Difunctional olefin block copolymer/paraffin form-stable phase change materials with simultaneous shape memory property

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ABSTRACT

In this work, a novel sort of form-stable phase change materials (FSPCMs) which possesses simultaneously the shape memory function was designed and prepared. The FSPCMs consist of paraffin with melting point of approximately 53 °C as a latent heat storage material and olefin block copolymer (OBC) as a supporting material. With mass percentage of paraffin up to 40 wt%, the FSPCMs exhibit good shape stability until temperature approaches 90 °C as shown by visual photographs, and this result is confirmed by dynamic mechanical analysis. At the same time, composites with 40 wt% paraffin also maintain the excellent mechanical property of OBC and exhibit large elongation at break as well as similar tensile stress, insuring excellent tenacity and deformation ability. The results of shape memory testing demonstrate that the composites possess good shape memory property, with nearly complete shape fixing and recovery. Compared with other reported FSPCMs, this material can be both temperature-controlled and temperature-sensitive, and may show advantages in some advanced applications such as intelligent textile. The diversity of paraffin endows the composites with flexibility of design for applications with wide range of temperatures. Considering the inexpensive sources and easy processing, this work may open up opportunities to produce difunctional FSPCMs in the industrial field.

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1. Introduction

As the level of greenhouse emission keeps increasing and the price of fuel climbs rapidly, great efforts have been made for effective utilization of renewable energy sources, such as solar energy. Among various methods, the latent heat storage based on fusion and freezing of phase change materials (PCMs) attracts great attention due to its high storage density and small temperature variation between storage and retrieval [1–3], and many potential applications have been studied, such as clothing, storage container, building materials and water tanks [2–5]. A series of inorganic and organic compounds as well as their mixtures have been studied as PCMs, including paraffin, which is widely used in latent heat thermal energy storage applications on account of its large latent heat and proper thermal characteristics such as little or no super-cooling, low vapor pressure, good thermal and chemical stability, and self-nucleating behavior [6–9]. However, PCMs based on paraffin often suffer from the disadvantage of liquid leakage when heated above the melting point, which would result in decrease of heat energy storage efficiency and pollution of application environment [10]. To solve this problem, many methods are developed for package. Among them, to construct form-stable phase change materials (FSPCMs) by blending PCMs with polymers is a widely used technique [11–14]. In this kind of FSPCMs, the polymer compound is considered as a supporting material and PCMs are dispersed into polymer network. Therefore, FSPCMs can keep their shape even when heated above the melting points of PCMs. The selection of supporting materials for specific PCMs is crucial to preparation of FSPCMs with high quality. Diverse polyolefins which possess good compatibility with paraffin, such as high density polyethylene (HDPE), were investigated as supporting materials [15–17], and some of them have been demonstrated to possess good package ability.

For FSPCMs, if the supporting materials possess phase separation morphology, which is essential for elasticity with regards to thermoplastic polymers, and the melting and crystallization of PCMs can bring about great variation of chain mobility of continuous phase, leading to temporary shape fixing and permanent shape recovery, the materials may also be provided with shape memory property [18–22]. Shape memory refers to the property that the material can be deformed and subsequently fixed into a temporary shape, which would remain stable unless an appropriate external stimulus triggers the material to recover to its permanent shape. Great interests have been attracted to the research of shape memory polymers (SMPs), which can be attributed to their promising potential applications in foams, textiles, morphing structures, deployable structures and biomedical devices [23–28]. For thermoplastic SMPs, phase separation
morphology, which accounts for the physical crosslinked network, is essential for recovery performance, but this requirement excludes most polyolefins that have been investigated as supporting materials so far. As FSPCMs have the ability of controlling temperature to some extent, while shape memory materials with heat as stimulus can be temperature responsive, we can obtain materials that are both temperature-controlled and temperature-sensitive by the combination of these properties, which may show great advantages in some advanced applications. However, to our knowledge, little exploration of this kind of interesting materials has been made up to now.

