

Compositional Analysis of NBR by pyrolysis GC/MS

Part 1: Fundamental investigation by Evolved Gas Analysis

[Background] Acrylonitrile-butadiene rubber (NBR) is a synthetic rubber which has excellent properties such as high oil resistance and wear resistance. It contains various additives to improve weather resistance, mechanical properties, etc. Therefore, not only main components of the NBR material but also additives need to be analyzed to assess the polymer properties. In this note, Evolved Gas Analysis (EGA)-MS is conducted as the fundamental investigation for the compositional analysis of NBR by pyrolysis GC/MS to determine the pyrolysis temperature. In addition, chemical species were surveyed from the averaged mass spectrum of each temperature zone of EGA curves.

[Experimental] A GC/MS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC injector was used for EGA-MS measurements. A deactivated metal tube (UADTM-2.5N) and a Vent-free GC/MS adapter were installed between the GC injector and the mass detector. Two types of NBRs with varied compounding ratios were used as samples. The NBR samples were scraped off with a cutting knife, and then the scraped pieces were put into an Eco-Cup, precisely weighed (ca. 0.1 mg each), and introduced into the pyrolyzer furnace for EGA-MS measurements.

[Results] EGA curves and averaged mass spectra of the NBR samples are shown in Fig. 1. Additives should mainly appear in the lower temperature zone, i.e. Zones 1-A and 2-A. Peaks detected in Zones 1-B and 2-B are derived from the pyrolysis of NBR. It is confirmed that the polymer type is both NBR based on the library search of the averaged mass spectra using the F-Search. For both NBR samples, evolution of gases from the polymer component was completed by 600 °C. Thus, pyrolysis temperature in Py-GC/MS was determined to be 600 °C. In the next note (PYA1-173E), the qualitative analysis of polymer and additives in NBR samples was conducted by Py-GC/MS. Further, the quantitative analysis of monomer composition (compositional ratio of acrylonitrile to butadiene) was carried out.

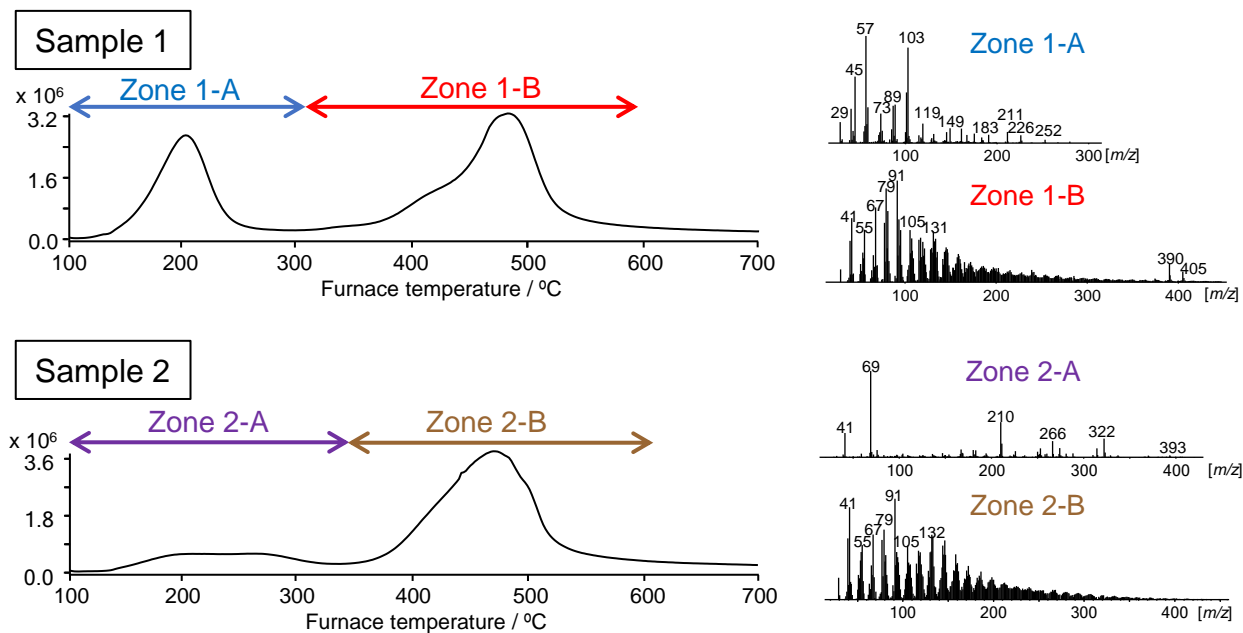


Fig. 1 EGA curves of NBRs and averaged mass spectrum of each temperature zone.

Furnace temp.: 100 – 700 °C (20 °C/min), EGA tube: UADTM-2.5N (L=2.5 m, i.d.=0.15 mm), Column flow rate: 1 mL/min (He), Split ratio: 1/20, GC oven temp.: 300 °C, MS scan range: m/z 29 - 600, MS scan rate: approx. 0.2 scan/s, Sample amount: ca. 0.1 mg.

Keywords : NBR, Compounded rubber, Compositional analysis, EGA-MS, Evolved gas analysis

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UADTM-2.5N, Eco-Cup LF, Quartz wool, F-Search, Vent-free GC/MS adapter

Applications : General polymer analysis, Additives analysis, Quality control, Material analysis, Defect analysis

Related technical notes : [PYA1-173E \(Part2\)](#), [PYA1-047E](#)

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