Offensive odor analysis of acrylic adhesive by thermal desorption (TD)-GC/MS

[Background] Acrylic pressure-sensitive adhesives (PSAs) have been widely used in various fields such as electronic, aerospace, and automotive industries. The heat curing process of PSAs sometimes causes an offensive odor. In this note, a defective acryl-based adhesive and a good (i.e., no odor during the heat curing) adhesive were analyzed by evolved gas analysis (EGA)-MS and TD-GC/MS. The differences in the two sets of data enable the process engineer to identify and eliminate problematic batches of adhesive.

[Experimental] The adhesive layer was removed and collected from the good and the defective acrylic adhesive tapes prior to analysis. EGA-MS and TD-GC/MS measurements were done by a pyrolysis-GC/MS system with a Multi-Shot Pyrolyzer directly interfaced to the GC injector. The furnace temperature was programmed from 100 ºC to 700 ºC at 20 ºC/min for EGA-MS and from 100 ºC to 200 ºC at 20 ºC/min for TD-GC/MS. Volatile components released from the sample were cryo-trapped at the head of the GC column prior to chromatographic separation.

[Results] The EGA thermograms of the good and defective adhesives are shown in Fig. 1. While, the thermograms are similar, they do differ in the temperature range from 100 ºC to 200 ºC (Zone A). Zone A corresponds to the process temperature at which the offensive odor was generated. The chromatograms of Zone A obtained by TD-GC/MS is shown in Fig. 2. Peaks derived mainly from the solvent and the monomer of the adhesive are observed. Only in the defective product, the presence of carbon disulfide which has a characteristic unpleasant odor is observed. In addition, the defective adhesive has a large peak for 2-ethylhexyl acrylate, the monomer. These results show that the defective adhesive contains carbon disulfide, the cause of the offensive odor, and that the polymerization of the monomer was inadequate. Also, a large amount of 2-ethylhexyl acrylate, which has an unpleasant odor, was present in the defective product.

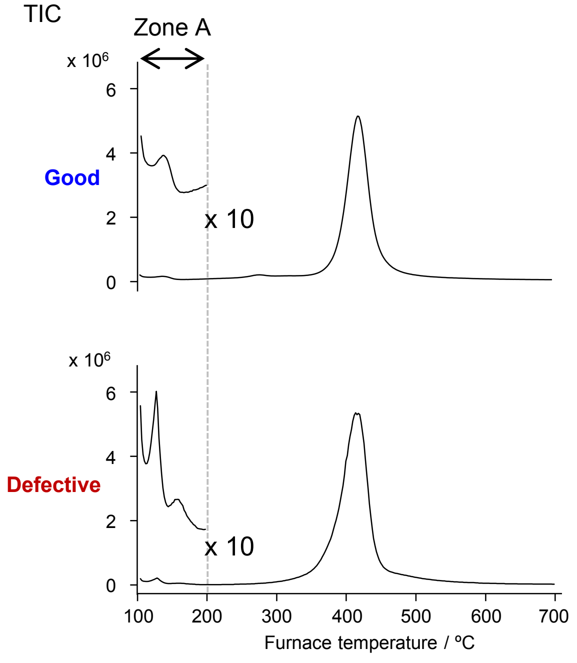

Fig. 1 EGA thermograms of good and defective products

Furnace temp.: 100ºC - 700ºC (20 ºC/min),
EGA tube: UADTM-2.5N (*L*=2.5 m, i.d.=0.15 mm),
Tube flow rate: 1 mL/min (He), Split ratio: 1/50,
GC oven temp.: 300ºC, Sample amount: ca. 0.2 mg.

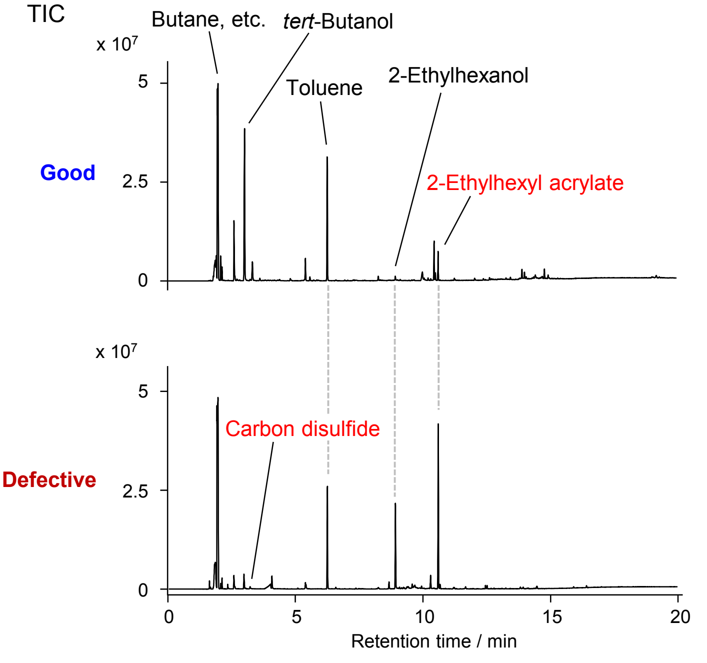

Fig. 2 TD-GC/MS chromatograms of Zone A of good and defective products

Furnace temp.: 100 ºC – 200 ºC (20 ºC/min, 1 min hold),
Sep. col.: UA⁺-1 (dimethylpolysiloxane, *L*=30 m, i.d.=0.25 mm, df=1.0 μm),
Column flow rate: 1 mL/min (He), Split ratio: 1/10,
GC oven temp.: 40 ºC (2 min hold) – 320 ºC (20 ºC/min, 4 min hold),
Sample amount: ca. 5 mg,
Thermally desorbed components cryo-trapped using MicroJet Cryo-Trap.

Keywords : Defect analysis, Quality assurance, Acrylic adhesive, EGA-MS, TD-GC/MS

Product used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UA$^+$-1, Eco-Cup LF, F-Search, Vent-free GC/MS adapter, MicroJet Cryo-Trap

Applications : General polymer analysis, Quality assurance

Related technical notes : PYA1-088E, PYA1-006E

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