Photo, thermal and oxidative degradation of EPDM rubber using online UV irradiation PY-GC/MS

[Background] Ethylene propylene diene rubber (EPDM) is widely used in auto parts because it reduces thermal and weather degradation. The online ultraviolet (UV)/Py-GC/MS system can be used to rapidly evaluate the photo, thermal, and oxidative degradation of EPDM rubber.

[Experimental] EPDM rubber (JSR. EP25, diene based rubber containing 5.1 wt% of ethylidene norbonene) was used as a test sample. 200 µg of the sample was placed in a sample cup which was attached to the end of the optical fiber on the UV irradiator (UV-1047Xe, 280-450 nm, 700 mW/cm^2) equipped with a xenon/Hg light source. The furnace temperature of the Multi-Shot Pyrolyzer (EGA/PY-3030D) was set to 60ºC and the cup was positioned in the furnace. The sample was irradiated in an air atmosphere for one hour. Volatile degradation products released during the irradiation were cyro-trapped at the head of a metal capillary separation column (Ultra ALLOY$^+$-1) using liquid nitrogen. After one hour of irradiation, the liquid nitrogen trap was removed and the volatiles analyzed using GC/MS. The irradiated sample residue was analyzed using evolved gas analysis (EGA)-MS[1].

[Results] A comparison of the volatile products released from the EPDM rubber sample during UV irradiation and from the control EPDM sample (no UV irradiation) is shown in Fig. 1. Acetaldehyde, acetone, acetophenone, acetic acid, and propylene are derived from the propylene unit. Straight-chain aldehydes such as nonanal are derived from the ethylene sequence. The EGA thermograms of the EPDM rubber sample before and after UV irradiation are shown in Fig. 2. The EGA thermogram of EPDM rubber which was irradiated exhibits a decrease in the intensity for the peak near 480ºC. The peak apex temperature shifts to lower temperatures by 10ºC and the half height width (Wh) increases from 30ºC to 50ºC. These results indicate that the photo, thermal, and oxidative degradation of EPDM rubber can be evaluated using the peak intensity, peak apex temperature, and the Wh of the peak in the thermogram. It is also shown that the UV degradation of EPDM can be evaluated in a few hours using the online UV/Py-GC/MS.

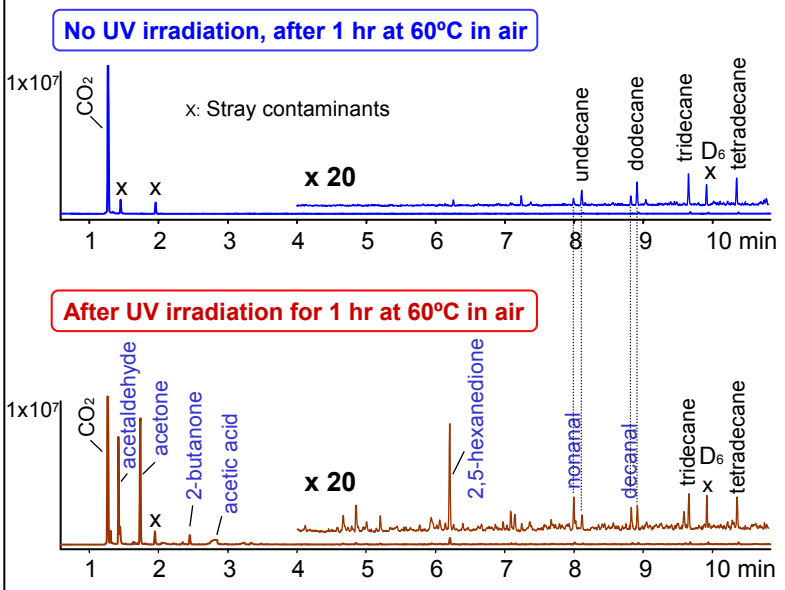

Fig. 1 Volatile degradation products formed after UV irradiation

GC oven temp.: 40ºC (2 min hold) – 280ºC (20 ºC/min)
Separation column: Ultra ALLOY$^+$-1 (dimethylpolysiloxane),
L=30 m, i.d.=0.25 mm, df=0.5 µm
Column flow rate: 1 mL/min He, Split ratio: 1/50, Sample wt.: 0.2 mg

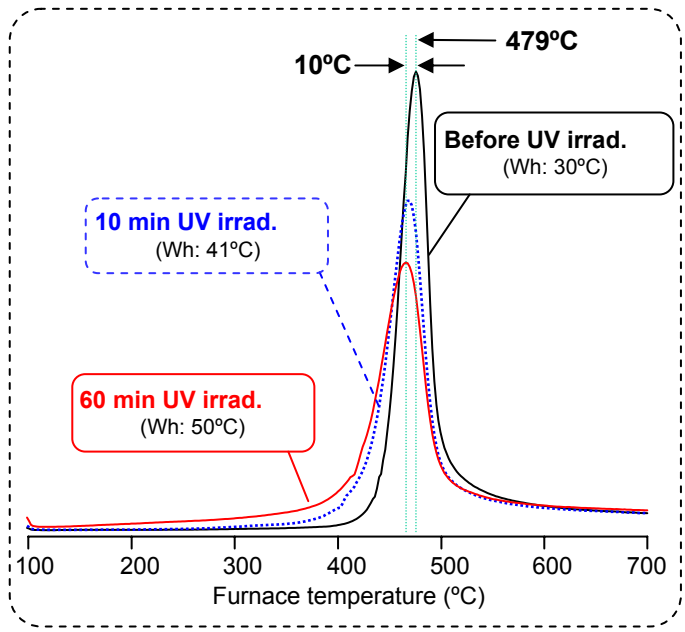

Fig. 2 EPDM thermograms before and after UV irradiation

Py. temp.: 100 – 700ºC (20 ºC/min)
GC oven temp.: 300ºC
EGA tube: deactivated metal tube, L=2.5 m, i.d.=0.15 mm,
Column flow rate: 1 mL/min He, Split ratio: 1/50, Sample wt.: 0.2 mg

Ref.: 1) Technical note PYA5-004E

Keyword : EPDM, UV irradiator, Weather meter, EGA, Photo, thermal, oxidative degradation, Volatile degradation products

Applications : Weatherability test

Related technical notes : PYA5-005E

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