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Production of Smart Textile Using Trimethylolethane as Phase Change Material †

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Abstract: Recently, the need for thermo-regulating fabric in the textile industry has motivated both researchers and scientists to explore this new type of smart fabric. This study aimed to develop a smart textile using a polyester fabric coated with microencapsulated trimethylolethane (TME) hydrate as phase change material. The TME microcapsules were produced via in-situ polymerization of melamine-urea-formaldehyde (MUF) at varying emulsification time, stirring rate, and TME hydrate concentration. A knife-over-roll coating method was incorporated using polyester resin as binder for the production of the smart fabric. Fourier Transform Infrared Spectroscopy (FT-IR) analysis, Scanning Electron Microscopy (SEM), and Differential Scanning Calorimetry (DSC) were conducted to examine the chemical, morphological, and thermal characteristics of the microcapsules and the smart fabric respectively. Results showed that the highest amount of microencapsulated TME phase change material obtained is 18.883 mg. FT-IR results confirmed the presence of TME hydrate and MUF resin in the microcapsule at 3300, 2870, 1148, and 1390 cm^{-1}. The SEM results revealed an amorphous and rough surface of microcapsules. Furthermore, the DSC results demonstrated favorable thermal characteristics, measuring the latent heat storage capacities of the microcapsules before and after application to the fabric as 205.1674 J/g and 224.7318 J/g, respectively. Finally, the encapsulation efficiency was calculated as 64.715%, indicating potential fabric thermal storage application.

Keywords: trimethylolethane; microencapsulated phase change material; smart textile; phase change material

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1. Introduction

Recently, the need for innovating new type smart materials like phase change containing textile has been the focus of both researchers and scientists. One particular phase change materials (PCM) of interests is Trimethylolethane (TME) hydrate. TME hydrate is classified as an organic or natural PCM, it has advantages over other PCMs because it is chemically stable, secure, and non-reactive, solidifies without much supercooling, is non-corrosive, and can dissolve congruently. TME hydrate was chosen for this research since few studies use this as a phase change material [1][2]. Paraffin is the commonly used PCM in the production of smart textiles. The TME hydrate will be subjected to in situ polymerization to create microcapsules and will be then incorporated to the fiber by knife-over-roll coating [3][4]. Thus study presented the potential of microencapsulated TME as phase change materials and applied to textile for the production of smart materials and other applications.

2. Materials and Methods

In this study the chemicals used for the formation of the shell monomers were melamine ($C_{12}H_{10}O_3$, 99%, MACKLIN), urea (CH_4N_2O, 99%, RI Chemical), formaldehyde (37 wt.% aqueous solutions CH_2O, Medicare Laboratories). Trimethylolethane ($C_5H_{12}O_3$, 97%, MACKLIN) was used as the core PCM. The emulsifier used was Styrene maleic anhydride copolymer ($C_{18}H_{29}NaO_3S$, 99%, MACKLIN). Triethanolamine (10 wt.% $C_6H_{15}NO_3$ solution, Chemford), citric acid ($C_6H_8O_7$, 99.5%, OBETECH PACIFIC), and sodium hydroxide (NaOH, 95%, Alyson's Chemical) were used to regulate the pH level. Also (H_2O, 18.2 resistivities, Neco) and Distilled water (H_2O, Absolute) were used to wash the microcapsules. The chemicals used for the preparation of the aqueous coating solution were polyester resin ($C_{27}H_{36}N_2O_{10}$, 99%, Polymer Products (PHIL.) Inc.), which acted as the binder between the substrate and solution containing the microcapsules, and deionized water (H_2O, 18.2 resistivities, Myron L) and sodium hydroxide (NaOH, 95%, Alyson's Chemical) for the pre-treatment of the cloth [5][6].

Fourier Transform Infrared Transform (Spectrum 10, PerkinElmer) determined the total composition of the MPCM. Differential Scanning Calorimetry (STA200RV, Hitachi) measured the endothermic and exothermic transitions of the core material. Scanning Electron Microscopy (TM3000, Hitachi) observed the surface morphology of the microcapsules.

3. Results and Discussions

3.1. Production of Microencapsulated Phase Change Material

Shown in Table 1 are the weights of PCM microcapsules produced in varying microencapsulation parameters such as concentration, time of emulsion, and RPM.

