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Separation and identification of ethylene–propylene block copolymer

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In this paper, a commercial impact-resistant polypropylene alloy sample was fractionated with preparative temperature rising elution fractionation (t.r.e.f.) technique and a series of ethylene–propylene block copolymer fractions with different ethylene contents were obtained. Combined with fractionation results, the block structure of them was identified with ^{13}C nuclear magnetic resonance (n.m.r.) spectroscopy and differential scanning calorimetry (d.s.c.). There are some transition segments between polyethylene and polypropylene segment in the block copolymer. It was found that the number and kind of transition segments decreased with elution temperature, the length and melting temperature of polyethylene segment in block copolymer fractions showed a similar tendency. © 1997 Elsevier Science Ltd.

(Keywords: ethylene–propylene block copolymer; fractionation; microstructure analysis)

INTRODUCTION

Because of its unique practical properties and theoretical value, ethylene–propylene block copolymer has been the subject of considerable research[1,2]. Ethylene–propylene block copolymer is one of the main components in impact-resistant polypropylene for its low brittle temperature and high tensile impact strength[3]. The possibility of synthesis ethylene–propylene block copolymer is related to the lifetime of active site in Ziegler–Natta catalysts[4,5].

Ethylene–propylene block copolymer is usually synthesized by sequential coordination copolymerization. Due to the complex character of coordination polymerization, the product obtained is inevitably a mixture of propylene homopolymer, ethylene–propylene block copolymer and ethylene–propylene random copolymer. The random copolymer can be readily removed by extraction with *n*-heptane[6]. However, block copolymer and propylene homopolymer cannot be separated with this method. In many previous studies, they were not separated, and so-called 'block' copolymer were still mixtures[7,8]. Additionally, the structural similarity between block copolymer and the PE/PP blend made it difficult to distinguish them and the judgement of block copolymer is still controversial[9–12]. The poor purity of block copolymer may also be partly responsible for this. Moreover, it is well known that there exist plural active sites in heterogeneous catalysts, which lead to the compositional heterogeneity of polymer[13,14]. The relative length of two segments in block copolymers may vary with different active sites. Therefore it is necessary to obtain relatively pure and compositionally homogeneous block copolymer for a better understanding of ethylene–propylene block copolymer. The fractionation of ethylene–propylene block copolymer was conducted by Besombers and Ito through extraction with different solvents or solvent/nonsolvent pair[3,15], but the emphasis was placed on the thermal behaviour of the fractions, and the block structure was not well characterized.

Recently, the temperature rising elution fractionation (t.r.e.f.) technique was developed for the fractionation of LLDPE and PP based on crystallinity[16,17]. However reports on the fractionation of blends with this method are rare. We believe that the crystallinity of various components in blends should be different and they could be separated with this technique. In the present work, a commercial impact-resistant PP alloy was fractionated and ethylene–propylene block copolymer was obtained. The block structure of the fractions was identified by combining fractionation results with ^{13}C n.m.r. and d.s.c.

EXPERIMENTAL

Materials

An impact-resistant polypropylene alloy sample was obtained commercially. It was produced according to the following steps: Propylene was firstly homopolymerized in bulk, then propylene was evaporated and polymer particles was transported into a gas tank. In this step, ethylene was introduced. To obtain products with good quality, propylene was not removed completely and a small amount of propylene was left in the second step. Some characteristics of this sample are listed in *Table 1*.

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