Recently, the Dow Chemical Company synthesized olefin block copolymer (OBC) by chain-shuttling technology in a continuous way [29–35]. The unique block architecture and outstanding mechanical property make it excellent candidate to construct olefin-based FSPCMs with shape memory property. This new type of multi-block copolymers comprise crystallizable ethylene/octane blocks with very low octane content, alternating with amorphous ethylene/octane block containing high octane concentration. It has a statistical multi-block architecture with a distribution in block length and blocks per chain, which differentiates it from anionically polymerized and hydrogenated olefin block copolymers. Because of the unique block structure, OBC exhibits mesophase separation morphology [31,36,37]. The crystallizable blocks assemble into dispersed phase, which acts as netpoints of physical crosslinked network, while the amorphous blocks conglomerate into continuous phase. The special architecture also imparts interesting mechanical property, such as a superior balance between flexibility and heat resistance, significantly improved compression set and elongation at break as well as high elastic recovery [38–41]. The similarity of chain structures between OBC and paraffin provides the possibility of OBC to be utilized as supporting materials. The paraffin can be stored in the amorphous blocks and the storage ability may be adjusted by altering the content. Moreover, as OBC presents phase separation morphology, and the melting and crystallization of paraffin can lead to a huge variation of chain mobility of amorphous phase, the OBC/paraffin composites may also possess shape memory property. Through adjusting the mass ratio between OBC and paraffin, this kind of composites may partly maintain outstanding mechanical property of OBC, insuring that the composites can be deformed into variable temporary shapes while exhibit excellent recovery performance.

In this work, we first introduced a novel sort of polyolefin-based FSPCMs combined with shape memory property, in which OBC acted as supporting materials, while paraffin was utilized as both PCMs and switch for shape changing. It can be inferred from our results that, with appropriate ratio between OBC and paraffin, the composites can not only store energy, but also exhibit excellent shape memory property, with fixing and recovery ratio approaching 100%. In this way, this material can be both temperature-controlled and temperature-sensitive, considering the diversity of paraffin, the OBC/paraffin composites can be designed to apply to circumstances with wide range of temperatures. Furthermore, the raw materials used for preparing the composites are all industrial supplies with low cost, and the composites are easy post-processing by existed apparatus. Therefore this sort of material has possibility of large-scale fabrication and may enlarge potential application range of OBC.

2. Experimental

2.1. Materials and sample preparation

The paraffin with a melting temperature $T_m = 52–54 \, ^\circ C$ was purchased from Sinpharm Chemical Reagent Company (Shanghai, China), and chosen as phase change material. OBC material synthesized by chain-shuttling technology is a commercial grade product produced by Dow Chemical Company; it has a density of 0.887 g/cm$^3$ and a melt flow rate of 5.0 g/10 min (230 $^\circ C$, 2.160 kg).

The OBC and paraffin granules were melt-blended by a Bra-bender Mixer (PLE651) at a roller speed of 60 rpm and a mixing temperature of 160 $^\circ C$ for 6 min. The FSPCMs samples containing 100, 70 and 60 wt% of OBC were prepared and denoted as OBC-100, OBC-70, OBC-60, respectively. The neat paraffin was denoted as OBC-0. All samples were compression molded to films at 160 $^\circ C$ for the subsequent characterization.

2.2. Characterizations

A Mettler DSC-821e apparatus was used to evaluate the thermal property of the OBC/paraffin FSPCMs. The measurements were performed at 10 $^\circ C$/min. The crystallization temperature ($T_c$) and the melting temperature ($T_m$) of paraffin were taken at the liquid-rotator transition peak, while latent heat ($\Delta H_m$) was determined from five cycles. To characterize the stability of our composites visually, we carried out paraffin leaching experiments. The sample strips placed in small glass bottles were heated from 60 to 100 $^\circ C$ at an interval of 10 $^\circ C$ and held there for 20 min, after that the photographs of sample strips at corresponding temperatures were taken and comparisons were made. The samples of OBC-60 treated at 80 $^\circ C$ for different times were characterized by differential scanning calorimetry (DSC), and corresponding visual photographs were taken as well. The thermal stability of the composites was examined by thermal gravimetric analyzer (TGA, Perkin-Elmer) in nitrogen atmosphere with a heating rate of 20 $^\circ C$/min. Dynamic mechanical analysis (DMA) testing was carried out using a DMA Q800 analyzer (TA instruments, USA). The tensile mode was used, and the measurement was carried out on a rectangular shaped part in the size of 10 mm × 10.2 mm × 4.2 mm (length × width × thickness) from −40 to 100 $^\circ C$ at a heating rate of 3 $^\circ C$/min and an oscillatory frequency of 1 Hz. The tensile testing was conducted on a SANS CMT-6503 universal testing machine (Shenzhen, China) with a crosshead speed of 50 mm/min. The capacity of the load cell is 50 N in axial load. Morphologies of OBC/paraffin composites were observed using a DM2500P polarized optical microscope (POM) with Linkam-THMS600 hot stage. Samples were melted at 160 $^\circ C$ and cooled to a desired temperature of 50 $^\circ C$/min and kept at this temperature for 5 min to allow complete melting. Samples were subsequently cooled to a desired temperature at 20 $^\circ C$/min.

