THE COMBINATON OF LIQUID CHROMATOGRAPHY AND MASS SPECTROMETRY TECHNIQUES FOR THE CHARACTERIZATION OF ALIPHATIC POLYESTERS

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ABSTRACT

Aliphatic polyesters are industrially implemented in high performance coatings, paints and varnishes, however it is difficult to correlate the resulting properties with the synthesis parameters as these polymers vary in reactivity and application properties. Due to the stepwise polymerization mechanism, it is often assumed that copolyester synthesis by direct polyesterification will lead to randomized products. The formation of cyclic products by intramolecular reactions of hydroxylic (OH) and carboxylic (COOH, acid) functional groups, including side-reactions such as transesterification, alcoholysis, ester-ester interchange allow even further randomization, enabling a highly complex system. Polyesters exhibit functionality type and branching distributions in addition to the molar mass distribution. The functionality type distribution influences the reactivity of the system while branching affects the flow properties. The properties are thus determined by not only one but by the correlation of two/more distributions. The different methods of polymer chromatography in combination with sophisticated spectroscopic techniques are useful tools for enabling the full description of the molecular heterogeneity of these complex polyesters [2].

The present study entails method development of interaction chromatography and mass spectrometry along with their combination, to facilitate the analysis of the various distributions of two simple aliphatic polyesters, phthalic and maleic anhydride respectively in combination with propylene glycol. Gradient-HPLC analysis enabled an oligomeric separation of the respective anhydride/propylene glycol samples and its off-line coupling to MALDI-TOF MS results revealed the presence of several distributions of varying functionality type and molar mass at different intervals throughout the polymerization. In addition, online gradient-HPLC-size-exclusion chromatography (SEC (2D-LC)) was conducted to obtain the dual functionality type-molar mass (FTD-MMD) distribution. The combination of the different coupling techniques provided the opportunity to a more in-depth analysis of the structure-property relationships.

References:

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