Poly(butylene succinate)-based Telechelic ionomers 의 비등은 결정화에 대한 연구

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Investigation of Non-isothermal Kinetics of Poly(butylene succinate)-based Telechelic Ionomers

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1. Introduction

Ionomers have been widely researched, since ionic aggregates induce to the change of significant physical properties such as enhanced mechanical properties and high melt rheological properties. Recently, we synthesized the biodegradable poly(butylene succinate) ionomers (PBS-is) and confirmed that they have remarkable potential due to the excellent physical properties and biodegradability [1]. Most investigations dealing with polyester-based ionomers have focused on random ionomers, in which ionic groups were randomly distributed as pendant groups, while a little investigation about the telechelic ionomer which possess with ionic group in both end functional groups has yet paid to attention [2]. Based on this, We prepared poly(butylene succinate)-based telechelic ionomers (PBS-Ts)by two-step polycondensation and investigated the overall properties for the novel telechelic ionomer as well as understanding of crystallization behavior under non-isothermal conditions. We expected that this research may become interesting foundation, which can compare random ionomers with telechelic ionomers about the physico chemical properties. Moreover, it can be anticipated that this approach can provide the fundamental information how ionic group regulates the non-isothermal crystallization behavior [3-5].

2. Experimental

2.1. Materials

Dimethyl succinate(DMS), 1, 4-butanediol(BD), and 3-sulfobenzoic acid monosodium salt(SBSS) were purchased Sigma Aldrich and used without further purification. Titanium tetrabutoxide (Aldrich) was also used as received.

2.2. Preparation of PBS - based Telechelic ionomers

Pure PBS and PBS-Ts cotaining 0.1, 0.5, 1.0 and 2.0 mol% SBSS were synthesized by 2 setp polymerization. First, monomer mixtures were melted and stirred at 190 $^{\circ}$ C for 5 hours to remove methanol condensate. After that, the reaction temperature was raised to 230 $^{\circ}$ C over a period of 30

min gradually applying a reduced pressure to remove oligomer and maintained until obtained polymer.

3. Results and discussions

From ¹H-NMR and ²³Na Solid state NMR, we confirmed that PBS-Ts were successfully synthesized in the presence of 3-sulfobenzoic acid monosodium salt (SBSS). PBS 0.1 ~ 2.0 revealed slightly changed in melting point with increasing of ionic group concentration. Nevertheless, the crystallization temperature were significantly reduced. The non-isothermal crystallization of pure PBS and PBS-T2.0 were measured by DSC at various cooling rates (5, 10, 30, 40 °C/min) in a nitrogen atmosphere. The development of the relative crystallinity with time for the above samples was shown in Figure 1. We observed the delay of the crystallization rate of PBS-T2.0 with ionic group, which means that the mobility of polymer chain is restricted by ionic aggregates.

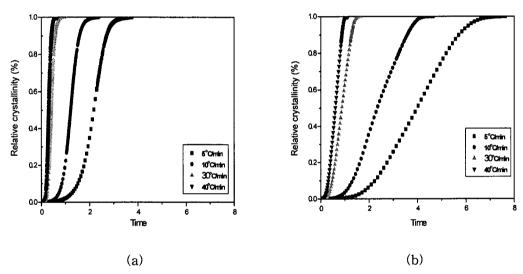


Figure 1. The development of the relative crystallinity as a function of crystallization time for PBS (a) and PBS-T 2.0 (b): cooling rate; 5, 10, 30, 40 °C/min

4. References

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