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Solar Energy Materials & Solar Cells

journal homepage: www.elsevier.com/locate/solmat

Polyethylene/paraffin binary composites for phase change material energy storage in building: A morphology, thermal properties, and paraffin leakage study



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ARTICLE INFO

Article history:

Received 22 May 2014

Received in revised form

22 November 2014

Accepted 8 January 2015

Available online 13 February 2015

Keywords:

Phase change materials

Paraffin

Thermal energy storage

Thermal transition

Leakage

ABSTRACT

Phase change materials (PCMs) are able to melt and solidify at a certain temperature with a high heat of fusion. These promising functional materials for acting as energy as latent heat storage units have one major problem, leakage of the PCMs when molten. Polymer/phase change material (PCM) blends were investigated as a possible solution of PCM leakage. In this research, paraffin was used as the PCM and three types polyethylene (PE) were used as the structural matrix. To investigate the morphology of PE/paraffin blends and evaluate the influence on blend behavior, paraffin was blended with either high density polyethylene (HDPE), low density polyethylene (LDPE), or linear low density polyethylene (LLDPE) using a parallel co-rotating twin screw extruder. Chloroform extraction was utilized to estimate the maximum amount of paraffin able to be released by the PE network at equilibrium. Scanning electron microscope (SEM) images of chloroform-extracted samples were collected to investigate the blend structure. Thermal transition temperatures and crystallinity of components in the blends were characterized by differential scanning calorimetry (DSC) and polarized optical microscopy (POM) was used to investigate crystal perfection changes of the polyethylenes before and after blending with paraffin. Paraffin leakage of the blends was investigated by accelerated degradation test in an oven at 60 °C. The leakage behavior was analyzed by Korsmeyer–Peppas equation to determine the underlying mechanisms. Chloroform extraction indicated that almost all the paraffin would leak out from the blends in the long run. A co-continuous structure of the blends was evident from the chloroform extraction and subsequent SEM images. While this co-continuous structure controlled leakage behavior, it had little influence on paraffin thermal transition temperatures and crystallinity based on DSC study. In contrast, PE crystal perfection decreased with paraffin level as evident in POM images. This structure resulted in leakage of paraffin in all formulations.

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1. Introduction

Phase change materials (PCMs) are studied [1–4] and used for storage of solar thermal energy in buildings to reduce heating and cooling demands. But, when a PCM melts, the molten material can leak over time. Schossig et al. notes that leakage of non-encapsulated PCMs may be an issue over the lifetime of the application [5]. Containment methods offer a promising strategy to create form-stable PCMs that maintain their shape even if PCMs melt. Methods such as macro- and micro-encapsulation [6], immersion, and direct incorporation can reduce leakage. For example, using a skeletal material with a higher melting temperature than that of the PCM is a promising solution [7,8].

To reduce costs for building application, commodity plastics such as polyethylene (PE) have been explored as a sealant to reduce paraffin seepage [9–16]. Estep and Bender explored the use of paraffin as a viable thermal energy storage material, and indicated that the leakage of PCM in HDPE/paraffin blend can be problematic [16]. They also noted that studies quantifying PCM leakage were lacking [16]. As follow-up, our study investigates PCM leakage amount in PE/paraffin blends and blends structure based on their work.

Accelerated testing methods are often used to analyze product quality, performance, and life span in harsh environments [17,18]. In studies to evaluate paraffin release, specimens have been placed in a solvent or high temperature environment to accelerate leakage, providing useful information for comparison. Estep and Bender designed a PCM leakage test to determine the total encapsulation of paraffin in a PCM/HDPE blend. Specimens were subjected to chloroform, which is a paraffin solvent that does not dissolve HDPE under the

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test conditions [16]. They commented that the solvent bath is a more stringent test of leakage than might be expected in field conditions [16]. However, the test is useful to determine the interconnectivity of the paraffin phase. Such a finding could establish a basis for whether long-term PCM seepage from the composites was possible but not necessarily accurate life prediction. To study PCM leakage, Beginn [19] developed a paraffin release test for shake-blended samples, which was made at a shaking frequency of 500 rpm. The ultra-high-molecular-weight polyethylene (UHMW-PE) powder was suspended into molten paraffin at 150 °C under constant shaking until complete dissolution of the UHMW-PE was achieved [19]. Rectangular UHMW-PE-paraffin gel blends were placed in a preheated oven on a sandwich of 10 cellulose filter sheets for 24-h at a defined temperature [19]. Weight loss was recorded for the samples as an indicator of leakage [19]. Zhang et al. applied this method to the quantitative analysis of paraffin leakage [20].

Time-dependent leakage curves can be prepared using the thermally activated paraffin release test of Zhang et al. [20]. Such curves are similar in function to those used to characterize drug release [21–24] that use numerical approaches based on Korsmeyer–Peppas equation. This approach is used to investigate the release kinetics of drugs from porous tablets and coefficients characterizing the process [21,22]. Paraffin leakage from PE/paraffin blends may share similar processes. Hence, it seems plausible to analyze paraffin leakage performance using the same method.

