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Shape-Stabilized Phase Change Materials: Frozen Gel fom Polypropylene and *n*-Octadecane for Latent Heat Storage

Tae-Won Son,[*1] Tae-Hun Kim,[2] Bong-Sick Kim,[3] Byung-Giu Kim[2]*

Summary: We prepared polymer-PCM gels such as prepared frozen gel from polypropylene and n-octadecane for thermal storage and release materials, their basic properties and possible applications especially in latent heat storage. The preparation methods are used to melting method and absorption method respectively. The composition and properties of prepared frozen gels from polypropylene and *n*-octadecane were observed by DSC, WAXD, FT-IR spectra, ARES and Elemental analysis. We can prepare frozen gels in different temperature for latent heat storage materials as controlling composition of phase change material as well as using different incorporating phase change materials. These frozen gels can be used to latent heat storage materials for several applications.

Keywords: frozen gel; heat of fusion; latent heat; *n*-Octadecane; PCM

Introduction

Significant research has been done in the last few years about the preparation of shape-stabilized frozen gels from phase change material (PCM) and polymers.[1] Heat is absorbed or released during the phase change process, but temperature keeps almost stable. This heat called latent heat. PCMs of an alkane allow for storage and transport of large quantities of thermal energy by simple techniques since the absorbed latent heat cannot be lost by conduction, convection or radiation.[2] During a Phase change, the amount of heat proportion to the mass and latent heat of the materials. In the word of latent heat which means hidden, so somebody call the latent energy as a hidden energy. And the latent heat depends on the substance and the nature of the phase change, so such a frozen gel can be used in building materials for the generation of thermal insulating and waste energy recycling.[3] In our works, n-octadecane and a polymer, Polypropylene, were mixed together to form a shape-stabilized phase change material called 'frozen gel'. The gel compounds of polymer and PCM are thought to have low density and high thermal storage capacity. It can be used to consumer products in the form of fibers, fabrics and clothing or formed as food or beverage containers.[4] In this research we prepared some polymer-PCM gels such as prepared frozen gel from polypropylene and n-Octadecane for thermal storage and release materials, and examined their basic properties and potential application fields especially in latent heat storage.

[1] Department of Textile materials and chemistry, Fiber Science., School of Textiles, Yeungnam University, Gyeongsan, Gyeongbuk 712-749, South Korea
Fax: 82-53-815-6876
E-mail: twson@yu.ac.kr
[2] Department of Textile Engineering, Graduate School, Yeungnam University, Gyeongsan, 712-749, Korea
[3] School of Chemical Engineering and Technology Yeungnam University, Gyeongsan 712-749, Korea

Experimental Part

Materials

Moplen HP653P (chemical pure; PolyMirae, Korea), Moplen HP563S (chemical pure; PolyMirae, Korea), PP 5014 (chemi-

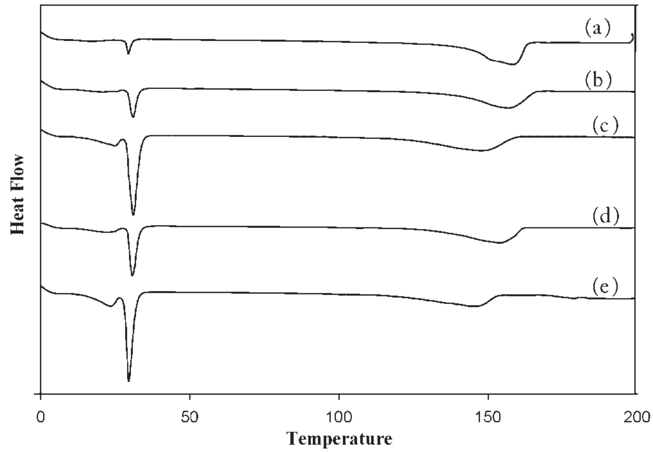

Figure 1.
DSC Thermo-diagrams of PP(MI17)/PCM(*n*-octadecane); (a) *n*-octadecane 10%, (b) *n*-octadecane 20%, (c) *n*-octadecane 30%, (d) *n*-octadecane 40%, (e) *n*-octadecane 50%.

cal pure; PolyMirae, Korea), *n*-octadecane (chemical pure; Tokyo kasei kogyo, Japan).

