Journal of Fiber Bioengineering and Informatics 13:2 (2020) 61–68
doi:10.3993/jfbim00330

Thermal Performance of A Multi-layer Composite Containing PEG/laponite as PCMs ⋆

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Abstract

Six phase change materials (PCMs) were prepared by blending polyethylene glycol (PEG) with laponite powders (LP) at different ratios (PEG:LP, 100:0, 97:3, 96:4, 91:9, 88:12 and 85:15) under 100 °C firstly. A multi-layer composite consisting of a PCM loaded layer, a nano barrier layer and a protective layer was used to hold PEG/LP PCMs. SEM was used to observe the morphological structure of multi-layer composites containing PEG/LP composites. DSC revealed the melting temperature, the cooling temperature and the enthalpy of PEG and laponite as PCM. The thermal insulation of all the composites containing PEG/LP was evaluated under 78 °C. The results gave that the multi-layer composite containing PCM was obtained and a better adhesion of between the PCM loaded layer, the nano barrier layer and the protective layer was found. LP functionalized as a nucleating agent to accelerate the crystallization of PEG and 15wt% LP in PCM resulted in a disordered PEG molecular. The composites with more laponite had the higher thermal insulation and highest value reached 0.25.

Keywords: PEG; Laponite; Multi-layer Composite; Nanofibrous Membrane; PA 6

⋆Project supported by the research project of Student Grant Competition of Technical University of Liberec No. 21307/2019 granted by Ministry of Education Youth and Sports of Czech Republic and the Ministry of Education, Youth and Sports of the Czech Republic and the European Union - European Structural and Investment Funds in the Frames of Operational Porgramme Research, Development and Education - project Hybrid Materials for Hierarchical Structures (HyHi, Reg. No. CZ.02.1.01/0.0/0.0/16_019/0000843), project 'Design optimization and application of smart heat-insulating nano-layers' [LTACH-17014, 18301] and project 'Intelligent thermoregulatory fibers and functional textile coatings based on temperature resistant encapsulated PCM' SMARTTHERM (Project No. TF06000048).

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Jun. 2020

1 Introduction

Phase change materials (PCMs) are a group of materials with an intrinsic capacity to absorb and release latent heat during the phase change cycle, which is a dynamic heat exchange process [1]. The storage of latent heat or thermal regulation is based on the phase transition of materials including the phase change from solid to liquid, liquid to gas and vice versa [2]. Nowadays, PCMs are aimed to be incorporated with textiles ensure the comfort or protection of wearers under extreme external thermal conditions [3-4]. The thermal behaviour of PCM-incorporated textiles, especially in thermal insulation applications, has been evidently improved [5]. To incorporate the PCMs with textiles, many efforts have been made including preparation of hollow fibers containing PCMs [6], fibers spun with PCMs [7-8], preparation of solid-solid phase change materials [9-10] and coating fabrics with various encapsulated micro-PCMs [11] have been consecutively applied in textiles. Recently, the nanofibrous membranes have also been shown a great potential for these purposes [12]. The quality of these nanofibrous membrane for storing or holding PCMs is related both to their fibrous structure, controllable porosity, and their chemical composition [13]. The overall thermo-insulating efficiency is also strongly influenced by the incorporation of these membranes into textile structures where one of the decisive factors is the thickness and porosity of fibrous component. Hence, the composite made of nanofibrous membranes and PCMs is expected to have the better thermal insulation for textile.

Polyethylene glycol (PEG) is a common PCM and known as its non-flammability, high heat of fusion, low vapor pressure chemical, thermal stability, non-toxicity, non-corrosive [14] and moderate melting temperature intervals suitable for humans [15]. PEG was incorporated with textiles to improve the thermal behaviour [16, 17]. The popular methods of preparation of PCMs based on PEG are physically blending PEG with other supporting materials including SiO2, microcrystalline cellulose and graphene aerogel [18] and so on. Laponite powder (LP) is a relatively uniform disc-shaped synthetic clay with 25nm dia. and 1 nm thickness [19]. It is reported that the PEG and laponite has the excellent thermal properties [20]. However, the details of PEG and laponite as PCM in textiles are not clear.