Table 1. Weight of Produced MPCMs**.**

Run	Concentration	Time	RPM	Weight of microcapsules
1	100	180	700	16.727
2	100	240	800	13.082
3	100	180	900	17.0488
4	100	120	800	18.883
5	80	120	800	16.6845
6	90	120	900	16.6451
7	90	240	900	16.4323
8	90	240	700	16.294
9	90	180	800	18.178
10	90	120	700	18.1136
11	90	180	800	18.4062
12	90	180	800	18.4471
13	80	180	900	16.0746
14	80	240	800	18.4208
15	80	180	700	18.2716

It can be observed that changes in the weights of the samples occur when concentration is varied. The weight increases as the emulsion duration increases [7]. The time of emulsion indicated its influence on the encapsulation weight produced. According to the study of Jusoh & Othman (2016), short emulsifying time results in unstable emulsion caused by the poor homogenization of the mixture [8][9]. The longer the emulsification time, the more stable the emulsion developed as a result of the interior droplets that

formed. As RPM increases, the weight of the microcapsules gradually increases [10]. According to the study of Yulianingsih & Gohtani (2020), the rate of stirring during emulsification has an influence on droplet size, emulsion viscosity, and droplet polydispersity [11]. However, the weight of the microcapsules is not solely determined by the PCM concentration but also by the shell material and any additional additives used during the microencapsulation process. These components contribute to the overall weight of the microcapsules, but PCM concentration has a major influence on the encapsulation efficiency [12].

3.2. Characterization of Microencapsulated Phase Change Material

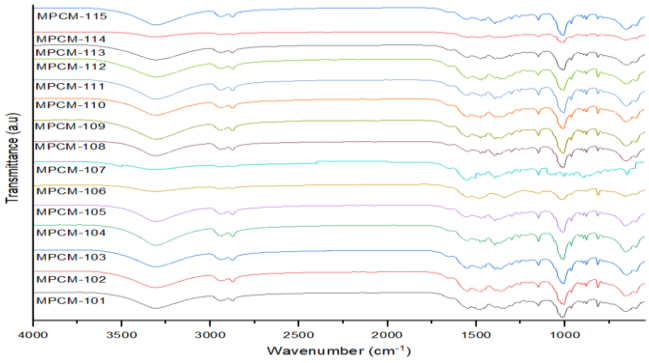

Figure 1. FT-IR Spectra of MPCM-101 to MPCM-115.

The samples showed absorption bands around 3300, 2870, and 1148 cm^{-1}. These absorption bands are attributed to the N-H stretching, C-H stretching, and C-N bending, which were the compound that made up most of the MUF resins as reported by the work of Xu et. al. [13]. The absorption band around 1390 cm^{-1} is assigned with the C-H bending, which was the compound made up the most of the TME hydrate (Ragadhita et al., 2019)[14].mThe spectra of MPCMs displayed a strong band around 3300 cm^{-1}. This absorption band is assigned to N-H stretching associated with the presence of a primary aliphatic amine. The C-H stretching around 2870 cm^{-1} corresponds to the presence of alkane, while the C-N bending band appearance around 1148 cm^{-1} is attributed to a secondary amine. The spectra shown in Figure 1 revealed that MPCMs contain an amine-related component, proving the presence of MUF resins. The C-H bending around 1390 cm^{-1} is associated with trimethyl, proving the presence of TME hydrate in the MPCMs (Ragadhita et al., 2019) [14].

On the other hand, Figure 2 shows an amorphous morphology of MPCM-101, MPCM-102, and MPCM-110, indicating that irregular particles were not absorbed, thus leading to the generation of a rough surface. Also several gaps were generated, which can contain air and minimize the contact area between the water droplets and the surface while also generating roughness in the structure. Several irregular microparticles were observed with MPCM-102. MPCM-110, produced with a lesser reaction time, rpm, and concentration, shows a conglomerated form resulting in smaller gaps.

