

## Material properties

## Crystallization kinetics, morphology, and mechanical properties of novel poly(ethylene succinate-co-octamethylene succinate)



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## ARTICLE INFO

## Article history:

Received 26 August 2015

Accepted 5 October 2015

Available online 8 October 2015

## Keywords:

Crystallization kinetics

Morphology

Mechanical properties

Poly(ethylene succinate-co-octamethylene succinate)

## ABSTRACT

The crystallization kinetics, morphology and mechanical properties of a novel poly(ethylene succinate-co-octamethylene succinate) (PEOS) copolyester with 82 mol% ethylene succinate (ES) units and 18 mol% octamethylene succinate (OS) units, and its homopolymer poly(ethylene succinate) (PES) were extensively investigated. The glass transition temperature, cold crystallization peak temperature and melting point of PEOS are around  $-24$ ,  $47.5$ , and  $80.5$  °C, respectively. The Avrami equation was used to analyze the isothermal melt crystallization kinetics of PEOS and PES. They display the same crystallization mechanism, and PEOS crystallizes slower than PES at the same degree of supercooling. The spherulitic growth rates of PEOS and PES exhibit a bell shape within the investigated crystallization temperature range, with the crystallization regime transition temperature of PEOS being lower than that of PES. In addition, PEOS has high thermal stability and good mechanical properties.

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## 1. Introduction

Poly(ethylene succinate) (PES) has attracted lots of attention in recent years due to its favorable mechanical properties and relatively high melting temperature [1–3]. The crystallization behavior, morphology, degradation, crystal modification and mechanical properties of PES have been widely investigated [4–13]. Blending, compounding and copolymerization are usually used to improve the physical properties of polymers [14–18], and PES has been blended with many polymers to extend its practical field. PES is miscible with poly(ethylene oxide), poly(vinyl phenol), tannic acid, poly(*p*-dioxanone) and poly(3-hydroxybutyrate) [19–24], and partially miscible with poly(vinyl chloride) and poly(butylene succinate) [25,26]. However, PES is immiscible with most polymers, for example poly(L-lactide) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) [27,28].

Biodegradable copolyesters, usually obtained via a copolymerization method, are regarded as promising materials because their properties may be adjusted by combining different chemical structures. In most cases, copolymer has a faster degradation rate

than the homopolymer, which can be attributed to the reduced crystallinity, because the amorphous region of semicrystalline polymers usually degrades more easily than the crystalline region [29,30]. In literature, many PES based copolymers have been synthesized, such as poly(ethylene succinate-co-ethylene adipate), poly(ethylene succinate-co-butylene succinate) and poly(ethylene succinate-co-trimethylene succinate) [31–34].

To our knowledge, the crystallization kinetics, morphology and mechanical properties of poly(ethylene succinate-co-octamethylene succinate) (PEOS) have not been reported. Therefore, in the present work, the crystallization kinetics, morphology and mechanical properties of a novel PEOS copolymer with 82 mol% ethylene succinate (ES) units and 18 mol% octamethylene succinate (OS) units were investigated in detail. The reason for choosing this PEOS copolyester with 18 mol% OS units is as follows. The composition of the OS units in the copolymer is 18 mol%, which is a moderate composition; therefore, it is still a semicrystalline polyester with a relatively high melting point; moreover, the mechanical properties of PEOS may include relatively high mechanical strength and elongation at break. Hence, this PEOS copolymer may have better balanced thermal and mechanical properties than other PEOS copolymers with low or high OS compositions. Consequently, it is expected that PEOS may find potential practical application as a novel biodegradable aliphatic polyester.

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## 2. Experimental

PEOS and PES were synthesized in our laboratory, and the details of the synthesis process were similar to those of our previous works [35,36]. Fig. 1 shows the chemical structures of PEOS and PES. The composition of PEOS is 82 mol% ES units and 18 mol% OS units, determined from the nuclear magnetic resonance ( $^1\text{H}$  NMR) spectra. The intrinsic viscosity ( $\eta$ ) values of PEOS and PES are 0.69 and 0.72 dL/g using chloroform as the solvent at 25 °C, respectively.

The diffraction patterns of PEOS and PES were obtained using an X-ray diffractometer (Rigaku D/Max 2500 VB2+/PC) from 5° to 45° at 5°/min at room temperature. The thermal properties of PEOS and PES were examined with a TA Instrument differential scanning calorimeter (DSC) Q100. Each sample was about 5 mg. For the nonisothermal cold crystallization, PEOS and PES were kept at 130 °C for 3 min to eliminate previous thermal history, cooled to -40 °C at 60 °C/min, and heated to 130 °C again at 20 °C/min. For the isothermal crystallization process, PEOS and PES were annealed at 130 °C for 3 min, cooled to the desired crystallization temperature ( $T_c$ ) at 60 °C/min, and reheated to 130 °C at 20 °C/min after the crystallization was completed. A polarized optical microscope (POM) (Olympus BX51) was used to investigate the crystalline morphologies and growth rates of PEOS and PES, which were first annealed at 130 °C for 3 min and quenched to the desired  $T_c$ . The thermal stability experiments were performed with a thermogravimetric analyzer (TGA) Q50 from ambient temperature to 580 °C at 20 °C/min under a nitrogen atmosphere. The mechanical properties of PEOS and PES were characterized by a universal tensile machine (Instron 1185) at 25 °C at a rate of 50 mm/min. At least three individual tests were performed for each sample, and the average taken.

