

THE EFFECT OF IN-PROCESS ETHYLENE INCORPORATION ON THE EVOLUTION OF PARTICLE MORPHOLOGY AND MOLECULAR CHARACTERISTICS OF COMMERCIAL HETEROPHASIC ETHYLENE PROPYLENE COPOLYMERS (HEPCS)

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ABSTRACT

The development of particle morphology, composition, crystallinity and microstructure were investigated for commercial heterophasic (high impact) propylene-ethylene copolymers (HEPCs).

Samples from gas phase polymerization processes were collected at set intervals after the introduction of ethylene into the second reactor. TREF profiles show pronounced differences between these samples where the amount of the lower crystallinity fractions (30 - 80°C), increased with increasing ethylene content, whereas the amount of the 90 - 120°C fractions decreased with increasing ethylene content. FE-SEM analysis of the external surfaces of all the samples as well as the internal structure of the particles (as revealed by microtomed slices through the particles) was used to gain a clear picture of the development of the rubber morphology as a function of reaction conversion. (Figure 1).

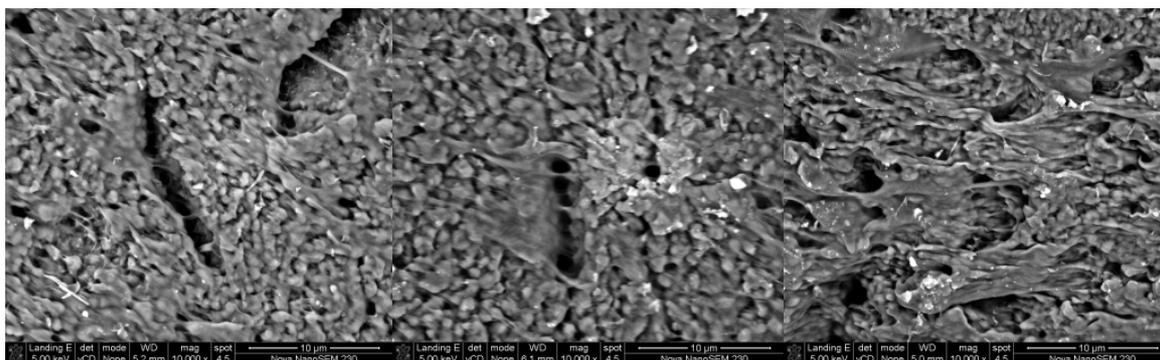


Figure 1. Internal morphology of samples with increasing ethylene content (1, 4 and 7% from left to right). FE-SEM images, scale bar is 10 µm

High temperature solution ¹³C NMR has shown a disproportionate microstructural development where continuous E sequences seem to increase at a higher rate than random-like mixed E & P sequences. The overall length of continuous E sequences is however limited by the total ethylene content. Solid state NMR has shown that the sample with the highest ethylene content has at least some ethylene monomers incorporated in rigid areas within the polymer. From GPC analysis, it was observed that molecular weight and – distribution increased with ethylene content. SEC-FTIR results for bulk samples have shown that both ethylene content and crystallinity is prevalent in the high molecular weight area of the curves, whereas the same properties for propylene were found in the lower molecular weight portion. Overall the development of particle morphology and the related chemical composition distribution could be directly related to the morphology and mechanical properties of the polymer after processing.