Curing analysis of two-component epoxy resins
Part 1: Evolved Gas Analysis (EGA)-MS

[Background] Two-component epoxy resins consist of resin and hardener are versatile adhesives and widely used across diverse applications owing to their excellent mechanical properties and high chemical resistance. Yet, inadequate mixing or insufficient agitation can result in poor curing. In this report, two-component epoxy resins cured with varied resin-to-hardener mix ratios were examined using evolved gas analysis (EGA)-MS.

[Experimental] A GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC injector was used for measurements. A deactivated metal tube (UADTM-2.5N) and a Vent-free GC/MS adapter were used to connect the GC injector to the MS detector. The examined adhesive was a room-temperature-curing two-component epoxy resin comprising a resin (primary component: bisphenol A diglycidyl ether; BADGE) and a hardener (primary component: isophoronediamine; IPDA). The resin and hardener were mixed on a paper at 3:1, 1:1, and 1:3 of resin to hardener ratios followed by curing for 2 hours at room temperature. Among these, the mixtures with ratios of 1:1 and 1:3 successfully cured, while the mixture with the 3:1 ratio resulted in a viscous liquid without curing. Each cured resin's surface was scraped or scratched using a cutter knife, and the collected sample was placed in an Eco-Cup. The Eco-cup was then introduced into the pyrolyzer furnace and EGA-MS measurements were conducted.

[Results] The EGA curves for the collected specimens are shown in Fig. 1(a). For both the resin alone and the resin-to-hardener ratio of 3:1, two peaks were observed in the temperature zones of 250-350 ºC and 350-450 ºC. For hardener alone and resin to hardener ratios at 1:1, 1:3, a single peak was observed at 100 ºC and in the 350-400 ºC zone. The furnace temperature for pyrolysis (Py)-GC/MS was determined to be 600 ºC based on the tailing of the peak derived from the polymer component in EGA curves. The next note (PYA1-159E) reports the result of the Py-GC/MS analysis of each collected sample.

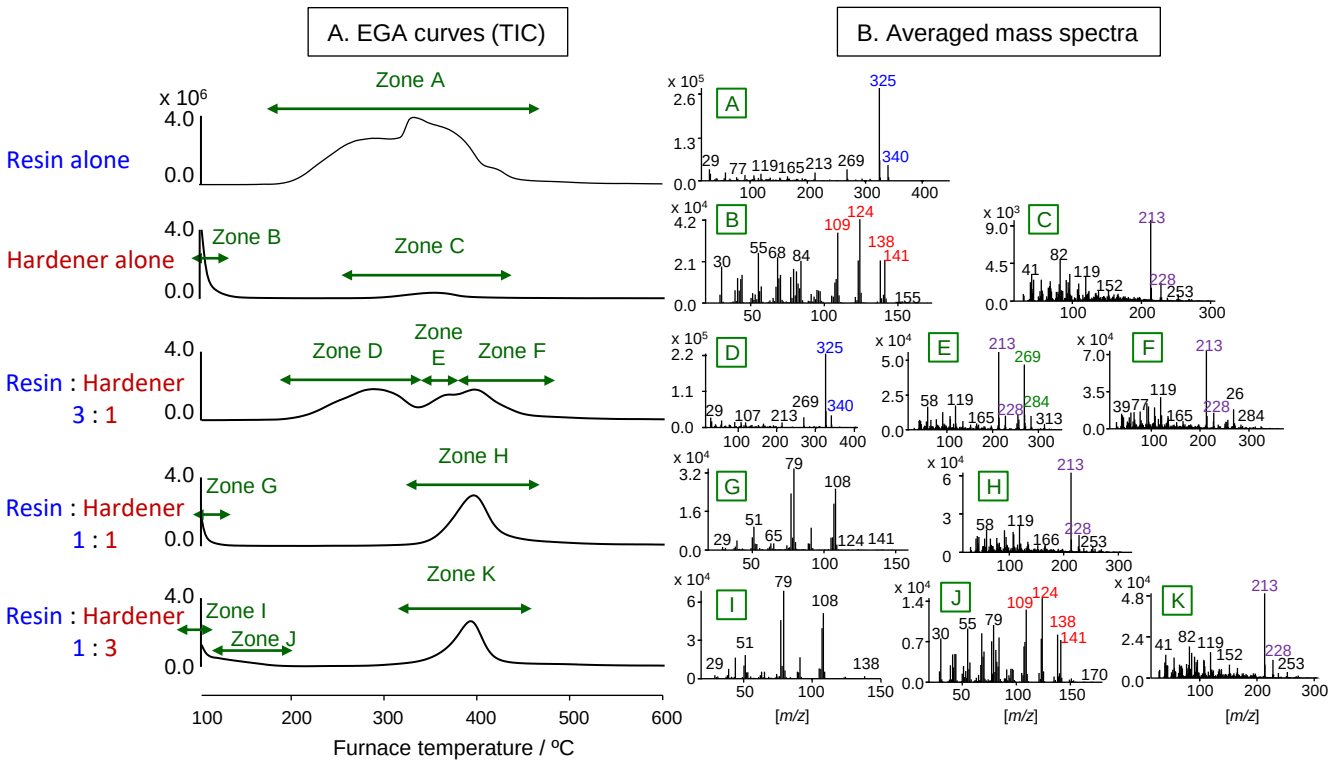

Fig. 1 (a) EGA curves (TIC) and (b) Average mass spectra a two-component epoxy resin.

Furnace temp.: 100 - 700 ºC (20 ºC/min), EGA tube: UADTM-2.5N (*L*=2.5 m, i.d.=0.15 mm), Column flow rate: 1 mL/min (He), Split ratio: 1/50, GC oven temp.: 300 ºC, MS scan range: *m/z* 29 - 600, MS scan rate: approx. 0.2 scan/s, Sample amount: approx. 0.1 mg.

Keywords : Epoxy resin, Quality assurance, Pyrolysis (Py)-GC/MS, Evolved Gas Analysis (EAG-MS)

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UADTM-2.5N, Eco-Cup LF, F-Search, Vent-free GC/MS adapter

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

Related technical notes : PYA1-159E (Part 2), PYA3-041E

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