Analysis of thermoplastic elastomers

EC-064

Purpose

A time-of-flight mass spectrometer equipped with a ChemZo ion source and an elevated temperature heating system was used to investigate the changes of a thermoplastic elastomer (O-ring) with time before and after use.

Experimental method

Ion source

- ChemZo BioChromato (With temperature rise heating device)

Mass spectrometer

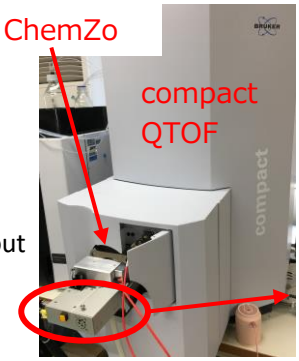
- compact QTOF Bruker

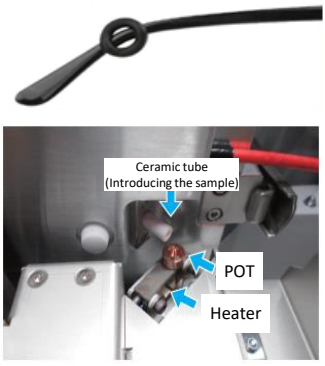
Measurement method

- The O-rings for glasses anti-slip before use and after use for about 4 months (0.8 mg each) were placed on a sample table (POT) and the thermal desorption components were measured while the temperature was raised from room temperature to 400°C.

Analytical method

- The TIC and mass spectra were analyzed using Spectra Scope and Data Analysis software.





Temperature gradient device

Fig.1 shows the TIC of the thermoplastic elastomer before and after use. In the sample after use, more numbers of thermal desorption components were detected at lower temperatures than the sample before use. Fig. 2 shows the mass spectra of the thermoplastic elastomers at 200-300°C. In the sample before use, continuous series of ions of the thermoplastic elastomer, which may have been derived from the polymer, were detected, but were hardly detected in the sample after use. Fig. 3 shows a radar chart of the differential analysis (IDS) results before and after use. In the thermoplastic elastomer after use, a large number of thermally desorbed components in the low molecular weight range were detected, probably derived by the degradation of the resin in the hard portion of the elastomer after long-term use. Also, for the IDS results, a database search was conducted for additives, and it was estimated that the $C_{18}H_{35}NO$ was included as a lubricant.

