Quantitative analysis of acrylic acid in acrylic pressure-sensitive adhesives by reactive pyrolysis GC/MS with two-step heating

Part 1: Procedures of trimethylsilyl derivatization

[Background] Acrylic pressure-sensitive adhesives (PSAs) have been used in a variety of fields. On the other hand, the composition of monomers is a very important factor in determining the adhesive performance. Since monomers, such as acrylic acid (AA), often used as raw materials for acrylic PSAs contain polar functional groups with high reactivity, it is not easy to accurately quantify AA in PSAs by conventional reactive pyrolysis (Py-) GC/MS. In this note, quantitative analysis of AA in PSAs is examined by reactive Py-GC/MS using *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) as a trimethylsilylation (TMS) reagent,[1] and the effective trimethylsilyl derivatization by a two-step heating method is presented.

[Experimental] A model acrylic PSA sample containing 6 % AA was synthesized by solution polymerization. Figure 1 shows the TMS derivatization procedures. Dichloromethane (DCM) was added to the dried PSA sample to dissolve it, and then BSTFA was added. The sample solution was heated at 60 ºC for 30 min to promote TMS reaction (1st heating). Then, 5 µL of the sample solution was taken in a sample cup and heated at 100 ºC for 10 min to remove residual BSTFA (2nd heating). As an internal standard, anthracene-d_{10} (Anth-d_{10}) was added. A Py-GC/MS system equipped with a pyrolyzer (EGA/PY-3030D) directly connected to the GC injector was used. The sample was introduced into the pyrolyzer furnace heated at 600 ºC.

[Result] Figure 2 shows the extracted ion chromatogram (EIC, *m/z* 129) of the TMS-derivatized AA (AA-TMS) obtained by Py-GC/MS measurement immediately after the 1st heating. In the 1st heating, the TMS reaction does not occur at room temperature (R.T.), but heating at 60 ºC for 30 min allows it to proceed. Figure 3 shows the change in the peak area of AA-TMS against the 2nd heating time. When the sample was heated at 100 ºC, the peak area remained almost constant regardless of the heating time. On the other hand, at R.T., the peak area was significantly larger than that at 100 ºC for 10 to 60 min, then decreased and became equivalent to that at 100 ºC after 120 min. The increase of the AA-TMS at R.T. is attributed to the reaction of residual BSTFA with AAs produced by the pyrolysis of acrylic ester units in addition to the originally compounded AA monomers, and the 2nd heating was found to be effective in removing the residual BSTFA. These results suggest that the present two-step heating method is useful for quantitative analysis of AA. In the next note (PYA2-036E), the quantitative analysis of AA in acrylic PSAs using this pretreatment method is reported.

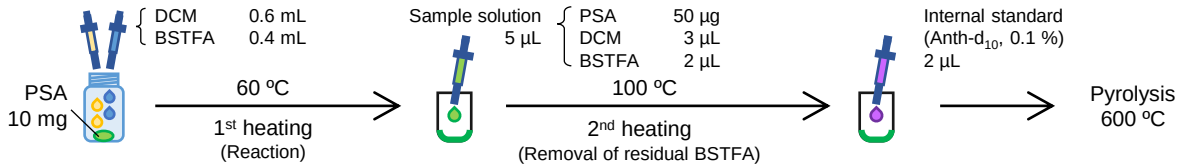

Fig. 1 TMS derivatization procedures.

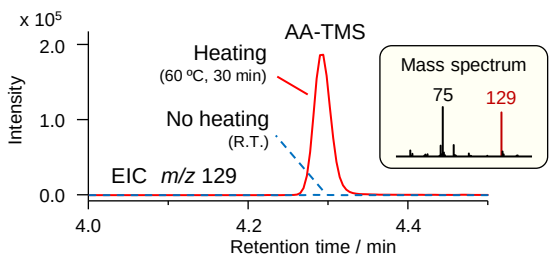

Fig. 2 EIC of AA-TMS with and without the 1st heating.

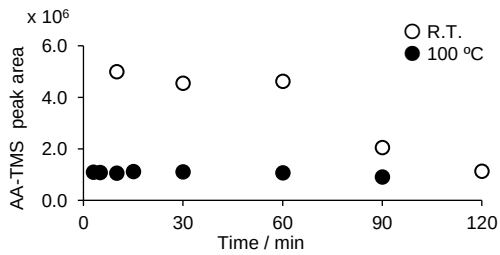

Fig. 3 Peak area of AA-TMS vs. time with and without the 2nd heating.

Furnace temp.: 600 ºC, Separation column: UA+-5 (5 % diphenyl-95 % dimethylpolysiloxane; *L*=30 m, i.d.=0.25 mm, df=0.25 µm), GC Inj. temp.: 300 ºC, Column flow rate: 1 mL/min, Split ratio: 1/100, GC oven temp.: 40 (2 min hold) - 320 ºC (20 ºC/min, 6 min hold), Detector: Quadrupole MS, Sample amount: 50 µg.

1) M. Matsueda *et al., J. Anal. Appl. Pyrolysis* 175 (2023) 106170.

Keywords : Reactive pyrolysis, Pressure-sensitive adhesives, Pretreatment

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UA+-5, Vent-free GC/MS adapter, F-Search

Applications : Quality assurance, Material analysis

Related technical notes : PYA2-036E, PYA2-020E

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