

Compositional Analysis of Isoprene-butadiene-styrene Blend Rubber by Py-GC

[Background] The compositional analysis of multi-component blend rubber is very important in quality control. The Py-GC technique is a useful tool to provide rapid compositional analysis of blend polymers. Here, a compositional analysis of a blend rubber consisting of polybutadiene (PB)-polyisoprene (PI)-polystyrene (PS), as a typical example, is described.

[Experimental] A Py-GC system used in this study was a Double-Shot Pyrolyzer® which was directly coupled with the split/splitless injection port of a GC and was connected to a FID via a metal capillary column. A calibration curve was created using standard samples (A~C) shown in Table 1 and was used to obtain the composition ratios of unknown sample D. It was later compared with the composition ratio of the starting materials in Table 2.

[Results] Figure 1 shows a pyrogram of PB-PI-PS 3-components blend rubber obtained at 550°C. Major pyrolyzates such as butadiene (BD), isoprene (IP), and styrene (ST) were observed as main constituent monomers. A good linear calibration curve was obtained from the relative peak areas of monomers and the composition ratios of starting standard material. An example shown in Fig. 2 is a calibration curve for PB component. Calibration curves for PI and PS components were similarly obtained. As an example shown in Table 2 in which the composition ratio of unknown D obtained from the calibration curve and that of the starting material are compared, this technique demonstrates that blend compositions can be determined with a high precision.

Table 1.
Composition ratio of standard and peak ratio of monomers

Sample	Composition ratio of standard (wt.%)			Relative peak area (%)			Total
	PB	PI	PS	BD	IP	ST	
A	9.9	80.0	10.1	4.5	63.0	32.5	100
B	29.8	40.0	30.2	9.5	28.1	62.4	100
C	44.6	10.0	45.4	13.2	7.8	79.1	100

Table 2.
Compositional ratios of unknown D

	PB	PI	PS
Composition ratio determined(wt. %)	37.1	24.9	38.0
Composition ratio of starting material (wt. %)	37.2	25.0	37.8

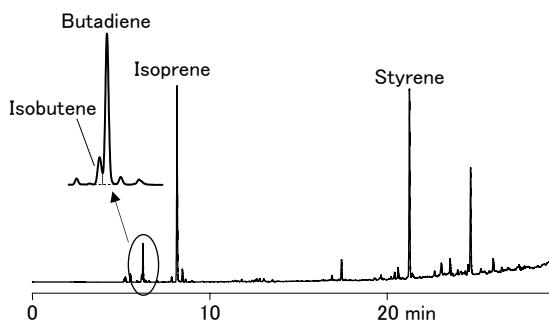


Fig. 1 A pyrogram of standard sample

Pyrolysis temp.: 550°C, detector: FID, sample: standard sample A
 Separation column: Ultra ALLOY+5 (5% diphenyl 95% dimethylpolysiloxane)
 Length: 60m, id: 0.25mm, film thickness: 1.0µm
 GC oven temp.: 50°C (7min hold) – 280°C (10°C/min), carrier gas: He
 Injection port pressure: 175kPa, split ratio: 1/60, sample size: ca.200µg

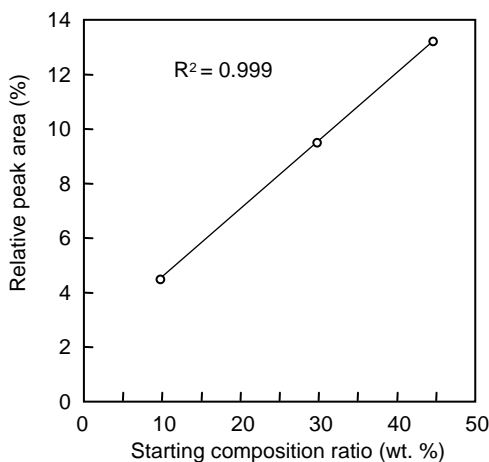


Fig. 2 Calibration curve for PB composition

Keyword : Blend rubber, Composition analysis, Butadiene, Isoprene, Styrene

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