

Quantitative Analysis of A Cationic Polymer Having A Quaternary Ammonium Salt on A Polymer Sheet by Py-GC/MS

[Background] Various cationic polymers with a quaternary ammonium salt are used as dye fixing agents and antimicrobials in textile, paper, and film industries. Because the amount of ammonium salt added to the products greatly influence the product performance, the quantitative analysis of the ammonium salt is very important. However, complicated pretreatments such as solvent extractions of interfering species are usually required, because coexisting organic compounds may interfere the analysis. Therefore, the development of a simpler analytical methodology has been long been desired. Previously, a simple quantitative analytical method using thermal desorption-GC/MS as an application of a Double-Shot Pyrolyzer® has been developed (PYA1-049E). Here, this analytical method was applied to real samples.

[Experimental] As real samples, standard samples (A-C) in which ca. 100-600ppm of polyquaternium-4 (PQ-4) was coated on three polyethylene terephthalate (PET) sheets, each coated with different concentrations, were used. For the Py-GC/MS system, a Double-Shot Pyrolyzer® (Frontier Lab. Ltd.) directly attached to the split/splitless GC injection port was connected to a MS via a separation column. At the head of the separation column, a MicroJet Cryo-Trap was attached to cold-trap volatile components.

[Results] Fig. 1 shows a chromatogram of thermally desorbed components of ca. 10mg of PET sheet coated by 345ppm of PQ-4 obtained at temperatures up to 340°C. A peak for chloromethane was observed at a retention time of about 1min. The reproducibility of the peak area for chloromethane's characteristic ion at m/z 50 in the mass spectrum was found to be 3.46%. Further, as shown in Fig. 2, a good linear calibration curve was created from varied concentrations of coatings on sample sheets and its correlation coefficient was found to be 0.994. These results indicate that this is a powerful quantitative analytical method for PQ-4.

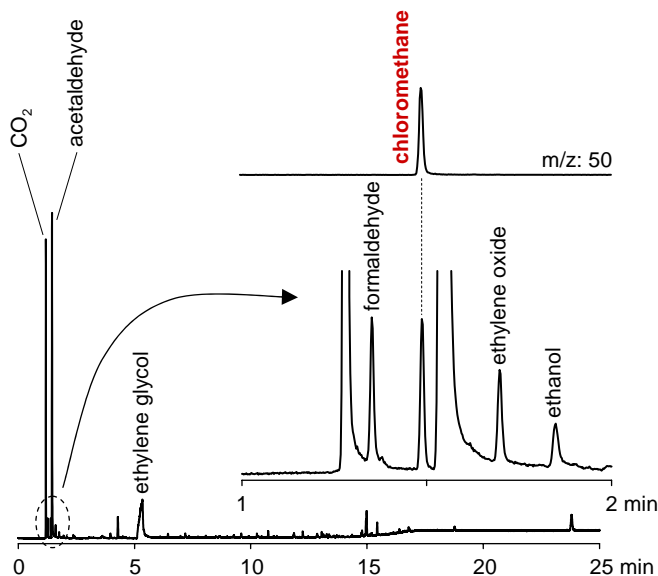


Fig. 2 Chromatogram of a PET sheet coated by PQ-4 obtained by thermal desorption-GC/MS

Pyrolyzer furnace temp.: 100-340°C (20°C/min, 2min hold), Flow rate : 1ml/min, split ratio : 1/50
 Separation column : Ultra ALLOY5+ (5% diphenyl 95% dimethylpolysiloxane, length 30m id 0.25mm, film thickness : 1.0µm), GC oven temp.: 40°C (2min hold) - 300°C (20°C/min)
 Sample size : 5.64µg, MS scan range : 29-600 (m/z), Scan rate : 2 scan/sec

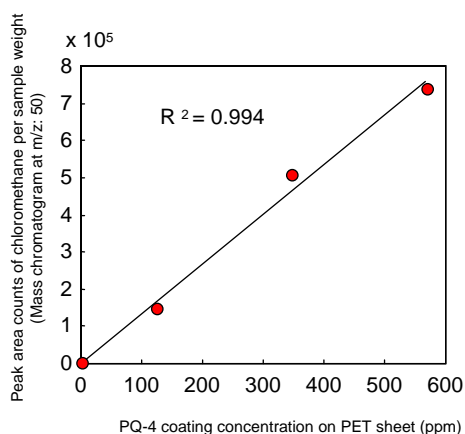


Fig. 2 Calibration curve for PQ-4 coated on PET sheets

Keyword : Cationic polymer, quaternary ammonium salts, polyquaternium, Thermal desorption

Applications : Paper industry, Film manufacture, General polymer analysis

Related technical notes : PYA1-049E, PYT-004E, PYT-007E, PYT-014E

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