CORRELATING CHEMICAL STRUCTURE AND POLYMER WEIGHT VIA SEC-IR DETECTION WITH A NEW QCL LASER SPECTROMETER

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ABSTRACT

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Polymers have three important molecular characteristics: the molecular weight distribution (MWD), the chemical composition and the topology. The MWD is usually determined using size exclusion chromatography (SEC). SEC detectors commonly in use, such as refractive index detectors, light scattering or viscometers, do not provide information about the chemistry or topology. This information is normally gained separately using spectroscopic methods. Especially when analyzing complex materials like copolymers, blends or unknown samples, the correlated measurement of size and chemical properties is of special interest, not only to better understand the influence of molecular properties on material characteristics, but also for industrial quality control, material design and product development.

Coupling IR spectroscopy with SEC is a promising approach to gain this correlated information. [1] An inherent problem to this pairing is the normally very high solvent signals, which arise from the low sample concentration necessary for SEC separation. For our online SEC-FTIR coupling we optimized a standard research FTIR spectrometer to maximize its sensitivity and constructed specialized flow cells for the SEC coupling. [2] A mathematical solvent suppression routine is used to subtract the solvent signals. For an even higher sensitivity, different infrared light sources are needed. We also present results from a SEC coupled with an IR spectrometer using a tunable Quantum Cascade Laser (QCL) light source, which has a higher light intensity, but a limited bandwidth. [3] In this application, the SEC-QCL-IR has the best sensitivity when operated in single wavelength mode. This makes SEC-QCL-IR ideal for investigating specific features of interest, such as an end-groups, functional groups or branching points. Therefore, SEC-QCL-IR measurements complement, but do not replace SEC-FTIR results. The method development for both SEC-FTIR and SEC-QCL-IR including the general setup, the flow cells, the mathematical solvent suppression and the influence of column dimensions will be presented. [2,3] As part of the method development, SEC separation columns with novel geometries were specially constructed and results on their impact on the spreading of the eluent peak will be discussed.

References:

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