

Characterization of the Network Structures in UV-Cured Acrylic Ester Resin by Reactive Py-GC

[Background] Py-GC is a powerful tool for the characterization of network structures in intractable polymeric materials. Py-GC, in the presence of an organic alkali such as tetramethylammonium hydroxide (TMAH), can be used to analyze the structures of insoluble cross-linking polymers containing polar components. This note describes the characterization of the network structures in an UV-cured acrylic ester resin, prepared from prepolymer compounds containing ester linkages.

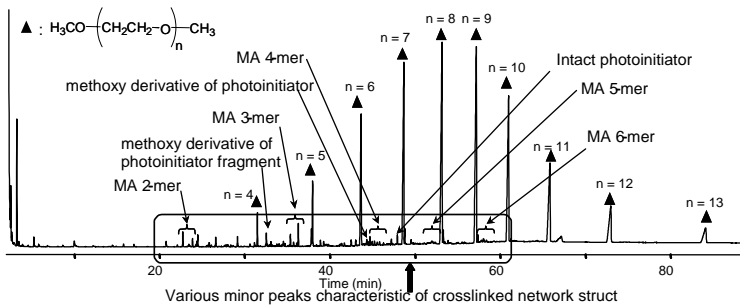
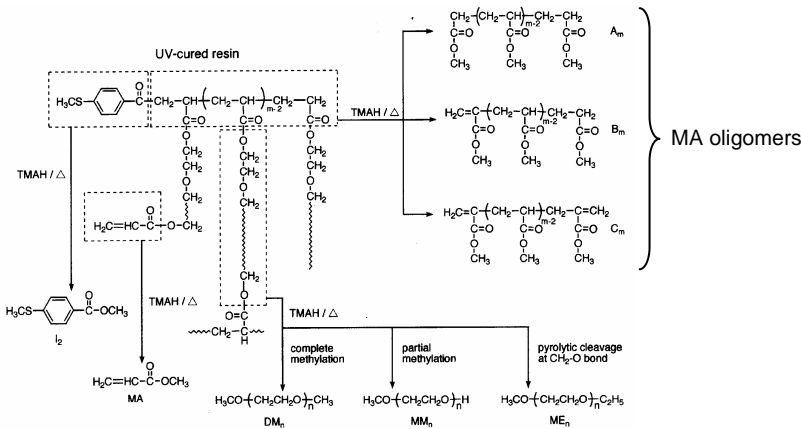


Figure 1. Typical pyrogram of UV-cured resin prepared from polyethyleneglycol diacrylate and morpholino-type photo-initiator.

Pyrolysis temp. : 400°C, GC oven temp. : 35°C (6 min)-5 °C/min-340°C
 Separation column : Poly(5% diphenyl)dimethylsiloxane
 Length 30 m, 0.25 mm i.d., Film thickness 0.25 µm
 Carrier gas flow : 50 mL/min, Column flow : 1.0 mL/min, Detector: FID

[Experimental] A UV-cured resin prepared from polyethylene-glycol diacrylate and morpholino-type photoinitiator (IRGACURE 907) is subjected to Py-GC. First the resin is ground into a fine powder using a freezer mill. About 100 µg of the powder is subjected to reactive Py-GC at 400°C in the presence of 4 µL of 25% TMAH methanol solution.

[Results] Fig. 1 shows a typical pyrogram of the UV-cured resin. In this pyrogram, minor but distinct peaks of various methyl acrylate (MA) oligomers directly reflecting the cross-linked structures are observed along with a series of methoxy derivatives of ethyleneglycol oligomers. The derivatives originate from the main chain after chemolysis at the acrylate linkages (Scheme 1). The relative peak intensities of the various MA units enable the analyst to determine the chain length distribution of the network junctions contained in the UV-cured resin.



Scheme 1. Formation pathway of typical products of the UV-cured resin after reactive pyrolysis

*Contents excerpted from H. Matsubara, A. Yoshida, Y. Kondo, S. Tsuge, H. Ohtani, *Macromolecules* **2003**, 36, 4750-4755.

Keyword : UV-cured resin, Network structures, Chain length distribution of network junctions, Reactive Py-GC

Applications : Analysis of cross-linking polymers

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