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Modification and evaluation of thermal properties of melamine-formaldehyde/n-eicosane microcapsules for thermo-regulation applications

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HIGHLIGHTS

- Modified eicosane microcapsules with the highest phase change enthalpies were made.
- Newton cooling law was fitted to determine thermal delay in PCM-substrates.
- Fine microcapsule units with diameters less than 0.5 μm were prepared.
- All pliable PCM-substrates can be thermally assessed using thermal logging method.

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ABSTRACT

A modified process to enhance the latent heat of fusion of n-eicosane microcapsules in melamine-formaldehyde shells is suggested for application in textiles. Deviations in melt enthalpy and phase change temperatures were determined for produced microcapsules by differential scanning calorimetry. Thermo-regulation efficiency of eicosane-microcapsule-treated fabrics was evaluated via fitting the Newton cooling law to the experimental data, and a new constant, α, was defined as the thermal delay factor. Scanning electron microscopy images and particle size distribution analysis were consistent and the particle size was found to be between 0.5 and 2.7 μm. Melamine-formaldehyde/n-eicosane microcapsule composition was confirmed using a Fourier transform infrared spectrophotometry. The microcapsules developed showed excellent heat storage capacities, over 162.4 J/g, over melting and crystallisation ranges compared with previous studies undertaken in this field.

1. Introduction

Application of phase change materials (PCMs) is an encouraging means to store and release thermal energy when supply and consumption of energy vary independently with time. Inorganic and organic PCMs are two major classes of such materials that have been widely investigated since 1980. Phase segregation, super-cooling, and inherent corrosiveness are some of the major drawbacks towards utilisation of inorganic PCMs as thermo-regulating agents [1,2]. Application of organic PCMs, which are non-corrosive in nature, was widely studied in textiles due to their high melt enthalpy (e.g. paraffin waxes and fatty acids) and thermal stability over many freeze–thaw cycles [3–5].

Temperature, homogenisation rate, pH, concentration of PCM, and shell (sheath) materials are key determining factors in the microencapsulation process and resulting performance attributes [4,8]. Microencapsulation of particles via in-situ polymerisation of formaldehyde polymers has been investigated previously [6,7]. Shin et al. [4] studied the microencapsulation of eicosane in melamine-formaldehyde shells, however, there was a significant decline (approximately 84 J/g) in amount of exchanged heat after microencapsulation of eicosane during the phase transitions. Low concentration of PCM and insufficient rate of homogenisation were assumed to be the major factors influencing such a decline in melt enthalpy for microencapsulated eicosane.

The main aim of the current study was to increase the amount of heat exchanged by microencapsulated PCM via ultra-homogenisation of concentrated eicosane emulsion. The heat capacities of eicosane

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and its microcapsules will be compared, and the amount of losses in heat capacities will be calculated accordingly. Thermoregulation efficacy of fabrics treated with eicosane microcapsules will be evaluated through fitting the Newton cooling law with the experimental data.

2. Experimental

2.1. Materials

Eicosane 99%, technical grade of sodium dodecylbenzenesulfonate (SDBS), melamine 99%, sodium hydroxide 97%, glacial acetic acid, and formaldehyde solution 37% were sourced from Sigma–Aldrich and were used as received. Polyester–viscose (80:20%·w/w) fabric (150 g/m^2) was purchased from Federal Mills, Australia; poly(vinyl alcohol) (PVA) with average molecular weight of 100,000 g/mol was purchased from Chem-Supply, and screen printing ink was obtained from Permaset, Australia.

2.2. Preparation of eicosane-microcapsules

Preparation of eicosane-microcapsules with melamine-formaldehyde as shell material was carried out via three separate steps:

A) An Ultra-Turrax T50 from IKA was used to homogenize 15 g of eicosane in 100 mL of a SDBS 2%·w/v solution at 70 °C and 8000 rpm for one hour. The emulsion was further processed using a Cole-Palmer VC33 ultrasonic homogenizer (500 W) for one hour at the same temperature.
B) To prepare melamine-formaldehyde pre-polymer, 100 mL of distilled water comprising melamine (0.1 mol/L) and 0.3 mol/L of formaldehyde (37%) was prepared, and pH was adjusted to 9 using 10%·w/v sodium hydroxide.
C) Homogenous eicosane emulsion was gradually added to the pre-polymer solution and 0.01 g of anti-agglomeration agent, PVA, was added to the solution at 50 °C. pH was adjusted to 4 using 10%·w/v acetic acid and stirring continued at 350 rpm for 2 h.