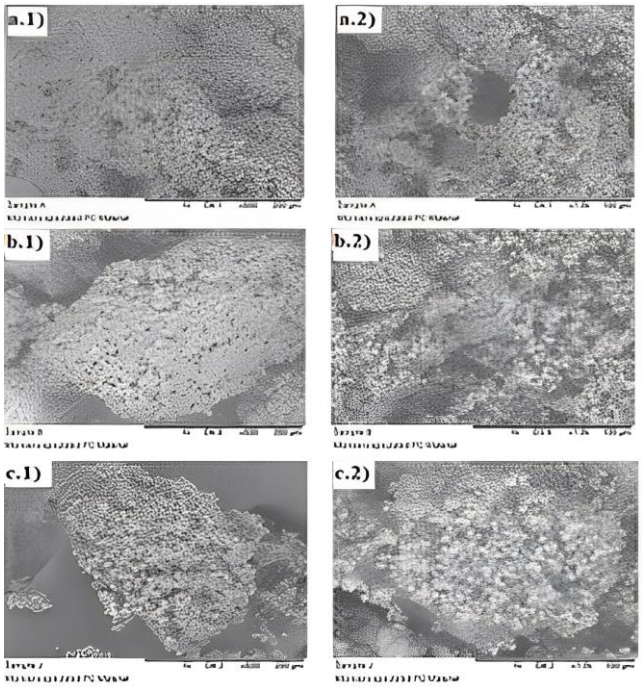

Figure 2. SEM Images of (a) MPCM-101, (b) MPCM -102, and (c) MPCM-110.

Figure 3 shows that the DSC curves move downwards and further to the right of the graph as the temperature increases. This movement indicates that changes in heat flow occur after the sample is heated at a stable temperature.According to Jang et al. (2019), the DSC curve has two important parts: baseline and peak [15]. A baseline is a straight line that appears parallel to the x-axis when the temperature difference between the MPCMs and reference is approximately zero. On the other hand, a peak is a curve that starts from the baseline and returns to the baseline. It consists of two connecting points: the point that moves down the baseline and returns to the baseline. These points created an area representing the latent heat of fusion of the sample.

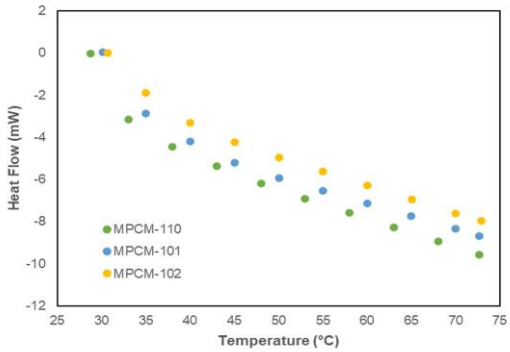

Figure 3. DSC curve of MPCM-101, MPCM-102, and MPCM-110.

Furthermore, MPCM-110 has a more stable temperature range than MPCM-101 and MPCM-102. A glass transition can be observed in the baseline of the measurement curve of MPCM-101 and MPCMP-102, meaning that there was a transition from amorphous to crystalline solid. The stability of the microcapsules during linear heating is a great indicator that MPCM-110 can be utilized as a heat storage material. Hence, MPCM–110 was chosen for fabric application before another DSC testing [16].

3.3. Production of Smart Textile with Microencapsulated Phase Change Material

As shown in Table 2, it shows the MPCM-110 has the highest encapsulation efficiency of 64.71%. Based on related studies, encapsulation efficiencies ranging from 60-90% were achieved with varying core-shell ratios. In this instance, it was discovered that higher latent heat can enhance a microcapsule's thermal conductivity. The amorphous form of the microcapsules indicated that they had good thermal stability. The MPCM-110 sample was used to coat the polyester fabric to produce smart textiles [17].

Table 2. Encapsulation Efficiency of MPCMs.

	Weight (mg)	**T_{onset} (℃)**	**T_{peak} (℃)**	**ΔH_{fusion} (J/g)**	**Encapsulation Efficiency (%)**
MPCM-101	16.73	30.15	72.68	124.30	39.20
MPCM-102	13.08	31.76	72.88	145.83	45.99
MPCM-103	11.15	28.79	72.64	205.17	64.71

Figure 4 reveals that the intensity of band frequencies at 2900 cm^{-1} and 1200 cm^{-1} was reduced due to a decrease in C-H stretching of the alkane and C-N bending of the secondary amine after applying the MPCM-110, respectively. For the peak at 3310 cm^{-1}, it shifted to the left and decreased in the N-H stretching of the primary aliphatic amine. The band appearing at 1390 cm^{-1} intensified after applying it to the fabric, which is associated with TME hydrate. The absorption peaks for smart textiles at 1720 cm^{-1} and 965 cm^{-1} were associated with C=O, and the vibration of O=C–O–C, respectively. These functional groups are identified as the carbon monoxide and ester in polyester fabric and in agreement with the work of Pradit et al. [18].