Quantitative shape memory testing was carried out by the following procedure: the rectangular sample with a length of 10 mm was immersed into water-bath under a controlled constant temperature of 70 $^\circ C$ and stretched by a strain of 100% ($e_m$). Then the sample was cooled to 0 $^\circ C$ while holding the stress constant for 5 min. The stress was quickly released when the sample was in the stretched state to witness the strain fixing, and the fixed strain ($e_f$) was recorded. The unconstrained strain recovery was triggered by immersing the sample again into a water bath of 70 $^\circ C$ and the resultant strain was obtained ($e_p$). Meanwhile, the recovery time was recorded as soon as the temporariness shape-fixed sample was immersed into the water-bath. When the whole recovery process finished, the time consumed was determined to be the recovery time. The fixing ratio ($R_f$) and recovery ratio ($R_r$) were calculated according to the following equations, where $N$ corresponds to the cycle number.

$$R_f(N) = \frac{e_f(N)}{e_m(N)} \times 100\% \quad (1)$$
$$R_r(N) = \frac{e_m(N) - e_p(N)}{e_m(N)} \times 100\% \quad (2)$$
3. Results and discussion

3.1. Thermal property and shape stability characterization

The heating and cooling thermograms of paraffin, OBC and OBC/paraffin FSPCMs are shown in Fig. 1. In order to maintain the mechanical property of OBC and achieve good shape memory performance, the maximum mass percentage of paraffin is restricted to 40 w%. The melting peaks located above 110 °C can be attributed to OBC, which determine the limitation of application temperatures. The DSC endotherm of pure paraffin exhibits two well-defined separated peaks (Fig. 1(a)). The first melting peak located in the temperature range from 23 to 37 °C should be attributed to the homogeneously nucleated crystal-rotator transition and the second peak located in the range from 43 to 57 °C may be attributed to the heterogeneously nucleated rotator-liquid transition [42–45]. Similar phenomenon can also be found in cooling thermograms (Fig. 1(b)). With regards to OBC/paraffin FSPCMs, the separated peaks still exist, but they become inconspicuous. The melting and crystallization parameters of all the samples including the melting peak temperature, crystallization peak temperature, and the latent heat, which were determined from DSC curves, are summarized in Table 1. The results exhibit that the endothermic peaks and exothermic peaks of OBC/paraffin composites both shift to lower temperatures with increase of OBC content, and the melting enthalpy of paraffin encapsulated in OBC becomes approximately 160 J/g, which is lower than pure substance. The differences between neat paraffin and OBC/paraffin composites may be attributed to that the three-dimensional network structure formed by OBC confines the thermal molecular movement of the paraffin during phase change [46].

In order to validate the shape stability of OBC/paraffin FSPCMs during phase transition, and explore the highest enduring temperature roughly, the samples were heated from 60 to 100 °C at an interval of 10 °C and held at corresponding temperatures for 20 min to insure an equilibrium state, after that the photographs were carefully taken for comparison. As shown in Fig. 2, the composites hold the shape of strip at room temperature. When heated to 60 °C, both samples maintain the original shape. This shows that the introduction of OBC endows the composites with the ability to keep stable shape even when paraffin melts. When temperature continues increasing to 80 °C, the composites still exhibit excellent shape stability, only a slight bending can be observed, which may be ascribed to softening of the samples. However, when temperature further increases to 90 °C, both samples show obvious folding, and the bottom parts become thick resulting from flowage of paraffin. This phenomenon indicates that OBC failed to encapsulate paraffin completely and the OBC/paraffin FSPCMs had better to be utilized under 90 °C.

With the understanding that the composites can keep good shape stability at 80 °C, the influence of heating treatment of 80 °C was investigated to confirm the persistence of stability. The heating and cooling thermograms of OBC-60 with different times of heating treatment at 80 °C are illustrated in Fig. 3, and the corresponding data of thermal property are summarized in Table 2. It is intriguing that there are no appreciable variations before and after heating treatment at 80 °C for Tm and Tc, while ΔHm increases slightly after heating treatment. This may be ascribed to that heating treatment decreases the confinement of OBC networks on paraffin movements to some extent, but the effect is very weak and ΔHm becomes stable with further increase of treatment time. The results suggest that heating treatment at 80 °C has little influence on thermal property of the composites. In order to further validate the shape stability at 80 °C, the visual photographs of OBC-60 with different heating treatment times were taken as well. As shown in Fig. 4, although placed at 80 °C for 2 h, there is still no obvious shape change apart from some slight bending, which can be attributed to softening of the composites, and no free paraffin can be found at the bottom of the bottle. We can conclude from the above results that heating treatment at 80 °C does not change the thermal property as well as the apparent shape, insuring the FSPCMs can be utilized repeatedly with temperature up to 80 °C.

