

MULTIDIMENSIONAL ANALYTICAL TECHNIQUES FOR THE CHARACTERIZATION OF PHTHALIC ANHYDRIDE BASED POLYESTERS

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ABSTRACT

The basis for the extensive industrial application of aliphatic polyester resins are the different physical and mechanical properties of the final products brought about by the implementation of various combinations of different acids and glycols and their molar ratios that provide products with a large variety of final applications. Commercial resins typically consist of prepolymers obtained by polycondensation of unsaturated and saturated diacids or anhydrides with diols. As the saturated constituent, phthalic anhydride plays an important role in the production of aliphatic polyester resins implemented in the coatings industry. Essentially, these resins are varied mixtures of molecules differing in molecular weight, chemical composition and degree of branching. While previous studies have investigated this particular diacid system in combination with other diacids and diols, detailed information regarding a model phthalic anhydride-1,2-propylene glycol system was lacking. The study focussed on kinetic samples ranging between 10^2 and 10^4 Da in molecular weight, which were removed at varying stages of the polyesterification reaction. The course of the polycondensation reaction and the evolution of the different distributions with regard molecular weight, chemical composition and functionality type were investigated by various characterization techniques. Different liquid chromatography techniques had to be developed. SEC and gradient HPLC conditions first had to be optimized in order to achieve successful separations, before their subsequent on-line coupling could determine the link between the dual functionality type-molecular weight distributions. Secondly, soft-ionization mass spectrometry techniques were employed to serve as core methods to determine the extent of chemical composition, identify the functionality-type distributions as well as assist in assigning structural conformations. Suitable sample preparation and analysis conditions had to be established in MALDI-TOF MS and ESI-QTOF MS for the bulk sample investigation. In serving as selective detectors, their off-line coupling with the aforementioned LC techniques had to be conducted effectively, to facilitate the identification of the narrowly dispersed fractions obtained from the chromatographic separations. Only through the hyphenation of these sophisticated polymer characterization techniques, information on the molecular heterogeneity of the model polyester prepolymer, showing a complex variety of possible distributions, could be obtained.

References:

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