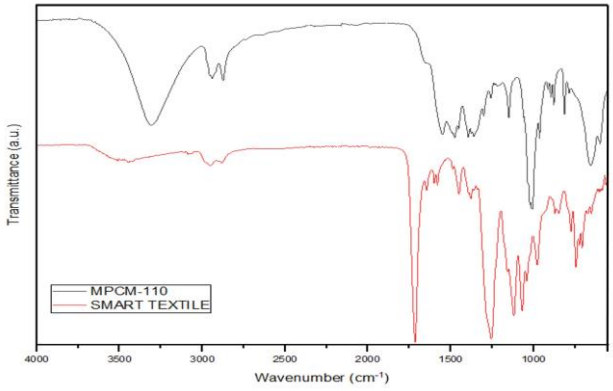

Figure 4. FT-IR Spectra of MPCM-110 and Smart Textile.

The MPCM-110 coated fabric's surface was rough and uneven, and the microcapsule coating solution may have been distributed unevenly, which would explain the surface roughness. Similar to the surface morphology of MCPM-110 in Figure 2, the produced smart textile shows an amorphous morphology as shown in Figure 5.

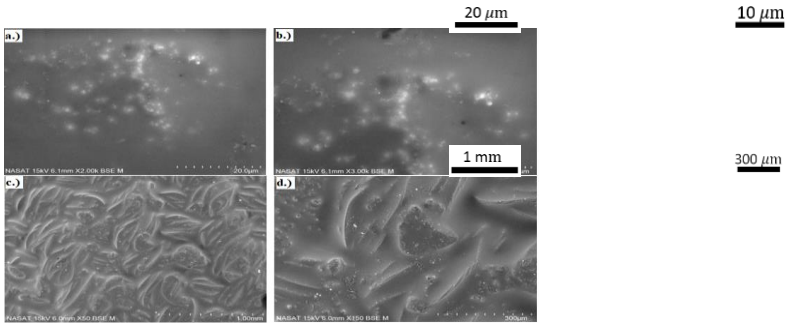

Figure 5. SEM images of Smart Textile (a) 2000x, (b) 3000x, (c) 50x, and (d) 150x.

The measured results of the smart textile are shown in Figure 6. The curve shows no endothermic peak at 30°C, corresponding to the melting point of TME hydrate. However, an enormous latent heat of fusion is obtained, this indicates that TME hydrate has potential as a material for latent heat storage at a relatively low temperature range. Additionally, the 90% composition of TME hydrate was not subjected to the phase separation phenomena., confirming the observation of Kiakuchi et al. [19] that the TME hydrate concentrations, where phase separation occurs, vary depending on the temperature and retention time.

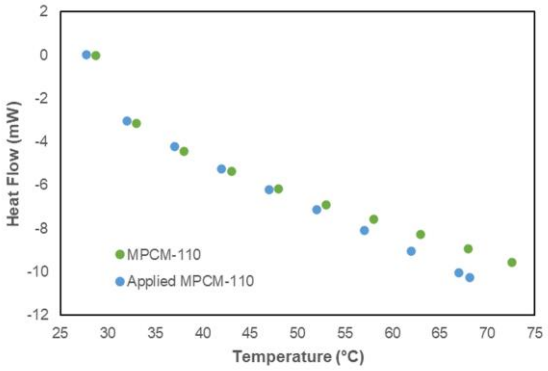

Figure 6. DSC curve of MPCM-110 and Smart Textile.

3.3.4. Elemental Distribution

Elemental compositions of the produced smart textile were determined using the Energy Dispersive X-Ray Spectroscopy (EDS), as shown in Table 3. The smart textile can be compared with the published elemental analysis of the untreated polyester fabric. The MPCM-110 containing fabric shows the presence of the C, O and N elements with 59.89%, 32.25% and 5.57% wt., respectively. The significant amount is due to the employed microencapsulated phase change material and binder, mainly composed of carbon, oxygen and nitrogen.

Table 3. Elemental Analysis of Smart Textile.

Element	Wt %
C	59.89
O	32.25
N	5.57

4. Conclusion

The presented the viability of producing a PCM containing smart textile using TME. It is confirmed that varying the parameters, such as temperature, concentration, time of emulsion, and rotation speed, results in noticeable disparities in the surface of the MPCM. The SEM-EDS images showed that the coating process used was successfully applied. The DSC curve showed that the MPCM-110 and the produced smart textile with higher latent heat and encapsulation efficiency. Thus, TME hydrate must be suitable as a PCM for fabric applications.

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