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Application News

MALDI-TOF Mass Spectrometry No.B42

Analysis of Recycled Polyesters by MALDI-TOFMS (1)

The recycling of plastic products has increased in recent years due to the enactment of recycling-related laws aimed at protecting the global environment. Along with this, there is a growing need for faster and more detailed analysis of recycled products. For example, instead of analyzing an entire polymer, a less-challenging analysis of only the related oligomers could provide sufficient information. Traditionally, oligomer analysis has been conducted using various techniques, including rough separation using the precipitation method, combined with a variety of chromatographic and spectrometric techniques.
More recently, MALDI-TOFMS has become widely used for oligomer analysis. Application of MALDI-TOFMS allows terminal group and monomer unit information to be obtained very quickly.
Here, we introduce an example of recycled polymer analysis using a combination of oligomer rough separation by the precipitation method with MALDI-TOFMS.

Fig. 1 shows an outline of the precipitation method. After dissolving the polymer in a good solvent, an aliquot is transferred to a poor solvent, and after filtering out the precipitated and suspended polymer substances, the supernatant is analyzed. First we investigated how this pretreatment procedure would affect the mass spectrum. Commercially-available PBT (polybutylene terephthalate) was used as a test sample.
Fig. 2 shows a comparison of the results obtained when PBT is simply dissolved in a good solvent and when it is processed using the precipitation method. In both cases, the 220 mass difference corresponding to the cyclic oligomer and indicative of PBT was detected.
However, only low molecular weight oligomers were detected in the unprocessed sample, compared to that using the precipitation method. This is thought to be due to oligomer ionization interferences due to co-existing polymer substances. Further, the PBT that was dissolved only in the good solvent was instantly precipitated when mixed with the matrix solution, and peeled away and fell off the MALDI plate, having a serious adverse effect on data repeatability. Therefore, not only was rough separation better accomplished using the precipitation method, it also contributed to improved repeatability.

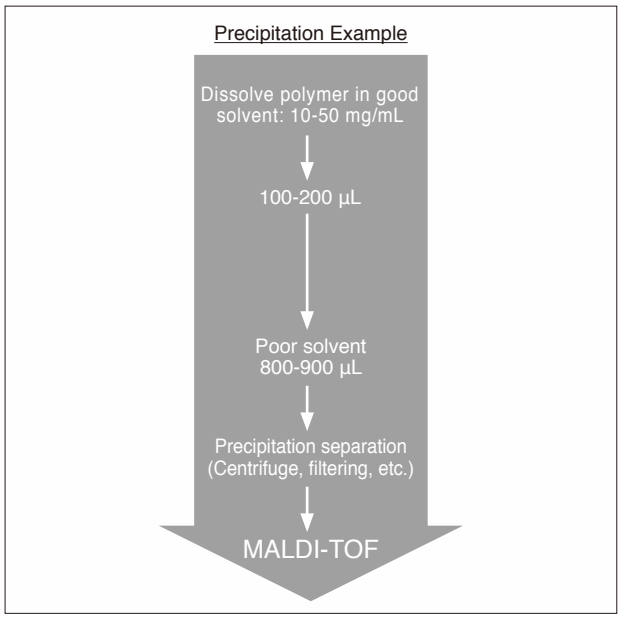

Fig. 1 Brief Description of Precipitation Method

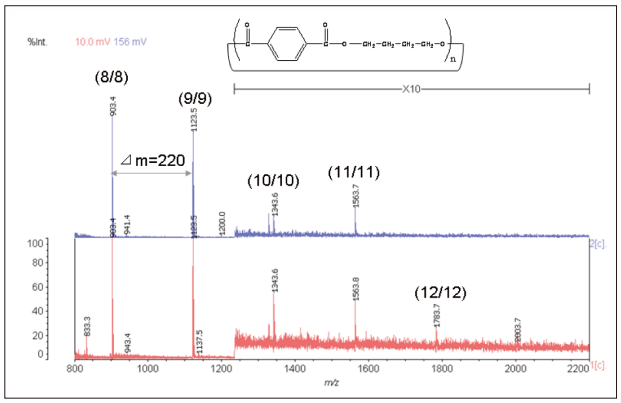

Fig. 2 MALDI-TOFMS Analysis of PBT
Upper; Dissolved in Good Solvent only, Lower; Additional Processing with Precipitation Method