In this work, we developed a smart heat storage composite based on a multi-layer structure consisting of PCM loaded layer which is supposed to make PCM layer even, barrier nano layers aimed to avoid leakage and protective layers protecting the structure of nano layers. Milife fabric, a non-woven polyester fabric with controlled density and thickness, was used as the PCM loaded layer as well as proactive layers. PA 6 nanofibrous membranes was used as the nano barrier layer. The main purpose was to show the difference in thermal behavior of multi-layer composite containing PEG modified with different laponite contents. Based on the literature review, this work is one of the initial ones that studied the thermal behavior of multi-layer composite containing PCM.

2 Experimental

2.1 Materials

Polyethylene glycol powders ($M_W = 1\,500$) was purchased from Aldrich and Laponite RD powders (LP) were purchased from BYK-Chemie GmbH. All these chemicals were used without further purification during the experiments. The milife fabrics (polyester) with 10 g/m^2 and 0.06 mm

thickness were purchased from CLAF@ and polyamide nanofibrous membranes with 7.48 g/m^2 and 0.01 mm thickness was provided by Institute for Nanomaterials, Advanced Technologies and Innovation.

2.2 Preparation of PCM

PEG was used as PCM and LPwere as fillers to stabilize PEG. Firstly, PEG powders were totally melt at 60 °C and then LP was added into melt PEG at high-speed stirring under 60 °C till the PEG/laponite mixture became homogenous. Here, six PCM solution with different ratios of PEG to laponite were prepared and labeled as L1, L2, L3, L4, L5 and L6. The components of the prepared PCMs were introduced in Table 1. Besides, all the PCM solutions were kept at 60 °C for the following preparation of a multi-layer composites containing PEG/LP.

Table 1: Content of LP-Modified PEG as PCM

PCM Number	PEG (wt%)	Laponite (wt%)
L1	100	0
L2	97	3
L3	94	6
L4	91	9
L5	88	12
L6	85	15

2.3 Preparation of Multilayer Composite Containing PCM

The multi-layer composite includes three kinds of layers including protective layer (PL), nano barrier layer (NBL) and PCM loaded layer (PLL). Milife fabrics was used in PL and PLL and PA6 nanofibrous membrane was used as NBL. In the first step of preparation of composites, PLL was placed on the surface of the first NBL and then PCM solution was applied homogeneously on the surface of the PLL. This step should be under 60 °C. In the second step, NBL was placed to cover the PLL totally and evenly, as well as kept connective with first NBL. This step should be under 60 °C. Finally, PL were applied on both sides of the samples created in the second step and then the multi-layer composite was obtained after cooling in room temperature.

In this work, six multi-layer composites were prepared with prepared PCMs separately. The weight of PCMs inside of multi-layer composites was kept same. The details of multi-layer composites were shown in Table 2.

2.4 Methods and Tests

2.4.1 Structure of Multi-layer Composites

The cross-section morphology of multi-layer composites containing PCM was observed via the scanning electron microscopy (SEM) (VEGA TESCAN Inc. USA) at 20 kV.

Table 2: Description of Multi-layer Composites Containing PEG/LP as PCMs

Composites Number	NBL	PL	PLL	PCM	W_1 (g/m^2)	W_2 (g/m^2)	p.(wt%)
C1	PA6 Nanofibrous membrane	Milife Fabric	Milife Fabric	L1	222.2	222.2	81.9
C2				L2	222.2	215.6	81.4
C3				L3	222.2	208.9	80.9
C4				L4	222.2	202.2	80.4
C5				L5	222.2	195.6	79.9
C6				L6	222.2	188.9	79.4

W1: Grams per Square Meter of Dry PCM inside of Composite (g/m^2)
W2: Grams per Square Meter of Dry PEG inside of Composite (g/m^2)
p.: Weight Percentage of Dry PEG inside of Composite (wt%)

2.4.2 Thermal Capacity of Multi-layer Composites Containing PCM

Differential scanning calorimetry (DSC) was used to measure the thermal properties of multi-layer composites containing PCM from 15 °C to 70 °C with a heating rate of 10 °C/min under N_2 gas purge with a rate of 20 mL/min.