Preparation of PP-PCM Composite

The preparation methods are used to melting method and absorption method, respectively. In melting method, the reaction mixture of two different kinds of polypropylene chip and one kinds of phase change materials such as normal octadecane. And the reaction mixture is heating up under nitrogen gas atmosphere. The reaction mixture in the reaction container was heating up the reaction mixture to 200 °C for 2 hour. The mixing time of lab scale preparation should be provided quit long, instead of the short working time in a compounder vessel. Melting point *n*-octadecane in this experiment is 28 °C, and melting temperature of polypropylene is around 154 °C. Used polypropylenes for melting method are chip type polypropylene with

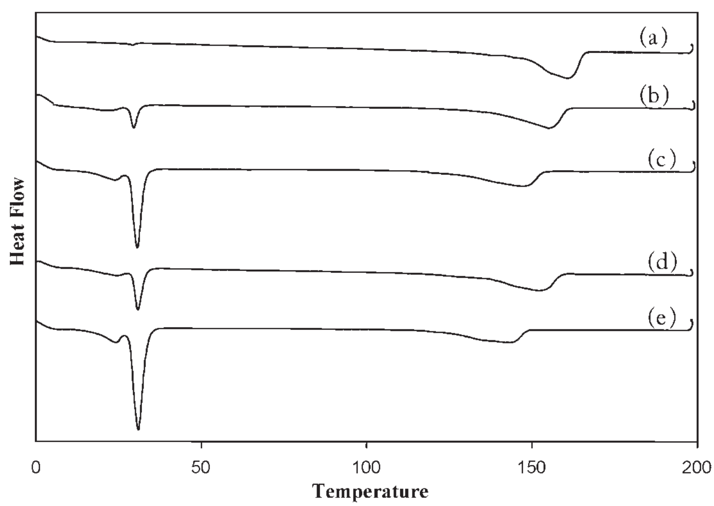

Figure 2.
DSC Thermo-diagrams of PP(MI38)/PCM(*n*- octadecane); (a) *n*-octadecane 10%, (b) *n*-octadecane 20%, (c) *n*-octadecane 30%, (d) *n*-octadecane 40%, (e) *n*-octadecane 50%.

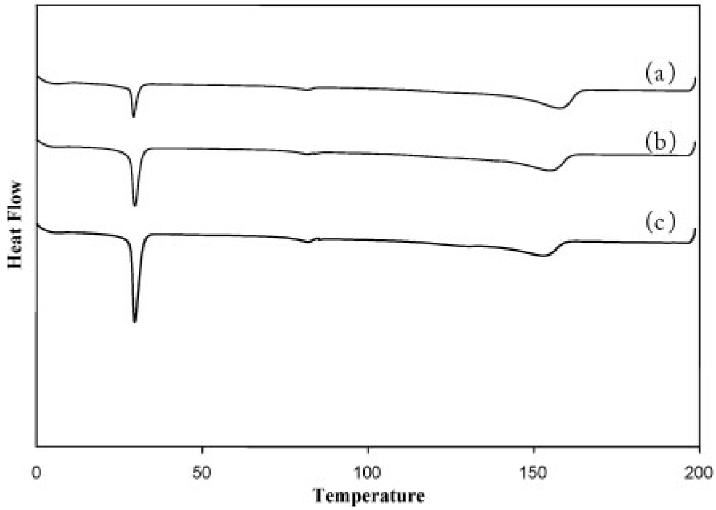

Figure 3.
DSC Thermo-diagrams of PP(MI4.8)/PCM(*n*-octadecane); (a) *n*-octadecane 10%, (b) *n*-octadecane 20%, (c) *n*-octadecane 30%.

melt index (MI 17) and 37 respectively, so the reaction mixture was heating up to 200 °C to melt down the reaction mixture perfectly. After completing the reaction, the reaction mixture is cooling down to room temperature. A melting method of frozen gel with 50/50 weight ratio of polypropylene-normal octadecane was prepared by adding polypropylene chip and normal octadecane. In absorption method, polypropylene powder was mixed in the molten phase change materials with stirring at relatively low temperature. The PP-PCM mixture in the reaction container was heating up the mixture around 60–80 °C for 12 hour. In here, powder shape polypropylene with melt index (MI 4.8) was used. We mixed up polypropylene powder and *n*-octadecane, respectively. An absorption method of frozen gel with 70/30 weight

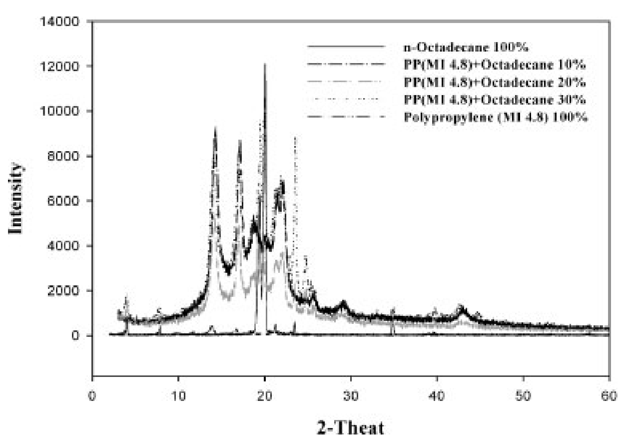

Figure 4.
Equatorial scanning wide angle X-Ray diffractograms of *n*-octadecane, Polypropylene (MI4.8) and PP/PCM composite(10–30%).

ratio of polypropylene 4.8-normal octadecane was prepared by adding polypropylene powder and normal octadecane.

