

Curing analysis of two-component epoxy resins

Part 2: Pyrolysis (Py)-GC/MS

[Background] The previous note (PYA3-047E) reported EGA-MS of two-component epoxy resins cured with varied mix ratios of the resin to hardener, and the pyrolysis temperature was determined. This note reports further investigation on the curing state of the epoxy resin with varied mix ratios using Py-GC/MS.

[Experimental] As in the previous note, a GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC injector was used for measurements. An epoxy resin (main component: Bisphenol A diglycidyl ether; BADGE) and a hardener (main component: Isophoronediamine; IPDA) were thoroughly mixed in the volume ratios of 3 : 1 (excess resin), 1 : 1 (stoichiometric), 1 : 3 (excess hardener) and cured for 2 hours at room temperature. Pyrolysis temperature was set at 600 °C.

[Results] Pyrograms of samples are shown in Fig. 1. Bisphenol A (BPA), a pyrolyzate of epoxy resin, was observed in all samples. In the case of the stoichiometric mixture, BADGE and IPDA were barely detected, indicating that all of them properly reacted. In the case of excess resin, unreacted BADGE and its pyrolyzate Bisphenol A monoglycidyl ether (BAMGE) were observed, and in the case of excess hardener, unreacted IPDA was detected. From the above, it was found that Py-GC/MS can be used to evaluate the curing state based on the presence or absence of the peaks derived from either resin or hardener.

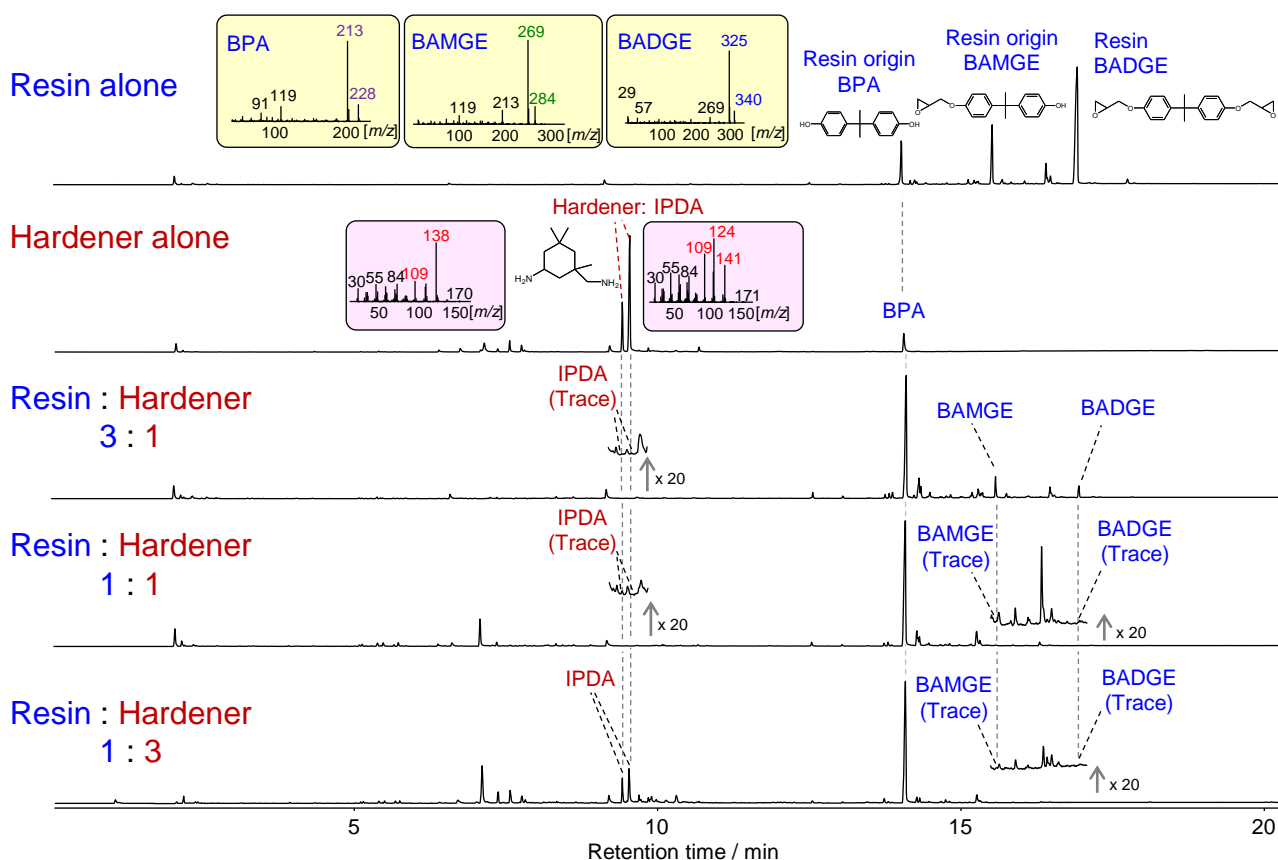


Fig. 1 Pyrograms of epoxy resins.

Furnace temp.: 600 °C, GC oven: 40 °C (2 min hold) - 320 °C (20 °C/min, 13 min hold),
Separation column: UA*-5 (5 % diphenyl 95 % dimethylpolysiloxane, L=30 m, i.d.=0.25 mm, df=0.25 µm),
Column flow rate: 1 mL/min, Split ratio: 1/30, MS scan range: m/z 29 - 600, Sample amount: approx. 0.1 mg.

Keywords : Epoxy resin, Quality assurance, Pyrolysis (Py)-GC/MS

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UA*-5, Vent-free GC/MS adapter, MicroJet Cryo-Trap

Applications : General polymer analysis, Quality assurance, Material analysis, Failure analysis, Differentiation

Related technical notes : PYA3-047E (Part 1), PYA1-034E, PYA1-035E, PYA1-088E

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