2.4.3 Thermal Insulation of Multi-layer Composites Containing PCM

Thermal insulation was tested by a custom-built set-up which included a thermal camera and a stable heater shown in Fig. 2. Thermal camera (FIR E5) emissivity was set as 0.98 and the stable heater (GFL) was used as hot plate. The hot plate was kept stable at a constant temperature around 78 ± 1 °C (T_H) and the room temperature (T_R) was around 25 ± 2 °C which was confirmed by thermal camera in advance. During the measurement, FLIR thermal camera was placed 400 mm away from the hot plate and then the sample was attached onto the surface of the hot plate. The temperature (T_F) of the sample was recorded by taking infrared thermo graphics after 600 s. The thermal insulation value of each sample was calculated by Equation (1).

$$I = \frac{T_H - T_F}{T_H - T_R} \tag{1}$$

3 Experimental

3.1 Structure of Prepared Composites Containing PCM

The structure and cross-section of prepared multi-layer composites with different layers containing PCM is shown in Fig. 1. From the microscopic cross-sectional image, the PCM layer was homogenous and there was no obvious gap between PCM and nano barrier layers which kept the composite layer intact. The good adhesion between different layers of the multi-layer composite was suggested.

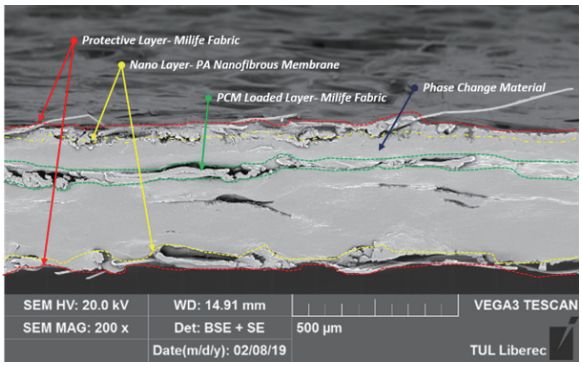


Fig. 1: Cross-section Morphology of a Multi-layer Composite Containing PEG/LP as PCM

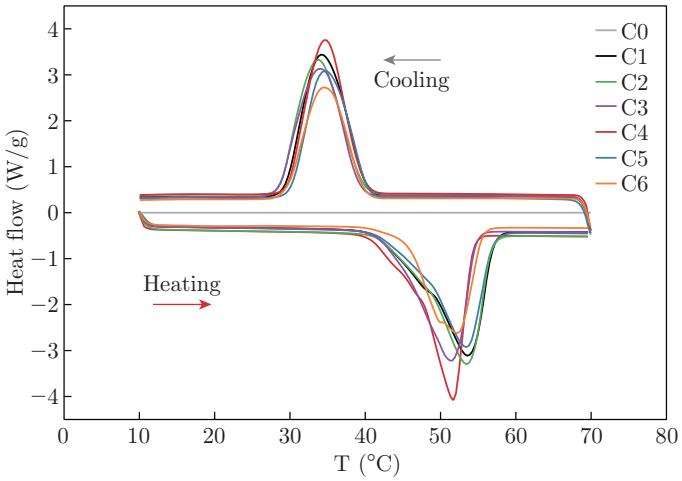


Fig. 2: DSC Curves of Different Multi-layer Composites Containing PCMs

3.2 Thermal Capacity of Composites Containing PCM

The thermal capacity of multi-layer composite containing different PCMs was measured by DSC. Fig. 2 showed the heating process and cooling process of multi-layer composite containing PCM ranging from 15-70 °C. Phase change temperature together with latent heat values of multi-layer composites containing PCM was also calculated from DSC curves and shown in Table 3.

It was obvious that no thermal capacity was found in C0 composites without PCM during the heating process, but those multi-layer composites containing different concentration of PCM showed a considerable effect in thermal capacity. From Table 3, T_{om} of multi-layer composites containing PCM (C1, C2, C3, C4 and C5) was found to range from 40 °C to 42 °C except for C6 whose T_{om} was approximately around 44 °C. In addition, C1 had the relative lowest T_{om} when compared with other composites. For T_{em}, C1 and C3 had the relative higher value as 57.11 °C and 57.69 °C respectively, and T_{em} of other composites were smaller than 57 °C. The difference in the change of T_{om} and T_{em} was caused by the content of LP in the composite and the result showed that the add of LP narrowed the latent heat process of multi-layer composite containing PCM in the heating stage in some degrees. For T_{pm}, the value of C1 which contained the pure PEG was the highest as 53.55 °C among all the composites. But for other composites containing PEG/LP as PCM, a decrease in T_{pm} was observed when compared with C1. Additionally, highest melting enthalpy 116.18 J/g was observed in C1 from Table 3, and then the melting enthalpy of C2, C3,

Table 3: Phase transition of PCMs inside Multi-layer Composites Calculated from DSC Curves

