Title: A combined SSA – NMR - HT 2DLC approach to elucidate compositional differences in impact PP materials

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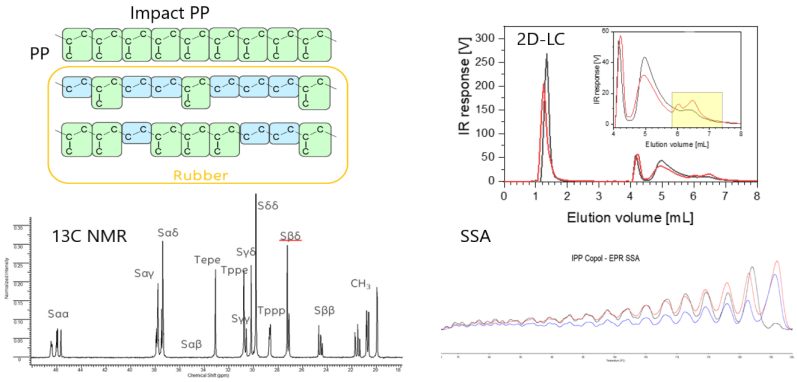
Images:



Abstract:

The identification of types, concentrations and distributions of regular / crystallizable sequences in polyolefins is an important structural information. This information correlates with the physical properties of these materials, but also enables researchers to extract information about the nature of the catalyst sites and the mechanisms of the polymerization processes. This aspect is particularly true (and challenging) for more complex materials such as high-impact polypropylene, usually produced in two stages: 1) PP homo-polymer synthesis and 2) copolymerization of ethylene-propylene to produce the rubber phase. [1]

Common characterizations used for fast material evaluation include melt flow index (MFI), rubber content and the amount of ethylene in the rubber. However, due to the inherent complexity of the catalytic systems used for their production (mostly Ziegler-Natta, based on Ti active species) it is possible to obtain materials that perform quite differently from each other despite very similar values of MFI and ethylene content. Some of these differences can be ascribed to the different ways in which the ethylene/propylene co-monomers are distributed in the ethylene-propylene rubber (EPR) phase. Meaning, similar E/P ratios can lead to varying copolymer architectures, depending on the tendency of the catalyst to build the two co-monomers across truly random, ethylene-rich or propylene-rich components. [2]

In these cases, an in-depth characterization of microstructural features is paramount to understand the effects of changing the catalytic conditions (type of catalyst, amount of monomers, process etc.) towards polymer microstructure and consequently on the morphology developed during processing and resulting properties.

In order to complete such a characterization process, complex and time-consuming material fractionation may be required to correctly identify the range of compositions in the bulk material.[3] In the present work, we have characterized several impact PP materials produced by different catalytic systems. The sample set includes materials with similar MFI, rubber content and ethylene content in the rubber phase that show different mechanical performances. The same set of characterization-tools, namely Successive Self-Nucleating and Annealing (SSA)[4], carbon-thirteen nuclear magnetic resonance spectroscopy (^{13}C NMR)[5], High-temperature size exclusion chromatography coupled with differential viscometer and infrared detectors (HT-SEC-DV-IR) and High-temperature two- dimensional liquid chromatography (HT-2D-LC)[6], has been applied to both fractionated (pMMF)[7] and bulk material, and the results are compared. This combined use of analytical tools enables a thorough understanding of material microstructure and the relationship with material properties and catalyst behavior.