

Analysis of Compounded Rubber by Double-Shot Technique

Fig. 1 shows an evolved gas curve of a compounded rubber. Weak peaks are observed in 100~300°C region due to the thermal desorption of various additives. In 300~500°C region, a broad peak due to thermal decomposition of the rubber is observed. From this result on hand, thermal desorption, the first stage of double-shot analysis, was performed from 100 to 300°C (20°C/min), and flash pyrolysis, the second stage, was done at 550°C. Fig. 2 shows results of analysis. In the chromatogram of thermal desorption shown in Fig. 2a, cyclic siloxanes (D3~D6) originated from silicon coupling agent, benzothiazole (vulcanization accelerator), higher aliphatic acid (vulcanizing agent), and waxes (antioxidants, marked with *) were observed. Because isoprene and limonene were mainly observed in the pyrogram shown in Fig. 2b, the major component of this sample is natural rubber; further, it contains butadiene rubber since a small amount of butadiene was also observed.

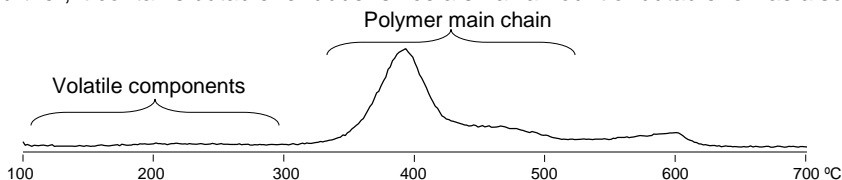


Fig. 1 Evolved Gas Curve of a Compounded Rubber

Pyrolysis temp.: 100~700°C (20°C/min), Carrier gas : He 50kPa, Split ratio : ca. 1/20
EGA capillary tube : 0.15mm id, 2.5m (UADTM-2.5N), GC oven temp.: 300°C
Injection temp.: 320°C, Sample : ca. 5µg, Detector : MS (m/z=29-400)

Fig.2a. Thermal Desorption Chromatogram (100~300°C (20°C/min))

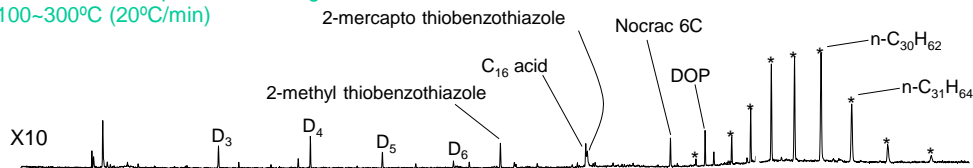


Fig.2b. Pyrogram (550°C)

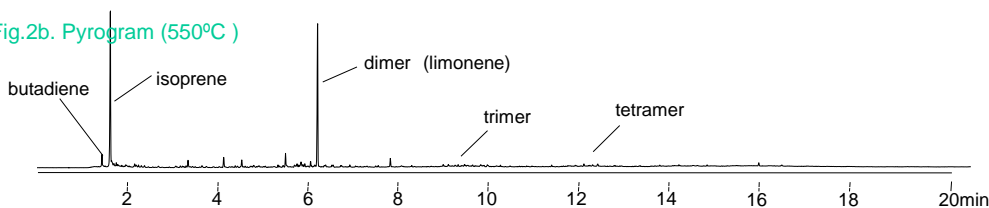


Fig. 2 GC/MS Analysis of Compounded Rubber by Double-Shot Technique

Column flow rate : 1ml/min (fixed flow rate), Split ratio : 1/20
Separation column : Ultra ALLOY*-5 (5% diphenyl polysiloxane), 30m, 0.25mm id, Film thickness : 0.25µm
GC oven temp.: 40~300°C (20°C/min), Sample : 5µg, Detector : MS (m/z=29-400, 2scans/sec)

Keyword : Double-Shot Technique, Rubber, Coupling Agent, Vulcanizing Agent, Antioxidant

Applications : General Polymer Analysis

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R&D and manufactured by:

Frontier Laboratories Ltd.

1-8-14, Saikon, Koriyama,
Fukushima, 963-8862 Japan

Phone: 81-24-935-5100 Fax: 81-24-935-5102

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