Legend
-(m/n)-: Linear oligomer
(m/n): Cyclic oligomer
m: Number of terephthalate units
n: Number of butylene glycol units

Next, oligomers that were extracted from various products (stationery implements, kitchen utensils, commercially-available PET chips) containing PBT as a raw material were compared (Fig. 3). It was clear from the results that the recycled PBT used as a raw material in the stationery implement contained more oligomers than the other two. In addition, some of the oligomers detected suggested the possibility that they did not originate from PBT, and because their mass unit interval was 192, it was believed that they might be PET oligomers. Thus, it was demonstrated that the use of MALDI-TOFMS together with a simple preparation procedure permits the comparative assessment of recycled polyesters.

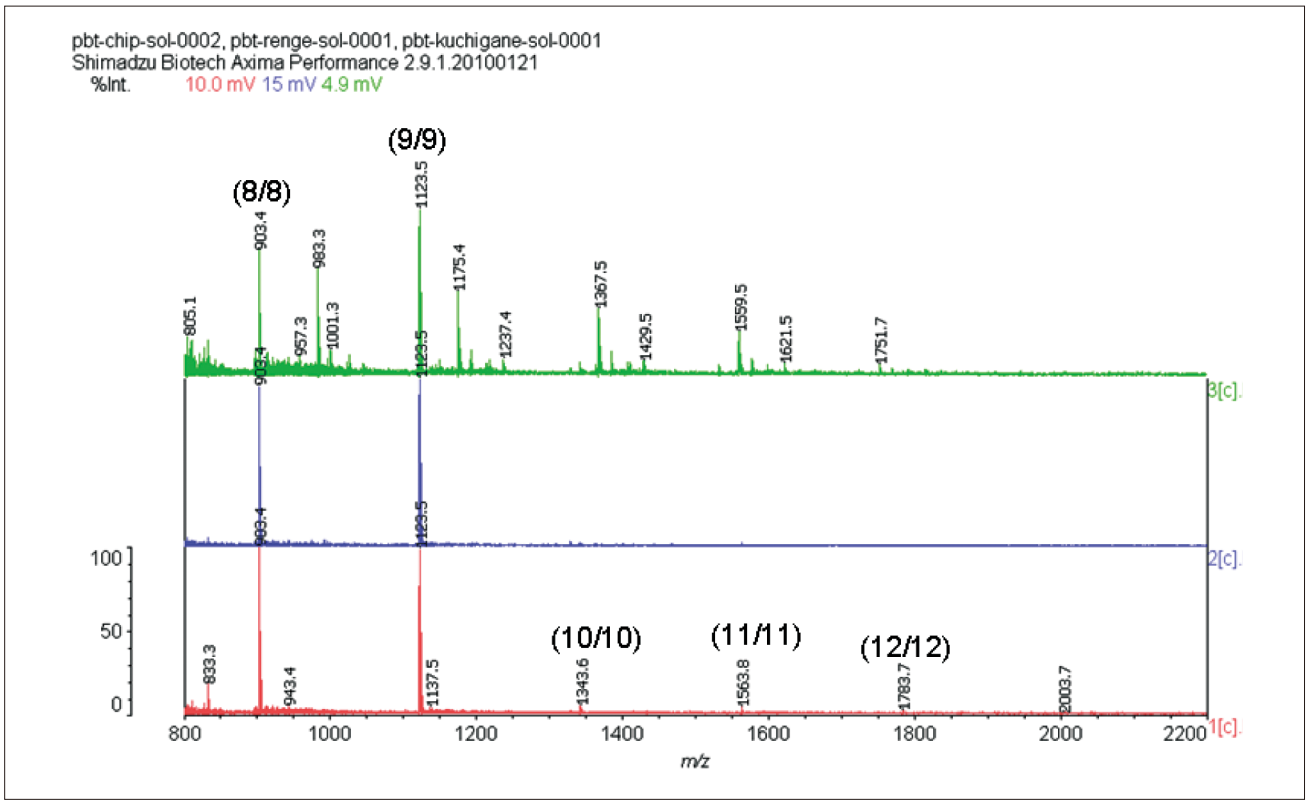

Fig. 3 Comparison of Various PBT Oligomers - Upper; Stationery Implement (recycled), Middle; Kitchen Utensil, Lower; Commercially-Available PET Chips

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