Our study fills a limitation in the literature by exploring morphological structures with paraffin leakage behavior in polyethylene/paraffin binary composites. To evaluate the leakage behavior of paraffin in PE for future form-stable PCMs, our specific objectives were to: (1) investigate the PE/paraffin binary composites structure; (2) compare structure influence on the thermal properties and leakage behavior of paraffin in HDPE, LDPE, and LLDPE.

2. Materials and method

2.1. Materials

A technical grade of octadecane paraffin from Roper Thermals (Lot # C0036824) was used as the PCM. Extrusion grade polyethylene was also sourced commercially. These included Ineos HP54–60 Flake, Petrothene[®] NA960000 (Lyondell Chemical), and Petrothene GA501021 (Lyondellbasell) for HDPE, LDPE, and LLDPE, respectively. Details of materials are available elsewhere [7].

2.2. Blending

An 18-mm parallel co-rotating twin-screw extruder (Leistritz ZSE-18HP) was used to compound PE/paraffin blends with different weight ratios. First, the solid PE powder was manually blended with molten paraffin in a beaker before compounding. Extrusion was then performed at a temperature of 180 °C and a screw speed of 80 rpm. Details of the manufacturing process are available elsewhere [7].

2.3. Characterization

2.3.1. Chloroform extraction

A solvent was needed to selectively dissolve the paraffin from the PE material. Lyu et al. indicated that chloroform did not dissolve HDPE at room temperature [26] and chloroform was commonly used as a solvent to extract impurities from LDPE and LLDPE polymeric matrix [27,28]. Chloroform is a suitable solvent for the paraffin according to Merck Index's research [29].

To estimate the total amount of paraffin movement through the PE network at equilibrium conditions, chloroform was used to extract the paraffin in the blends. For this test, the PE/paraffin composites were

extruded into cylindrical strands through a die with approximately a diameter of 0.095-in. The strands were cut to length producing specimens of approximately 15-mg in mass. The cylindrical test specimens were subsequently soaked into a chloroform bath of 25-ml in volume. The vial containing the chloroform was maintained in a heated water bath to 40 °C. Although paraffin is soluble in chloroform at ambient temperatures, the low heating temperature facilitated more rapid extraction, while reducing chloroform evaporation and allowing the PE component to remain solid. Samples were air-dried in a fume hood for ca. 12-h after extraction to facilitate chloroform evaporation and accurate weight loss measurement.

2.3.2. Morphology characterization

The cylindrical specimens submerged in a chloroform solvent bath at room temperature, were removed after 6 and 18-h. The specimens were subsequently dipped in liquid nitrogen for 10-min and manually fractured at the center of the strand to produce a cross-sectional fracture surface. This surface was subsequently examined using a scanning electron microscope (SEM, FEI 200F) after sputter coating with gold priorly. Three types of PE/paraffin blends were compared. PE crystallization in PE/paraffin blends was observed with a polarized optical microscopy (POM). Samples were isothermally crystallized at a specific temperature for each formulation. Images were collected after the crystals formed completely.

2.3.3. Paraffin leakage test

Leakage tests for PCM materials were thoroughly researched in the literature. The methods described to detect or measure leakage amount are very limited and all fall within 3 different types: (1) by keeping in heated oven on top of filter papers and recording weight change [19,20], (2) by simple observation [15,33], (3) by observation under repeated cooling and heating cycles [9]. The advantages and disadvantages of the three methods include different standards of different researchers and time consuming of observation, simplicity of observation and so on. We selected the following method because it is a quantitative method to characterize the changes of the materials during PCM seepage. Leakage test can be conducted at elevated temperatures to reduce testing time. To investigate the amount of PCM leakage M_t at time t , we placed composites specimens ($0.5 \times 0.5 \times 0.125$ -in) in an oven maintained at 60 ± 1 °C on 10 layers of filter paper (United #1). Sample mass was recorded using a Mettler Toledo AG204 scale at 6-h intervals and replacing the filter papers following each weighing.

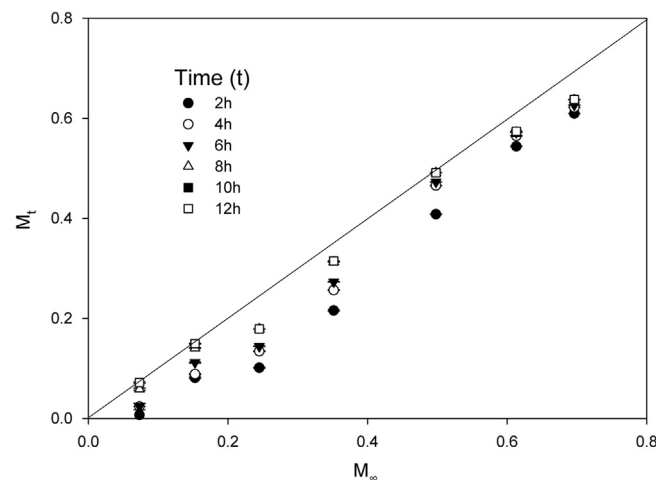


Fig. 1. The variation of total mass fraction of paraffin loss during chloroform extraction (M_t) at time t in a water bath of 40 °C with the total paraffin mass fraction incorporated into the composite (M_∞).

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