Evaluation of the aged deterioration of PE pipes used for a hot-water heating system (1)

[Background] To understand the deterioration mechanism of polymeric products, it is necessary to evaluate the chemical changes in the polymers and additives. Evolved gas analysis (EGA) and thermogravimetry are complementary techniques[1]. Both provide information about the thermal properties of polymeric materials. This note describes the analytical results obtained by EGA-MS of deteriorated cross-linked polyethylene (PE-Xb) pipes used in a hot-water heating system.

[Experimental] EGA-MS measurements were done by a Multi-Shot Pyrolyzer (EGA/PY-3030D, Frontier Labs) interfaced directly to the split/splitless injection port of a GC/MS system. PE-Xb samples were collected by scraping the surfaces of the inner wall of “new” and “used” pipes. 0.2 mg of the sample was placed in a deactivated stainless sample cup.

[Results] EGA-MS thermograms of “new” and “used” PE-Xb pipes are shown in Fig. 1. A single peak due to the thermal decomposition of PE is observed in both thermograms. A slight decrease in the peak top temperature of the “used” sample suggests some structural differences in the polymer. To focus on the additive analysis, the sample weight was increased to 1 mg, and the split ratio and furnace maximum temperature were set to 1/10 and 400ºC, respectively. The EGA thermograms are shown in Fig.2. The “new” sample shows small peaks (300 – 350ºC) prior to the PE decomposition in the extracted ion chromatograms (EICs) of a fragment ion of Irgafos168 (*m/z* 441) and of Irganox 1076 (*m/z* 530), compared to the “used” one.

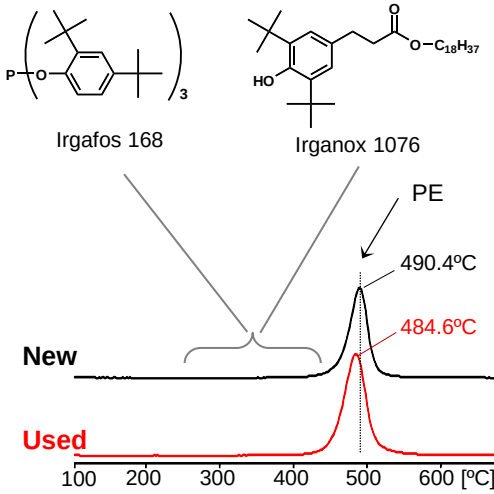

Fig.1 EGA thermograms of PE-Xb samples

Furnace temp.: 100 – 400ºC (20 ºC/min), EGA tube at 300ºC, *L*=2.5 m, i.d.=0.15 mm, Column flow rate (He): 1 mL/min, Split ratio: 1/50, Sample amount: ca. 0.2 mg

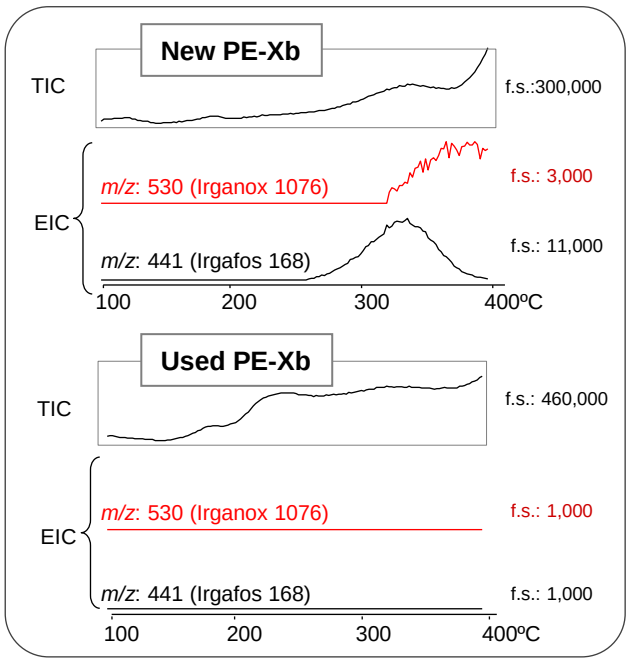

Fig.2 EGA thermograms of new and used PE-Xb samples (100 - 400ºC, TIC and EICs)

Sample amount: 1 mg, Split ratio: 1/10, Others condition: same as in Fig.1

Ref 1) A. Shiono, *et al., Polym. Test,* **2015**, *42*, 54–61.

Keywords : EGA, additives, antioxidant, Irganox 1076, Irgafos 168, cross-linked polyethylene, deterioration

Products used : Multi-functional pyrolyzer, Vent-free GC/MS Adapter, UADTN-2.5(EGA Capillary Tube), Eco-Cup LF

Applications : Analysis of additives

Related technical notes : PYA1-004E, PYA1-085E

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Frontier Laboratories Ltd.
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