Crystallization and Recrystallization of PB-1: Impact of Sorbitol-Based Nucleating Agent

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Polybut-1-ene (PB-1) is semicrystalline polymer interesting for its polymorphic nature, undergoing phase transitions between different crystalline forms over time. Initially, PB-1 crystallizes into a kinetically preferred tetragonal phase (phase II), which gradually transforms into the thermodynamically stable hexagonal phase (phase I), especially under ambient conditions [e.g. 1]. This transition is of great interest due to its impact on material properties, e.g. mechanical strength or long-term stability. A deeper understanding of the crystallization and recrystallization kinetics of PB-1 is thus essential for optimizing processing conditions and enhancing their performance in practical applications.

The work is focused on crystallization and recrystallization of PB-1 homopolymer and random PB-1 copolymer with low content of ethylene. The effect of the addition of nucleating agent (NA) 1,2,3,4-bis(3,4-dimethylbenzylidene) sorbitol, commercial clarifying agent designed primarily for polypropylene (PP), Millad 3988, on the crystallization and subsequent recrystallization is studied, particularly in relation to the kinetics of the process [2]. The NA was applied in two common concentrations 0.2 and 0.6 wt. %. The recrystallization from tetragonal phase II into hexagonal phase I in defined times of ageing at room temperature was characterized by spectrophotometry, wide-angle X-ray scattering (WAXS) and differential scanning calorimetry (DSC). The structure was also obserd via polarized light and scannning electron microscopes.

It was found, that NA Millad 3988 is effective, particularly for the copolymer. The addition of 0.2 wt. % significantly increases the crystallization temperature, and, at the same time, narrows the crystallization peak. On the other hand, the haze increased dramatically in PB-1/NA systems. Thus, the NA used does not act as a clarifying agent and a two-phase system is formed. Under the given sample preparation conditions, NA did not form an organogel leading to a decrease in transparency as is commonly done in PP. It was found that at a concentration of 0.2 wt.% dissolution of NA in PB-1 does occur, but the melting temperature is crucial. As the temperature increases, the solubility also increases. The dissolved nucleating agent, however, was found to form rod-shaped crystals with a diameter of about 1 μm on cooling. These cause nucleation of PB-1. WAXS analysis revealed that NA accelerates recrystallization from phase II to I in homopolymer. In the case of the copolymer, the effect of NA is not as pronounced, however, it also interferes with the recrystallization process.

According to results, it can be confirmed that the addition of commercial NA Millad 3988 has a significant effect on the crystallization and recrystallization behavior of both the PB-1 homopolymer and the PB-1 copolymer, even at a concentration of 0.2 wt.%. Millad 3988 very significantly increases the rate of crystallization in copolymer while it considerably accelerates the recrystallization of homopolymer. However, the crystallization process may be affected by the solubility of NA in PB-1 melt at high processing (melt) temperatures at such low concentrations.

Keywords: polybut-1-ene, crystallization, recrystallization, nucleation

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