3.2. Thermal stability characterization

The thermal stability of the composites was evaluated by TGA in nitrogen atmosphere. The results are exhibited in Fig. 5. It can be found from TGA curves that both paraffin and OBC degrade in one step. The degradation of paraffin mainly occurs at the temperature range from 190 to 300 °C, while no obvious degradation occurs for neat OBC below 400 °C. For OBC/paraffin composites, the degradation turns into a two-step process. The first degradation step takes place from 200 to 340 °C, which can be attributed to the degradation of paraffin; and the second step occurring above 400 °C is caused by the degradation of OBC. Although the degradation behavior varies for different composites, all the samples maintain excellent thermal stability below 90 °C.
which suggests that the degradation can be neglected before leakage of paraffin.

3.3. Polarized optical microscopy observation

Both OBC and paraffin are crystalline materials and exhibit strong birefringence under polarized light. Therefore POM can be used to observe the morphologies of OBC/paraffin FSPCMs as well as the dispersion of paraffin in OBC matrix. Fig. 6 shows the pictures of OBC-100, OBC-70 and OBC-60. As can be seen from the pictures, the spherulite crystallization of OBC dispersed uniformly as island phase, while the amorphous blocks assemble into continuous phase between spherulites. The phase separation morphologies insure good elasticity of OBC/paraffin composites [20,25,47]. As for OBC-100, no change can be found when sample is cooled from 80 to 30 °C. However, with the introduction of paraffin, space between spherulite crystallization of OBC brightens obviously after cooled to 30 °C, which is low enough for the complete crystallization of paraffin. The results indicate that a great deal of paraffin is stored in amorphous blocks. Thus the crystallization and melting of paraffin may lead to a huge variation of chain mobility of continuous phase, suggesting the feasibility of shape memory property. We can also discover that the accession of paraffin cause a serious decrease of the spherulite size, and this may be ascribed to that the paraffin dispersed in amorphous blocks can retard aggregation of crystalline blocks to some extent. Interestingly, ringed spherulite was found in OBC/paraffin composites, and we will investigate this phenomenon intensively in our later work.

![Fig. 2. Photographs of OBC-60 (the sample at left side of each photograph) and OBC-70 (the sample at right side of each photograph) at room temperature, 60 °C, 70 °C, 80 °C and 90 °C.](image)

![Fig. 3. DSC (a) heating curves and (b) cooling curves of OBC-60 before and after heating treatment at 80 °C.](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Time of heating treatment (h)</th>
<th>Tm (°C)</th>
<th>Tc (°C)</th>
<th>ΔHm (J/g)</th>
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Table 2
Thermal property of OBC-60 before and after heating treatment at 80 °C.
3.4. Mechanical property characterization

The change of mechanical property with temperature for our FSPCMs was investigated by DMA. As exhibited in Fig. 7, no abrupt transition can be found in our testing temperature range for OBC-100. With regards to OBC-60 and OBC-70, both samples show much higher storage modulus than OBC-100 resulting from the introduction of paraffin, which is more rigid than OBC elastomer in solid state. Meanwhile, OBC/paraffin composites display two distinct transitions. One located in the range approximately from 40 to 60 °C corresponds to the melting of paraffin, the storage modulus decreases rapidly from about 150 MPa to the value of below 10 MPa. The great decrease of storage modulus indicates a huge elevating of chain mobility [47,48], which may account for the shape recovery from temporary shape to permanent shape. It should be noted that the storage modulus detected by DMA is provided by OBC above 60 °C. Therefore it shows a strong dependence on the OBC content. When OBC loading decreases, the modulus referred to the physical network of OBC will decrease correspondingly, while this modulus is closely related to the recovery force which drives the recovery process. The other transition takes place when the temperature approaches 90 °C, the storage modulus of OBC/paraffin FSPCMs drops tremendously. This may be attributed to paraffin flowage, which can result in inhomogeneity of the sample and thus destroy the network to some extent. The result is in consistent with our shape stability testing and confirms that the OBC/paraffin FSPCMs should be utilized under 90 °C.

