Cure failure analysis of a two-component epoxy resin

[Background] Two-component (resin and hardener) epoxy resins are used in a wide range of applications due primarily to their high mechanical strength and chemical stability; however, “cure failure” arising from incorrect mix ratios and poor mixing may degrade product performance. This note illustrates how “cure failure”, due to incorrect mixing ratios can be investigated using pyrolysis (Py)-GC/MS.

[Experimental] Epoxy resin (major component: bisphenol A diglycidyl ether, BADGE) and hardener (major component: isophoronediamine, IPDA) were mixed thoroughly by volume ratios 3:1 (excess resin), 1:1 (equal amounts), and 1:3 (excess hardener). Each sample (thickness ca. 1 mm) was cured for two hours at room temperature prior to analysis. The analytical system used for the Py-GC/MS analysis consisted of a Multi-Shot Pyrolyzer (EGA/PY-3030D) interfaced directly to the injector of a GC/MS system. Flash pyrolysis was performed at 600ºC.

[Results] Epoxy resin samples with a 1:1 mix ratio (equal amounts) and 1:3 mix ratio (excess hardener) completely cured; however, the sample with a 3:1 ratio (excess resin) did not cure and ended up as a viscous liquid. The pyrograms of the three samples are shown in Fig. 1. Bisphenol A (BPA), a pyrolyzate of epoxy resin was observed in all three pyrograms. Since residual BADGE and IPDA were not observed in the1:1 mix ratio sample, one can conclude that these stoichiometrically reacted in the curing process. When the resin was in excess, BADGE and its pyrolyzate, bisphenol A monoglycidyl ether (BAMGE) were observed, while when there was excess hardener, unreacted IPDA was observed. These three pyrograms show how Py-GC/MS is used to characterize the epoxy resins whose performance is affected by various in-use factors (e.g., mixing ratio, uniformity of the mixing, storage temperature, cure time, *etc.*).

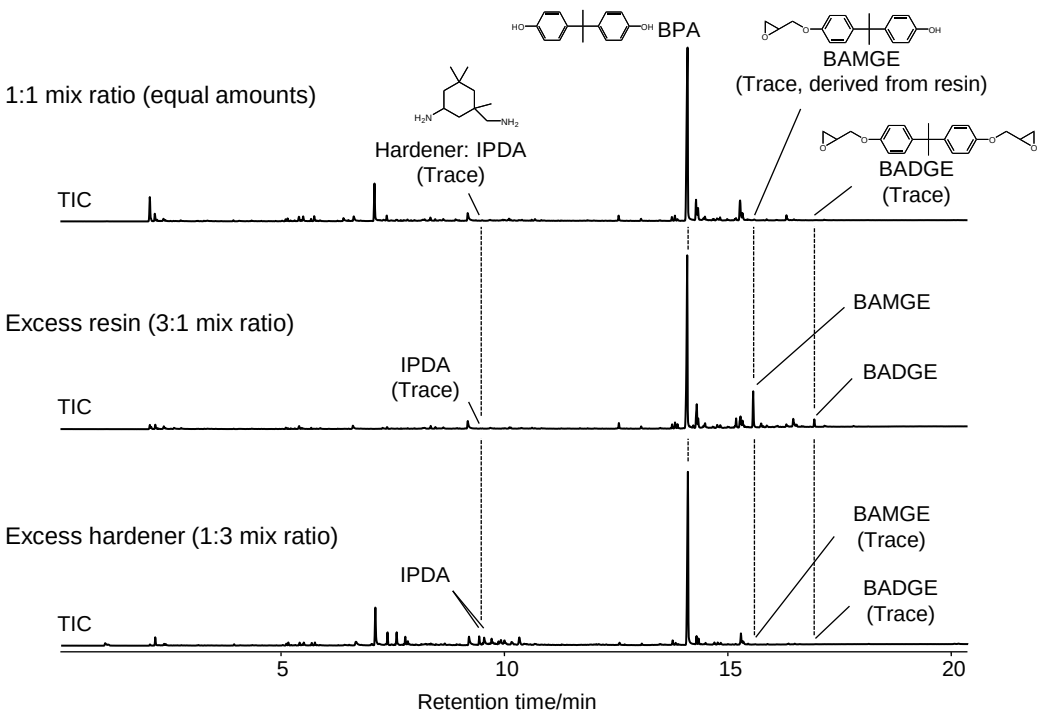

Fig. 1 Pyrograms of a two-component epoxy resin

Furnace temp.: 600ºC, GC oven temp.: 40ºC (2 min hold) - 320ºC (20 ºC/min, 13 min hold)
Separation column: Ultra ALLOY⁺-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: ca. 0.1 mg

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

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

Applications : General polymer analysis, Defect analysis

Related technical notes : PYA1-034E, PYA1-035E, PYA1-088E

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