Results and Discussion

DSC Thermograms of Frozen Gels PP/PCM Composite

Thermal properties of frozen gels via melting method are measured by using differential scanning calorimetry with ramping rate 5.0 K/min.

And the line shows a thermogram of frozen gel from melting and absorption method using polypropylene chip 10–50 wt% and polypropylene powder 10–30 wt% of normal octadecane as phase change material. As shown in here, we can see two melting points around 28.0 °C and 154 °C. and we measured heat of fusion from low temperature region that comes from normal octadecane because only this heat of fusion is important for latent heat energy storage for application. Also we can prepare frozen gels having different thermal properties with controlling compositions of phase change materials.

XRD Prepared Frozen Gels PP/PCM Composites

In order to confirm the incorporation of phase change materials into biding polymer by melting method, X-ray diffraction patterns of polypropylenes, phase change materials, and prepared frozen gels were measured.

In order to confirm the incorporation of phase change materials into biding polymer by melting method. This figure shows X-ray diffraction patterns of PP5014 powder, n-octadecane and obtained frozen gel having 10–30 wt% of n-octadecane in polypropylene powder with melting index 4.8. Prepared frozen gels were measured. As shown in a pure n-octadecane, n-octadecane X-ray diffraction patterns are characterized by major reflections at low 2θ below 20° and the X-ray diffraction patterns appeared around 10° and 30°.

FT-IR Spectra Prepared Frozen Gels PP/PCM Composite

In order to confirm the incorporation of phase change materials into binding polymer by absorption method, infra-red spectroscopy of polypropylenes, phase change

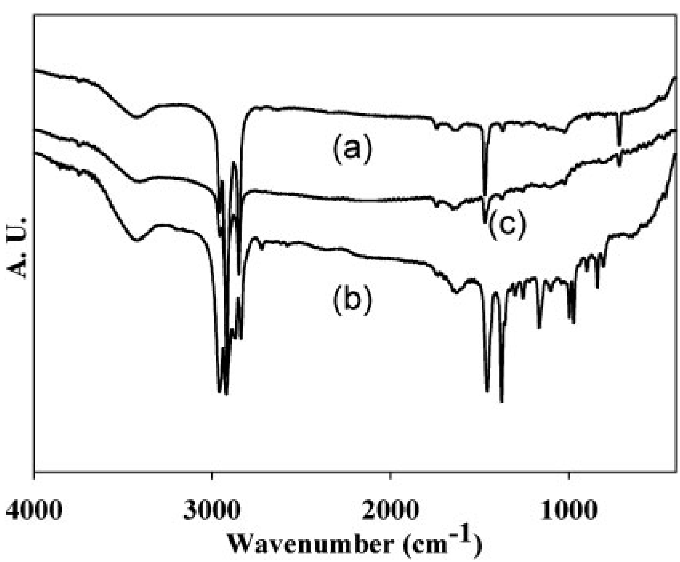

Figure 5.
FTIR spectra of *n*-octadecane, Polypropylene (MI4.8) and PP/PCM composite: (a) n-octadecane, (b) PP, (c) PP/PCM composite.

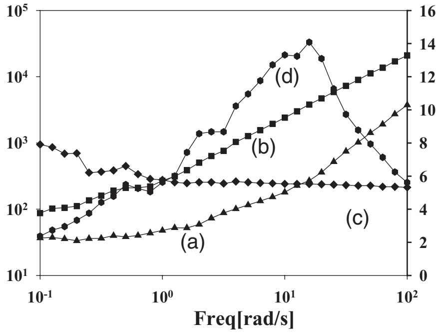

Figure 6.
Rheological properties of PP(MI38)/PCM(*n*-octadecane) 30% composite: (a) loss modulus G′[dyn/cm^2], (b) storage G″[dyn/cm^2], (c) viscosity Eta*[P], (d) Tan_-delta.

materials, and prepared frozen gels were measured. Obtained infra spectra were shown in Figure 5.

In this figure shows infra-red spectra obtained from polypropylene powder, normal octadecane and obtained PP4.8-normal octadecane frozen gel. As shown in this figure, we can see both characteristic peaks of polypropylene and normal octadecane in the frozen gel infra spectra obtained from absorption method using polypropylene and normal octadecane. From these infra-red spectra, the prepared PP4.8-normal octadecane frozen gel contains normal octadecane as phase change materials. We confirmed that phase change materials were successfully incorporated into the binder polymer from absorption method from infra spectroscopy results.