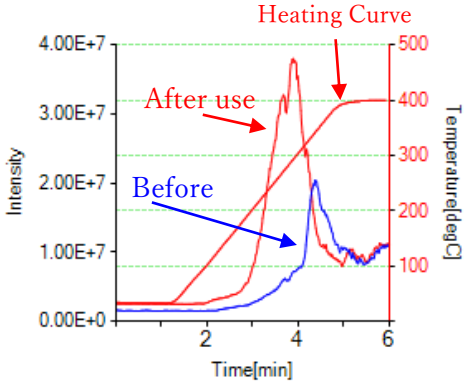


Fig.1 TIC of new and used elastomer

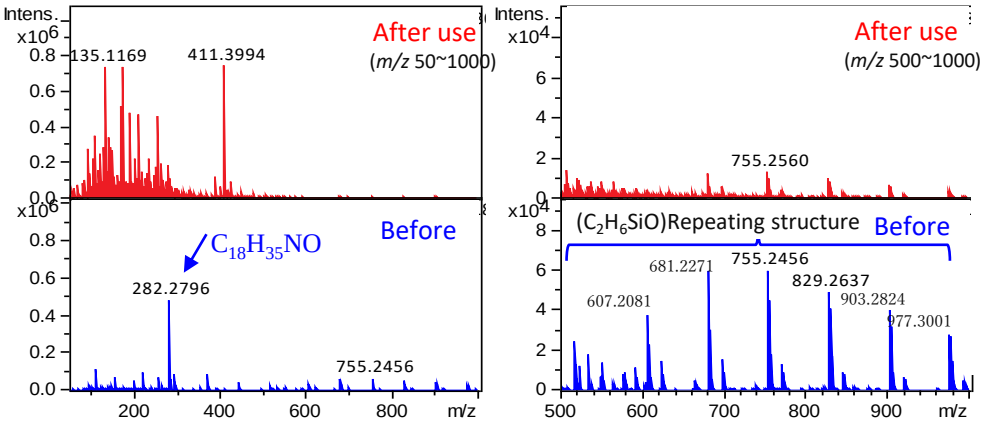


Fig.2 Mass spectra of new and used elastomer at 200～300℃

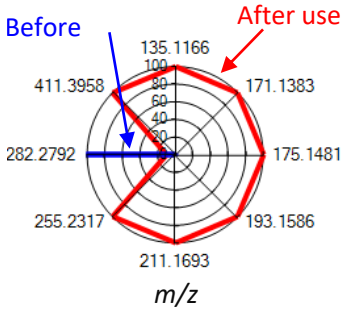
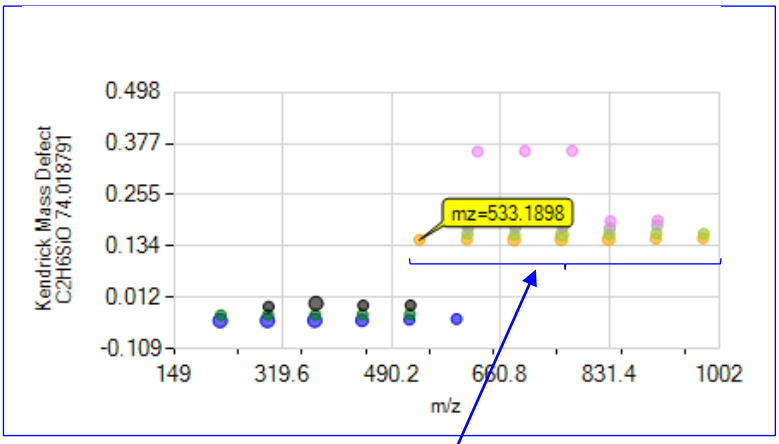


Fig.3 Radar chart of IDS results of new and used elastomer at 200～300℃

Analysis of thermoplastic elastomers



AutoKMD Result Table

No	Base Unit	MW	Detected	Divisor
1	C2H6SiO	74.018791	8	MW-1

Annotation	Color	Enable	Base m/z	Intensity	KMD	Repeat	Compound Result	IDS	IDS points
1		☑	221.0848	92726	-0.042	6	1	5	1, 2, 3, 4, 5,
2		☑	222.0857	20064	-0.029	5	0	4	1, 2, 3, 4,
3		☑	297.0982	16522	-0.009	4	0	0	
4		☑	533.1898	18890	0.149	7	0	4	1, 2, 6, 7,
5		☑	608.2086	22760	0.163	6	0	3	1, 5, 6,

Four of the seven ions (first, second, sixth, and seventh) corresponding to the repeating structures of 74.0188 each, starting at *m/z* 533.1888, are shown to be present in IDS.

In order to compare the measurement results of the resins before and after use, analysis using Polymer Engine was conducted in the 200-300°C region of TIC. Fig. 4 shows the bubble plots and the list of those analyzed by Polymer Engine.

Polymer Engine is a software program that extracts the repeating structure of polymers from their mass spectra, and the repeating ion of C_2H_6SiO (74.0188), which was thermally desorbed, is displayed here. The X-axis of the graph is the m/z value and the Y-axis is the Kendrick Mass Defect. For example, the line with m/z =533.1898 in Fig. 4 indicates that there are seven consecutive repeating structures from m/z 533.1898, and the IDS and IDS points in the list indicate the number of hits and the order number of ions if the IDS result contains the same m/z value as the one extracted by Polymer Engine.

Fig.4 Bubble plots and the list of new and used elastomer at 200～300℃

Also, by adding the m/z values up and creating an EIC (Fig. 5), molecular species with the same repeat structure in different resins can be compared, and the degree of degradation can be easily compared.

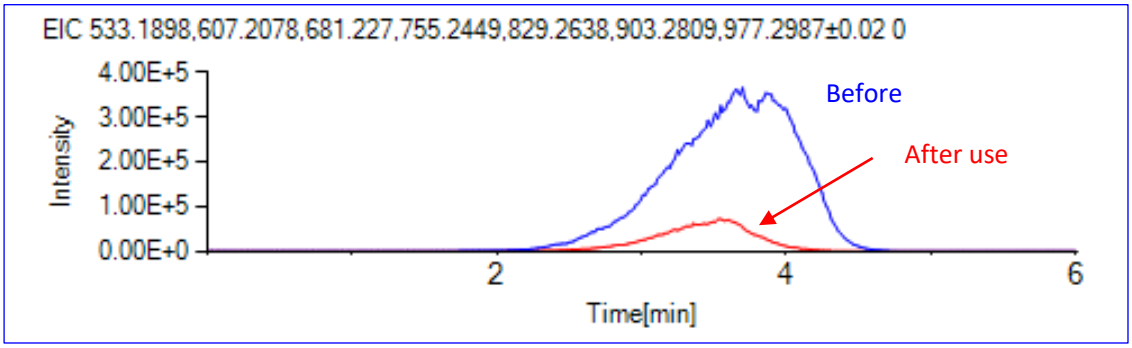


Fig.5 Summed EICs from *m/z* 533.1898 to 977.2987