In recent years, recycling related laws have been enforced for the purpose of global environmental preservation, which in turn has increased the amount of recycled plastic products around us. This trend entails the needs of rapid and detailed analyses of recycled products. In such cases, sufficient information may be obtained by analyzing not the entire polymers but oligomers. Conventionally, oligomers are analyzed by combining rough separation using the dissolution/reprecipitation method, etc., and various chromatographic or spectroscopic techniques. On the other hand, recently MALDI-TOF mass spectrometers are extensively used for oligomer analysis. By using such instruments, the information of terminal groups and monomer units can be obtained rapidly. This article introduces an example analysis of polycarbonate, which was performed by combining rough separation of oligomers by the dissolution/reprecipitation method and measurement and analysis using the benchtop MALDI-TOF mass spectrometer “MALDI-8020”.

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**Benchtop MALDI-TOF MS: MALDI-8020**

The MALDI-8020 (Fig. 1) is a compact-design, minimal-space linear mode-only MALDI-TOF mass spectrometer. Its performance in linear mode (positive ion) is comparable to the same mode of a conventional floor-standing MALDI-TOF mass spectrometer. Equipped with a 200 Hz solid-state laser and a load lock chamber mechanism that enables a target plate change while maintaining the degree of vacuum at the measuring position, the instrument ensures rapid measurements.

**Molecular Weight Distribution Measurement of Polymers**

As polycarbonate (PC) samples, a PC standard sample and a PC oligomer sample extracted from a compact disc were used for analysis (Fig. 2). In the results of both samples, we can see that monoisotopic peaks are detected and that PC monomer units spaced by 254 u are repeated (insets in Fig. 2).
Analysis Example of Terminal Groups

The mass spectra in Fig. 3 show that three repetitions of the 254 u monomer unit of PC. Since PC uses an end capping reagent, we assumed and analyzed the following three chemical structures using Polymerix software (Sierra Analytics). Three oligomer components are assigned to both-end capped type, single-end capped type and cyclic type, respectively (Fig. 3).

We found that the oligomers extracted from a compact disc are mainly the both-end capped type. It is known that the polymers used for optical discs require high quality so low-molecule oligomers are intentionally removed. This MALDI-TOF mass spectrometry result reflects this manufacture processing. On the contrary, in the case of oligomers extracted from a stationery item made from recycled PC, cyclic oligomers are relatively high and those with single terminal OH are also high. In the case of commercially available chips, those with single terminal OH are relatively high and cyclic oligomers are low. This trend is reported from similar analyses using MALDI-TOF mass spectrometry.1 As indicated above, we found that with the use of MALDI-TOF mass spectrometry and a simple preparation method, comparative evaluation of recycled polymers can be performed.

Conclusion

These examples demonstrate that the benchtop MALDI-TOF mass spectrometer “MALDI-8020” can be used for molecular weight measurement and terminal group analysis of polymers at the same level as larger-sized MALDI-TOF mass spectrometers.

In recent years, MALDI-TOF mass spectrometers have evolved into high specification instruments and have increased in size and complexity. As a result, the instruments are typically over-specified for most routine applications requiring linear mode analysis. Size and initial/running costs can pose a barrier for introducing the instrument into some laboratories.

The MALDI-8020, the world’s smallest class of commercially available MALDI-TOF mass spectrometer, satisfies the needs of polymer analysis in linear mode, and its future dissemination and wider use are expected.

Reference

1 Shimadzu Application News No. B43, Analysis of Recycled Polyesters by MALDI-TOF MS (2)

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