Composite Number	T_{om} (°C)	T_{pm} (°C)	T_{em} (°C)	ΔH_m (J/g)	T_{oc} (°C)	T_{pc} (°C)	T_{ec} (°C)	ΔH_c (J/g)	ΔT (°C)
C0	–	–	–	–	–	–	–	–	
C1	41.21	53.55	57.67	116.18	40.77	34.28	28.79	112.25	0.44
C2	40.22	51.69	54.47	112.33	39.47	34.70	29.6	108.17	0.75
C3	41.26	53.41	57.69	108.83	40.5	34.73	30.07	97.52	0.76
C4	41.32	53.44	56.89	107.57	40.17	33.84	28.42	104.52	1.15
C5	41.32	51.36	54.82	105.75	40.02	33.92	28.28	101.24	1.3
C6	44.66	52.21	55.79	77.30	44.10	34.70	29.60	82.80	0.56

T_{om}: onset melting temperature; T_{pm}: peak melting temperature; T_{em}: endset melting temperature; T_{oc}: onset cooling temperature; T_{pc}: peak cooling temperature; T_{ec}: endset cooling temperature; ΔH_m: melting enthalpy change; ΔH_c: solidification enthalpy change; ΔT: $T_{om} - T_{oc}$

C4, C5 and C6 decreased with add of LP respectively, and C6 had the lowest melting enthalpy 77.3 J/g. Similarly, it was obvious that C0 without any PCM inside had no thermal capacity during the cooling process, but those multi-layer composites containing PCM had thermal capacity. However, the DSC curves of all the composites containing PCM were similar. The T_{om}, T_{em} and T_{pm} calculated from DSC curves in Table 3 also proved it. So, the results suggested that LP made little effect on the cooling process. But for solidification enthalpy, the change was similar as the change of melting enthalpy. C1 had the highest solidification enthalpy 112 J/g from Table 3, and the solidification enthalpy of C2, C3, C4, C5 and C6 decreased generally. C6 had the lowest cooling enthalpy as 82.80 J/g. It was also found that cooling enthalpy was higher than melting enthalpy in C6 whose PCM containd 15wt% LP.

In addition, super cooling degree (ΔT) increased with more content of LP in PCM except for C6. In this case, LP was a nucleating agent for PEG during heating or cooling process. The rate of thermal energy release of LP/PEG during the cooling process was increased, which resulted in acceleration of the crystallization of PEG.

3.3 Thermal Insulation of Multi-layer Composites Containing PCM

From Table 4, it was obvious that C0 without any PCM had the lowest value and I value increased from 0.130 to 0.253 when PCM was deposited inside the multi-layer composites. Such

Table 4: Thermal Insulation of Multi-layer Composite Containing PCM under 78 °C

Composite Number	Thermal insulation I
C0	0.130
C1	0.147
C2	0.147
C3	0.158
C4	0.196
C5	0.223
C6	0.253

upward trend proved that 1) PCM inside of the multi-layer structure supported the higher thermal insulation because of their instrinsic thermal capacity; 2) more weight percentage of laponite in PEG/laponite reduced the overall thermal conductivity. It may be caused by the lower thermal conductivity of laponite.

4 Conclusion

Various PEG/LP composites as PCM were prepared and then placed in a multi-layer structure consisting of PCM loaded layer, nano barrier layer and protective layer. The melting enthalpy in all the multi-layer composites containing PEG/LP as PCM was suitable for human body. Add of LP in PEG narrowed the phase transition of PCM. Espeically, LP in PEG/LP functionalized as a nucleating agent to accelerate crystallization rate of PEG. PCM inside multi-layer composites increased thermal insulation and more LP in PCM also improved the thermal insulation of the multi-layer composite.

Acknowledegement

This work was supported by the research project of Student Grant Competition of Technical University of Liberec No. 21307/2019 granted by Ministry of Education Youth and Sports of Czech Republic and the Ministry of Education, Youth and Sports of the Czech Republic and the European Union-European Structural and Investment Funds in the Frames of Operational Porgramme Research, Development and Education-project Hybrid Materials for Hierarchical Structures (HyHi, Reg. No. CZ.02.1.01/0.0/0.0/16_019/0000843), project 'Design optimization and application of smart heat-insulating nano-layers' [LTACH-17014, 18301] and project 'Intelligent thermoregulatory fibers and functional textile coatings based on temperature resistant encapsulated PCM' SMARTTHERM (Project No. TF06000048).

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