

Determination of monomer compositions of acrylic pressure-sensitive adhesives by pyrolysis GC/MS

[Background] The monomer composition of acrylic copolymers for pressure-sensitive adhesives (PSAs) has a significant impact on physical properties such as adhesion and peelability. In this note, three-component acrylic copolymers for PSAs having different monomer compositions (three monomer types of butyl acrylate BA, 2-ethylhexylacrylate 2EHA, and 2-hydroxyethylacrylate 2HEA) were analyzed by pyrolysis (Py-) GC/MS to investigate whether the monomer compositions of acrylic PSAs can be determined from the relative peak intensity of acrylic monomers to an internal standard (IS).

[Experimental] Five acrylic copolymers with known monomer compositions were synthesized by solution polymerization in ethyl acetate and used as standard samples for creating calibration curves. The molecular weight ranged from 300,000 to 500,000. Solutions of acrylic copolymers were prepared by dissolving 20 mg of copolymers in 1 mL of dichloromethane, and an IS solution, prepared by dissolving 1 mg of eicosan in 10 mL of hexane. 5 μ L each of copolymer and IS solutions were put together in a sample cup. After evaporation of the solvent, a thin film containing 100 μ g of acrylic copolymer and 0.5 μ g of eicosan was formed on a sample cup wall. The sample cup was then introduced into the pyrolyzer furnace and subjected to Py-GC/MS measurements. Calibration curves were constructed by plotting the peak area ratio of monomer to IS against the monomer mass. A commercial acrylic copolymer containing BA, 2-EHA, and HEA as monomers was used to demonstrate the usefulness of the calibration curves.

[Results] Calibration curves with good linearities ($n=5$) for BA, 2EHA, and 2HEA monomers were obtained using the copolymers having five different monomer compositions (Fig. 1). Using these calibration curves, the monomer compositions of a commercial PSA sample (an acrylic copolymer) were determined from its pyrogram (Fig. 2), and the results are well agreed with the originally formulated compositions provided by the manufacturer as summarized in Table 1. In conclusion, it was found that the monomer compositions of acrylic PSAs could be determined based on the monomer peak intensities on the PSA pyrograms.

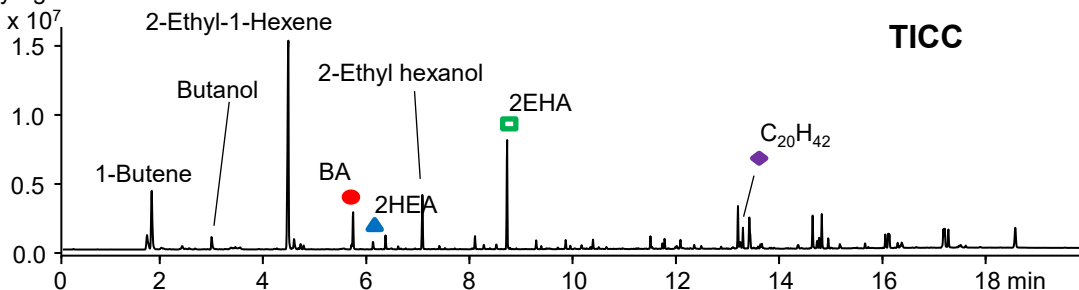


Fig. 2 Pyrogram of a commercial acrylic PSA sample.

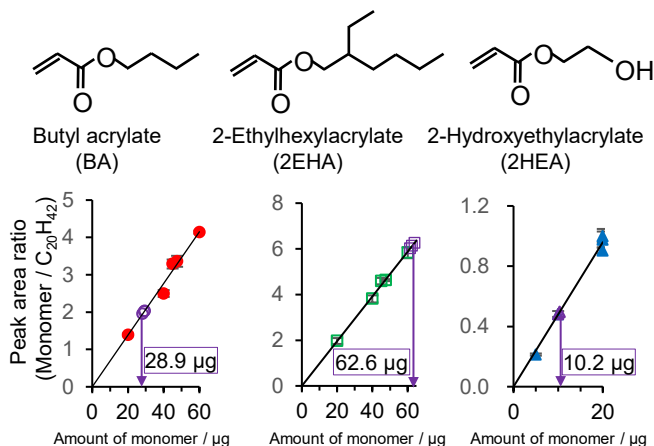


Fig. 1 Calibration curves for monomers and the determined monomer mass in a PSA sample.

Table 1 Monomer compositions of a PSA sample.

Monomers	Mass compositions		
	Butyl acrylate (BA)	2-Ethylhexyl acrylate (2EHA)	2-Hydroxyethyl acrylate (2HEA)
Mean monomer amount ($n = 3$)	2.00 $\pm$ 0.03	6.13 $\pm$ 0.02	0.49 $\pm$ 0.04
Compositions (normalized)	28.4 wt%	61.5 wt%	10.1 wt%
Formulation	30.0 wt%	60.0 wt%	10.0 wt%

Keywords : Acrylic copolymer, Adhesive, Monomer composition ratio, Acrylic monomer peak

Products used : Multi-Shot Pyrolyzer, UA+5 separation column, Vent-free GC/MS adapter

Applications : Forensic investigation, Compositional analysis

Related technical notes : [PYA1-172E](#)

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