[V-37]

Structural Characterization of Branched Polyesters Using TOF-SIMS Combined with Transesterification

Yeonhee Lee[1], Seunghee Han[1], Jung-Hyeon Yoon[1], Hyuneui Lim[2], Moojin Suh[1]
[1]Advanced Analysis Center, Korea Institute of Science & Technology
[2]Department of Chemistry, Korea University

Mass spectrometry technique provides the molecular weight distribution, data on the sequence of repeat units, polymer additives, and impurities, and structural information. Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) has been used for structural characterization of various polymers[1,2] The masses of repeat units and terminal groups and molecular weight distributions of polymers have been determined from their TOF-SIMS spectra. TOF-SIMS provides good sensitivity and structural specificity for high mass ions so that intact oligomers and large polymer fragments are observed.[3]

In this study, we investigated the detailed structural information on the oligomers and fragment ions of branched poly(1,3-butylene adipate) and branched poly[di(ethylene glycol) adipate] and the transesterification products of branched polyesters with trifluoroacetic acid or chloro difluoroacetic acid. Branched polyesters were chosen because they are important polymers but difficult to characterize; thus branched polyesters provide challanging test for TOF-SIMS. TOF-SIMS spectra of polyesters are obtained from thin polymer films cast from solution on a silver substrate. A good solvent for a polymer solution disrupts intermolecular forces between polymer chains but leaves the polymer intact. Transesterification reactions are potentially useful for characterization of high molecular weight and intractable polyesters. Transesterification products of polyesters and trifluoroacetic acid or chlorodifluoroacetic acid were identified from secondary ion mass spectra to be diesters consisting of an integral number of polyester repeat units and an additional diol. The progress of such reactions was monitored using peak intensities of reactants and products in TOF-SIMS spectra. The increasing abundance of tagged ions indicates that the reaction has progressed with time.

References

[1] Bletsos, I.V.; Hercules, D.M.; Greifendorf, D.; Benninghoven, A. Anal. Chem. 1985, 57, 2384
[2] Bletsos,I.V.; Hercules, D.M.; vanLeyen, D.; Benninghoven, A.; Karakatsanis, A.G.; Rieck, J.N. Macromolecules 1990, 23, 4157
[3] E. Niehuis, In Secondary Ion Mass Spectrometry-SIMS VIII; A. Benninghoven, K.T.F. Janssen, J. Tumpner, H.W. Werner, Eds.; John Wiley&Sons: New York, 1992