Rheological Properties of Frozen Gels PP/PCM Composite

In order to measure physical properties, dynamic measurements were carried out on advanced rheometric expansion system – ARES. ARES is synonymous with high performance rheological measurements.

Figure 6 show the obtained rheology properties of melting at 220°C of prepared frozen gels via melting method using polypropylene chip with melting index 38 and 30 wt% of normal octadecane as phase change material. From the ARES experiment, we can obtain the storage modulus and loss modulus. From the result, we can get tan delta value. The tan delta value can be obtained from storage modulus value divide loss modulus value. This graph shows obtained storage modulus and loss modulus and calculated tan delta at a time.

Morphology of PP/PCM Composites by Melting Method

The morphology of prepared frozen gels from melting method was measured by

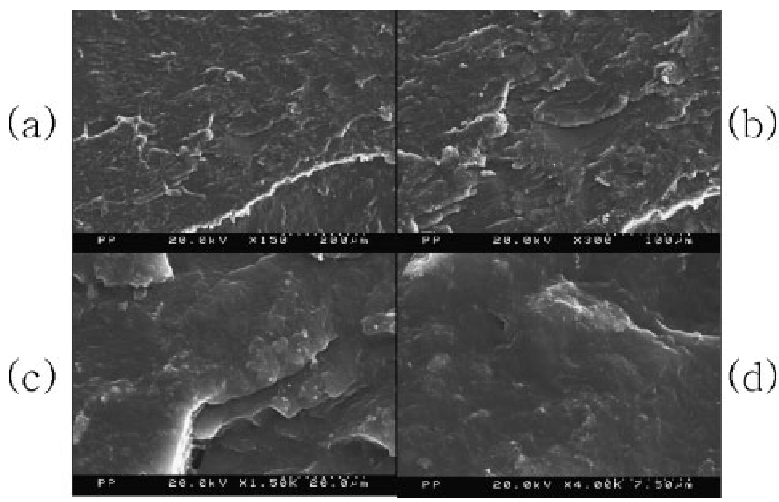

Figure 7.
SEM micrographs of cross-section of PP(MI17)/*n*-Octadecane composites: (a) PP/PCM composite (×150), (b) PP/PCM composite (×300), (c) PP/PCM composite (×1500), (d) PP/PCM composite (×4000).

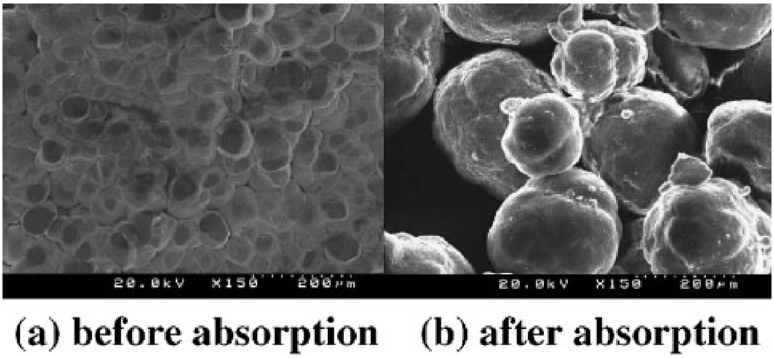

Figure 8.
SEM micrographs of surface of PP(MI4.8)/*n*-Octadecane composites: (a) Polypropylene(MI 4.8) powder (×150), (b) PP(MI 4.8)/PCM(*n*-octadecane) composite (×150).

using the scanning electron microscopy. Figure 7 shows scanning microscopy images of frozen gel after introducing normal octadecane as phase change material into polypropylene chip with melting index 17.

In this scanning electron microscopy, frozen gel is prepared by melting method. In here, I didn't prepare scanning microscopy image before melthing treatment, but the surface of the polypropylene is very smooth before treating. However, the surface of prepared frozen gel show very rough surface after treating by using melting method. We have measured all of frozen gels prepared by melting method.

Morphology of PP/PCM Composites by Absorption Method

Figure 8 show scanning microscopy images of frozen gel before and after absorption of normal octadecane as phase change material. In this scanning electron microscopy, frozen gel from absorption method uses polypropylene powder and normal octadecane.

This is a evidence of incorporated phase change material into the binder polymer from absorption method. We have measured all of frozen gels prepared by absorption. We have measured all of frozen gels prepared by absorption method. It means that our absorption method to prepare frozen gels is effective way to prepare materials for latent heat storage.

Conclusions

The composition and properties of prepared frozen gels from polypropylene and *n*- octadecane were observed by DSC, WAXD, FT-IR spectra, ARES and Elemental analysis. From the experimental results, we can prepare frozen gels in different temperature for latent heat storage materials as controlling composition of phase change material as well as using different incorporating phase change materials. These frozen gels can be used as latent heat storage materials for several applications.

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