The resulting slurry was filtered-washed with distilled water, 3 times, and left to dry at 35 °C and 65% relative humidity for 3 d to result in microencapsulated eicosane-particles. The yield of microcapsule production was calculated to be 17%.

2.3. Application of microcapsules onto fabrics

A coat-dry-cure method was used to incorporate the microcapsules to two polyester–viscose fabric pieces (12 cm × 12 cm). Semi-viscose PCM-dope comprising 20%·w/v microcapsules in screen printing solution was prepared and was stirred at 50 °C for one hour until a homogenous dope was produced. Both sides of fabrics were coated with the PCM-dope using a Type SV-MATIS laboratory coating machine. The coated fabrics were initially dried at 80 °C for 10 min and were subsequently cured at 130 °C for 3 min. The relative add-on weight percent after treating the fabrics was 31% to the weight of fabric (WOF). In order to determine the weight proportion of PCM-microcapsules on the cured fabric, first, the same polyester–viscose fabric was coated with printing paste (without PCM-microcapsules) and the weight fraction of remaining paste after curing was calculated to be 0.358, Equation (1). According to Equations (2) and (3) the weight ratio of added PCM-microcapsules and the unevaporated printing paste was determined to be 10% WOF and 21% WOF, respectively.

$$W_u = 0.358W_0 \tag{1}$$

$$W_{pcm} = W_t - W_u \tag{2}$$

$$\left(\frac{W_t}{W_i}\right) \times 100 = \quad = \left(\frac{W_{pcm} + W_u}{W_i}\right) \times 100 \tag{3}$$

where: W_0 is the weight of the coated printing paste on a polyester–viscose fabric before curing, W_u is the weight of the unevaporated part of printing paste coated on a polyester–viscose fabric after curing, W_t is the total add-on weight after curing the printing paste containing PCM-microcapsules, W_{pcm} is the weight of eicosane-microcapsules added to the fabric after curing, and W_i is the initial weight of the untreated fabric.

2.4. Evaluation of thermal behaviour

The thermoregulation efficiency of prepared fabrics was evaluated via two methods: thermal delay efficiency and differential scanning calorimetry.

2.4.1. Thermal delay efficiency

An online thermal data-logger equipped with NI4350 high precision temperature and voltage meter (made by National Instruments, Ireland) was used to record the changes in temperature of both treated and untreated fabrics over one thaw–freeze cycle. All PCM-treated and control (untreated) fabrics were tightly wrapped around the thermal sensor (single-ply and two-ply) and were initially placed in the freezer at 0 °C prior to data logging. After reaching the initial temperature (around 0 °C), temperature logging started as the fabrics were placed in an oven (54 °C). When all the fabric specimens equilibrated with the oven temperature, they were placed in a freezer until they reached their minimum temperature (approximately 0 °C).

Datafit–version 9 software was used to fit the experimental data to the Newton cooling law with regression equations (Equation (4)). The thermal delay factor, α, in the Newton cooling law was considered to be an indication of the thermoregulatory effect for all treated and untreated fabrics. By definition, low or high values of α corresponds to high or low efficiency in rendering thermoregulation, respectively.

$$T = T_h + (T_0 - T_h)\exp(-\alpha t) \tag{4}$$

where T is specimen temperature at specific time, t (in s), T_h: environment temperature (°C), T_0: initial temperature (°C), and α is a constant defined as the Newton thermal delay factor.

2.4.2. Differential scanning calorimetry

A Pyris 1 differential scanning calorimeter (DSC) from PerkinElmer was used to quantify the melting and crystallisation enthalpies of eicosane and microcapsules over one thaw–freeze cycle (7 °C–52 °C and 52 °C–5 °C). The test was conducted under the nitrogen purge (20 mL min^{-1}) and at the same heating and cooling rate (5 K min^{-1}).

2.5. Characterisation of microcapsules

2.5.1. Fourier transform infrared spectroscopy

Chemical characterisation was carried out using a Fourier transform infrared (FTIR) spectroscopy, PerkinElmer 400 FTIR–FT-NIR, ranging between 650 cm^{-1} and 4000 cm^{-1}.