## 3. Results and discussion

### 3.1. Crystal structure of PEOS

Wide-angle X-ray diffraction (WAXD) experiments were performed to study the crystal structures of PEOS and PES, which were crystallized at 50 °C for 24 h. The WAXD patterns are displayed in Fig. 2. PEOS exhibits three main diffraction peaks. PES displays the similar diffraction peaks at almost the same locations as PEOS, implying that the incorporation of OS unit does not change the crystal structure, and the OS units may exist in the amorphous region of PEOS. The three diffraction peaks from (021), (121), and (200) planes are at around 20.3°, 22.7° and 23.3°, respectively [37]. The crystallinity values of PEOS and PES were calculated to be 30% and 40%, respectively, from the WAXD patterns. In brief, on the basis of the WAXD study, PEOS and PES share the same crystal structure, but crystallinity of PEOS is smaller than that of PES because of its copolymer characteristics.

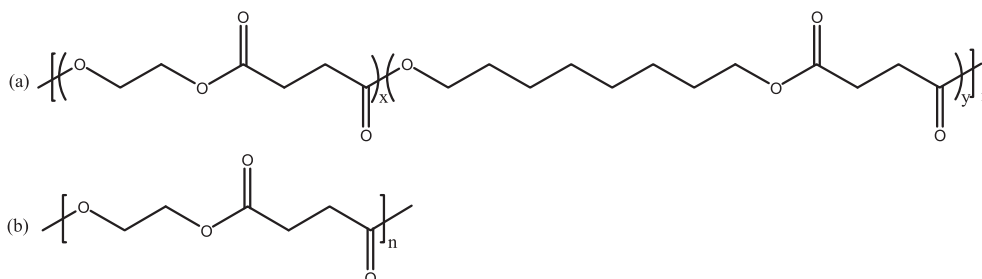


Fig. 1. Chemical structures of (a) PEOS and (b) PES.

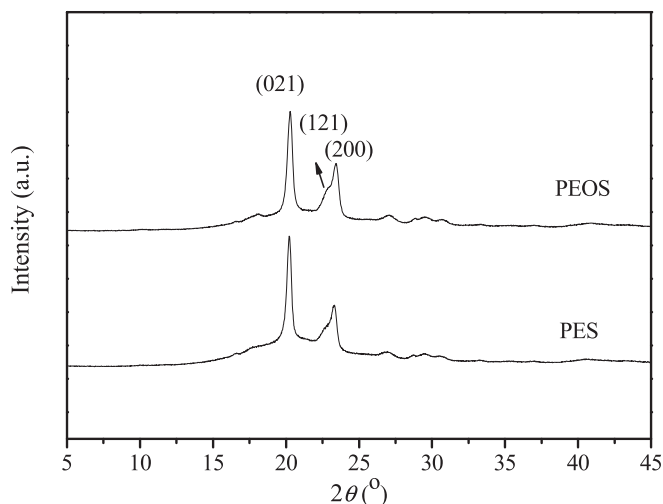


Fig. 2. WAXD patterns of PEOS and PES after crystallizing at 50 °C for 24 h.

### 3.2. Basic thermal properties of PEOS

Fig. 3 displays the DSC traces of PEOS and PES on the melt-quenched samples. As seen in Fig. 3, PEOS shows a glass transition temperature ( $T_g$ ) of about -24 °C, and the  $T_g$  value is around -10 °C for PES, indicating that the chain segments of PEOS are more flexible than those of PES. For PEOS, the intensities of the cold crystallization peak and the melting peak are weaker than those of PES. The corresponding data are summarized in Table 1.

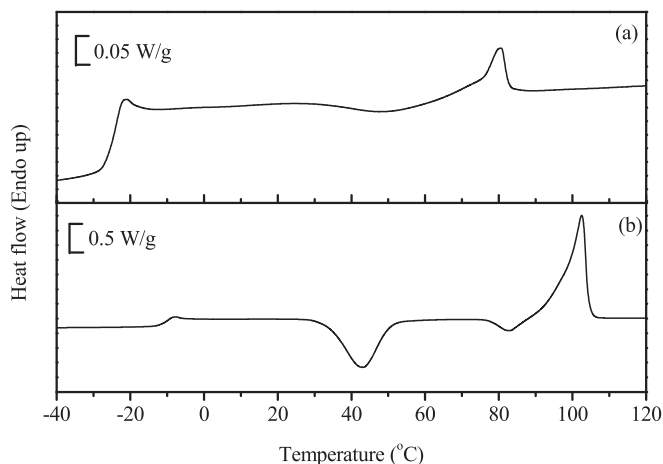


Fig. 3. DSC heating traces of (a) PEOS and (b) PES at 20 °C/min after quenching from the melt at 60 °C/min.

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