

Characterization on Molecular Weight of Polyamic Acids Using GPC Coupled with Multiple Detectors

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Abstract : Polyamic acid (PAA) is the precursor of polyimide (PI) prepared by a two-step method, its molecular weight and molecular weight distribution not only play an important role during the preparation and processing, but also influence the final performance of PI. However, precise characterization on molecular weight of PAA is still a challenge because of the existence of very complicated interactions in the solution system, including the electrostatic interaction, hydrogen bond interaction, dipole-dipole interaction, etc. Thus, it is necessary to establish a suitable strategy which can completely suppress these complex effects and get reasonable data on molecular weight. Herein, the gel permeation chromatography (GPC) coupled with differential refractive index (RI) and multi-angle laser light scattering (MALLS) detectors were applied to measure the molecular weight of (6FDA-DMB) PAA using different mobile phases, LiBr/DMF, LiBr/H₃PO₄/THF/DMF, LiBr/HAc/THF/DMF, and LiBr/HAc/DMF, respectively. It was found that combination of LiBr with HAc can shield the above-mentioned complex interactions and is more conducive to the separation of PAA than only addition of LiBr in DMF. LiBr/HAc/DMF was employed for the first time as a mild mobile phase to effectively separate PAA and determine its molecular weight. After a series of conditional experiments, 0.02M LiBr/0.2M HAc/DMF was fixed as an optimized mobile phase to measure the relative and absolute molecular weights of (6FDA-DMB) PAA prepared, and the obtained Mw from GPC-MALLS and GPC-RI were 35,300 g/mol and 125,000 g/mol, respectively. Particularly, such a mobile phase is also applicable to other PAA samples with different structures, and the final results on molecular weight are also reproducible.

Keywords : Polyamic acids, Polyelectrolyte effects, Gel permeation chromatography, Mobile phase, Molecular weight

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