Analysis of oil-based black inks

- Comparison of good and defective inks -

[Background] Pyrolysis (Py)-GC/MS is widely used for the quality assurance of organic polymeric materials; however, it is often difficult to clearly identify subtle differences in the pyrograms of similar samples. These differences may become more apparent by analyzing fractions of each sample and comparing the results. To illustrate the value of this approach, two samples were analyzed. The results obtained from the analysis of a “good” and “defective”-[non-standard drying characteristics] oil-based black inks are compared. First, each sample is analyzed using EGA-MS. The thermograms are used to define the temperature range over which the volatiles (solvents, monomers, *etc*.) and semi-volatile additives evolve. Following this analysis, the residual sample is pyrolyzed. This second analysis provides information about the base polymer.

[Experimental] The samples are both oil-based black inks. 0.5 mg of a good ink and a defective ink were placed in sample cups. The Py-GC/MS system used for the analysis consisted of a Multi-Shot Pyrolyzer that was directly interfaced to a GC/MS system. EGA-MS analysis was performed using a temperature program: 100ºC to 600ºC at a rate of 20 ºC/min. The TD-GC/MS was performed by programming the furnace from 100ºC to 160ºC at a rate of 20 ºC/min. The evolved gases were cryo-trapped at the head of a separation column prior to GC/MS analysis.

[Results] EGA thermograms of the good and defective inks are shown in Fig. 1. Both thermograms are similar but the evolved gas profiles differ: Zone A (100ºC to 160ºC). The total ion chromatograms of Zone A, obtained using TD-GC/MS analysis are shown in Fig. 2. The defective ink has a large 2-naphthol peak (identified using F-Search). As expected, at higher temperatures, peaks associated with solvents and pyrolyzates of acrylic resin and dyes are observed in both the good and defective inks. Both inks contain a dye (Solvent Black 29) that has a chemical structure in which 2-naphthol and a phenol derivative are azo-bonded (the inset of Fig. 2). It is apparent that the defective ink contains unreacted 2-naphthol, which may be the source of the drying problem.

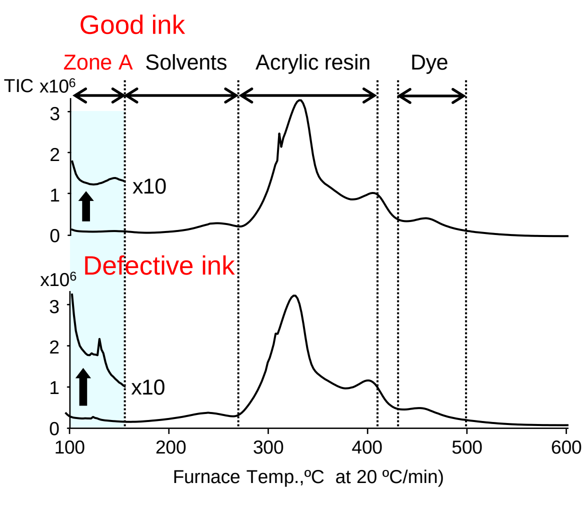

Fig. 1 EGA thermograms of good and defective inks

Furnace temp.: 100ºC – 600ºC (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: 1/30,
GC oven: 300ºC, Sample amount: ca. 0.5 mg.

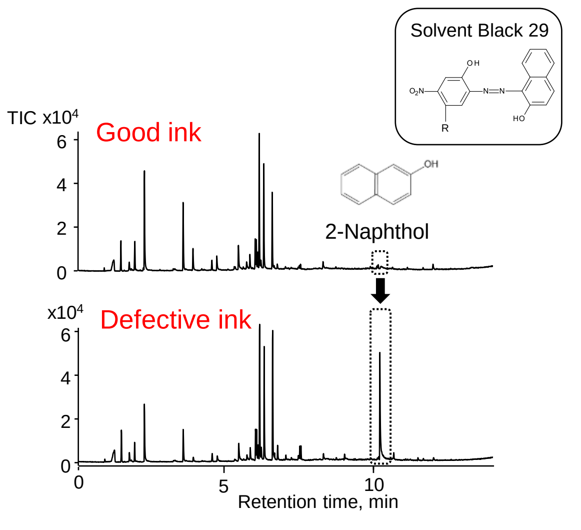

Fig. 2 TD-GC/MS chromatograms of good and defective inks in Zone A

Furnace temp.: 100ºC – 160ºC (20 ºC/min),
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 (He), Split ratio: 1/30,
GC oven: 40ºC (2 min hold) – 300ºC (3 min hold, 20 ºC/min),
Sample amount: ca. 0.5 mg,
Thermally desorbed components were cryo-trapped by MicroJet Cryo-Trap (MJT-1035E).

Keywords : Defective ink analysis, Ink, Dye, Quality assurance, EGA-MS, TD-GC/MS, F-Search

Product used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UA+-5, Eco-Cup LF, F-Search, Vent-free GC/MS adapter, MicroJet Cryo-Trap

Applications : General polymer analysis, Quality assurance

Related technical notes : PYA1-067E

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