Besides the small-strain dynamic mechanical behavior, the large-strain tensile tests have also been carried out and the results are illustrated in Fig. 8. OBC-100 displays a typical elastomeric characteristic with no distinctive yield point. The elongation at break is close to 1800%, demonstrating excellent tenacity and deformation ability. Comparatively speaking, paraffin is a stiffer material over OBC, incorporating this component endows the composites with small yield point, while the overall behavior is still elastomeric, retaining low tensile stress. As for OBC-70, adding 30 wt% paraffin does not induce an obvious variation for tensile property, only with the tensile stress elevated to some extent at low strain ratio. When the mass ratio of paraffin increases to 40 wt %, the elevation of tensile stress at low strain ratio become limited, while tensile stress at large strain ratio and elongation at break begin to exhibit a decreasing tendency. This may be attributed to paraffin flowage, which can result in inhomogeneity of the sample and thus destroy the network to some extent. The result is in consistent with our shape stability testing and confirms that the OBC/paraffin FSPCMs should be utilized under 90 °C.

3.5. Shape memory property characterization and potential application

Quantitative evaluation of shape memory behaviors has been conducted according to previous literatures [22,49]. Samples were stretched to a strain of 100% under external stress after which fixing and recovery process was investigated. The shape memory property of the composites represented by shape fixing ratio ($R_f$), shape recovery ratio ($R_r$), and recovery time of five cycles was summarized in Table 3 (the calculation equation of $R_f$ and $R_r$ can be found in the experimental section). It shows that OBC-100 possesses a bad shape memory fixing performance with only 40% elongation preserved after the release of external stress. Apparently, lacking of switch which responses to the decrease of temperature is the main reason for this bad shape memory fixation. After incorporating paraffin as switch phases, OBC-70 sample exhibited a greatly improved fixing performance, as plenty of paraffin dispersed homogeneously in continuous phase and the crystallization process can bring an effective constrain of the relaxation of deformed OBC matrix. The fixed ratio of OBC-70 reaches 90% and nearly no decrease of recovery ratio is detected.
With elevated paraffin concentration, the fixing performance of OBC-60 is further optimized, while a nearly complete recovery can still be achieved from the second cycle, manifesting perfect shape memory property. The data also illustrate that the shape fixing and recovery performance of OBC/paraffin composites is fairly stable in our testing cycles, although the processing history and other factors can lead to irreversible deformation in the first cycle. The stability of shape memory property guarantees that the composites can be used repeatedly. Furthermore, all samples complete recovery process in 5 s, showing good thermal sensitivity, which is an important advantage in some practical applications.

As the OBC/paraffin composites possesses the ability of energy storage and shape variation at the same time, the combination is sure to show advantages in some specific aspects. One important potential application may be the intelligent textile. Though the process of energy storage and retrieval, the textile comprising OBC/paraffin composites can regulate temperature and preserve heat to some extent. Meanwhile, shape memory property also endows textile with some interesting features. If the transition temperature ($T_{trans}$), which corresponds to the shape fixing and recovery, is lower than room temperature, the textile tends to exhibit soft texture as well as strong elasticity to resist wrinkle. As for materials with $T_{trans}$ higher than room temperature, they give a hard-hand feel and are appropriate to fabricate skirt and western style suit, which require that the shape of clothes is relatively fixed. Moreover, this kind of clothes can recover to original shape automatically after immersed into hot water. Therefore the wrinkles can be erased easily, just as illustrated by Fig. 9, the

<table>
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<th>Sample</th>
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folded square slice would nearly recover to the permanent shape completely after immersed into water bath of 60 °C, which is higher than \( T_{\text{m,n}} \) of our OBC/paraffin composites. Considering the diversity of paraffin, the OBC/paraffin composites may be designed to apply to various application circumstances by utilizing paraffin with different melting points as well, which suggests great application prospects.

4. Conclusions

In this work a novel kind of FSPCMs with shape memory property, which consist paraffin with melting points of approximately 50 °C as latent heat storage material and OBC as supporting material, was prepared. As OBC presents phase separation morphology, and the melting and crystallization of paraffin can lead to a huge variation of chain mobility of continuous phase, the OBC/paraffin composites can exhibit excellent shape memory property. According to the results of visual photographs and DMA, the shape of FSPCMs with paraffin concentration up to 40 wt% can keep stable until temperature reaches around 90 °C. The tensile testing confirms that composites can also maintain the excellent mechanical property of OBC with large elongation at break higher than 1500%, which is an important character in the application of films and textile. The results of shape memory testing demonstrate that the composites are provided with ideal shape memory property, with shape fixing and recovery ratio both approaching 100% for OBC-60. With the ability of energy storage and shape variation at the same time, this material may show advantages in some advanced applications, and the diversity of paraffin endows the composites with flexibility of design for different applications. Furthermore, the raw materials used for preparing the composites are all industrial supplies with low cost, and the composites are easy post-processing by existed apparatus, so this material has possibility of large-scale fabrication.

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