirmed by the DSC results, that the latent heat energy storage of the treated fabric $PEGS_{133}$ is 3.19 J/g, which is higher than that of the other treated fabrics.

6. Conclusion

The developed PEG and paraffin wax nanocapsules were successfully embedded onto cotton fabric using pad-dry-cure method. The morphology of the coated fabric was analysed using SEM. The results of the SEM illustrated that both PEG nanocapsules were successfully coated onto the cotton fabric without breaking or any deformation. The tensile strength test result showed that the treated fabrics with PEG nanocapsules exhibited decreased strength compared to the untreated fabric. The water absorbency test results demonstrated that the PEGS131 shows quick water absorbency rate than the other PEG nanocapsule treated fabrics. The result indicates that the least time taken due to the polar OH groups present in the cellulosic cotton makes the pure cotton fabric more hydrophilic than the fabrics treated with hydrophobic nanocapsules. The results also indicated that the water absorbency rate is based on the amount of coating materials deposited on the surface of the treated fabrics. According to DSC test results, the treated fabrics shows the latent heat energy storage in the order of PEGS133>PEGS132>PEGS141> PEGS131. The result is clearly indicates that the PEG nanocapsules with ratio 3:3 related to nanocapsules/binding agent shows superior latent heat energy storage than the other PEG nanocapsule treated fabrics. The result also indicates that the PEG nanocapsule plays a crucial role in the development of thermoprotected cotton fabric. This is due to the higher encapsulation ratio of PEG nanocapsules (59.5%). While considering the thermal stability of the treated fabrics analysed using TGA, the results indicated that the treated fabric PEGS133 exhibited good thermal stability than the other PEG nanocapsule treated fabrics. The results found that it is similar to the DSC results. This reason is due to the influence of larger amount of loaded nanocapsule binding with cellulose onto the cotton fabric using binding agent. Finally, it can be concluded that all the treated fabrics with PEG nanocapsules exhibited enhanced active thermal protection compared to passive thermal protection shown by cotton fabric. However, the treated fabric PEGS133 showed outstanding properties with regard to the thermal protection.

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