Kinetic Reaction Analysis of an Anhydride-Cured Thermoplastic Ep-oxy:PGE/NMA/BDMA

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ABSTRACT:

A comprehensive reaction analysis of a linear epoxy resin cured with an anhydride was performed to evaluate the reaction rate expressions. Monomers included phenyl glycidyl ether and methyl- 5-norbornene-2,3-dicarboxylic anhydride or nadic methyl anhydride; the catalyst was *N*,*N*-dimethylbenzylamine; the initiator was *n*-propanol. Emphasis was initially placed on the molar dynamics of monomeric and oligomeric molecules. Molecular fractionations were achieved using reversed phase, high performance liquid chromatography. Chemical reaction rate constants were examined as a function of degree of polymerization. For the chain-initiated polymerization, the initiation rate constant was observed to be approximately 3 times greater than the propagation constant associated with oligomeric molecules. Both Poisson and Gold distributions were used to fit data. Examinations of polymeric fractions obtained by gel permeation chromatography in conjunction with a multiangle laser light scattering photometer revealed a minor side reaction that broadened the polydispersity index and resulted in the reduction of the cumulative, molar concentration of molecules as a function of conversion.

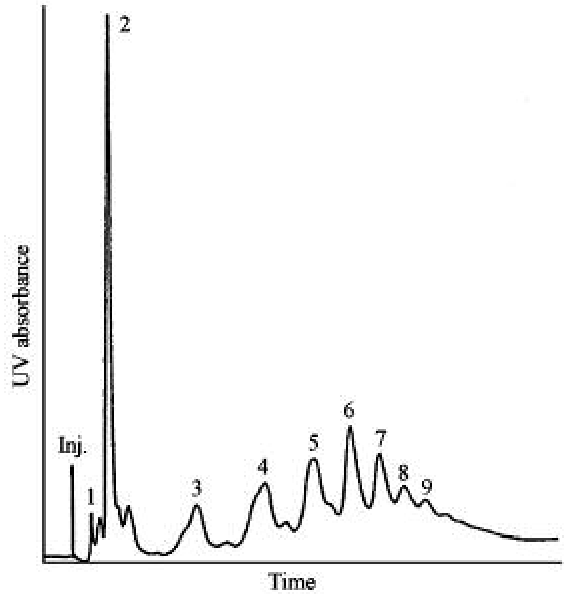

Figure 1. Representative HPLC chromatogram for the PGE/NMA/BDMA cure (initiator/anhydride ratio 0.15, 80 °C, 240 min): (1) NMA; (2) PGE; (3) P_1; (4) P_2; (5) P_3; (6) P_4; (7) P_5; (8) P_6; (9) P_7.

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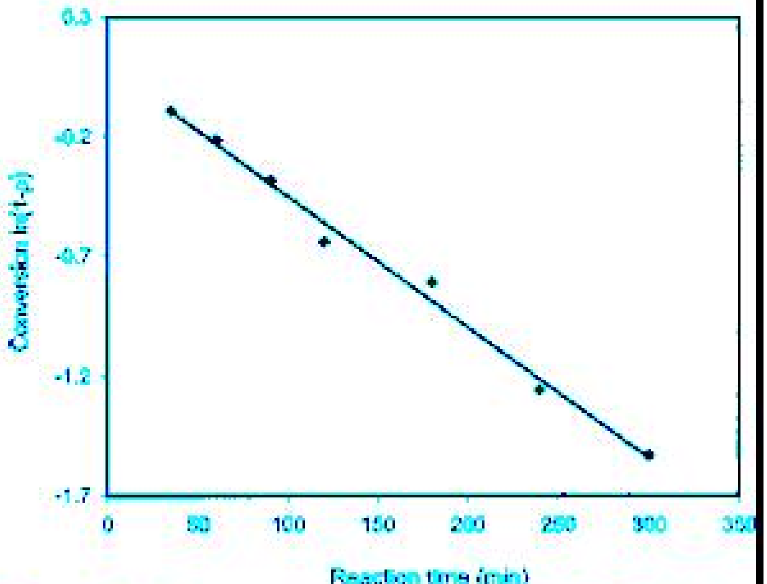

Figure 2. Monomer decay for PGE/NM (80 °C, initiator/anhydride ratio 0.15).

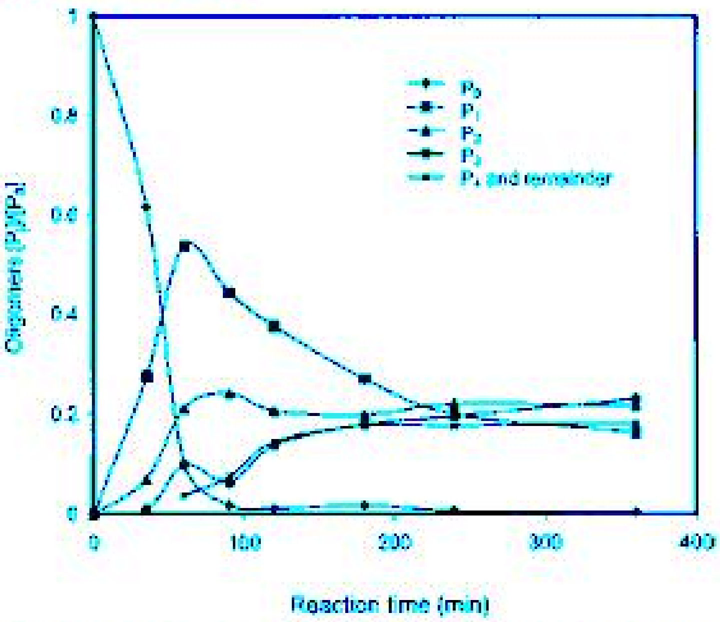

Figure 3. Oligomeric dynamics for PGE/NMA/BDMA (80 °C initiator/anhydride ratio 0.15).

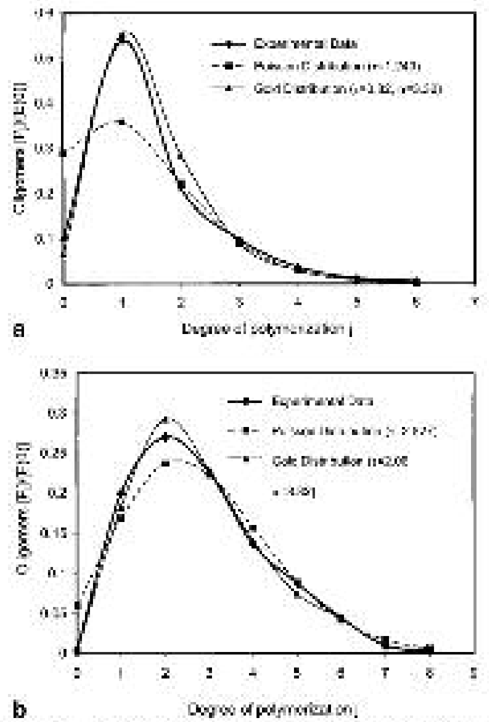

Figure 4. Theoretical experimental comparison of PDDs: (a) PGE/NMA/BDMA (60 min at 80 °C, initiator/anhydride ratio 0.1); (b) PGE/NMA/BDMA (180 min at 80 °C, initiator/anhydride ratio 0.15).

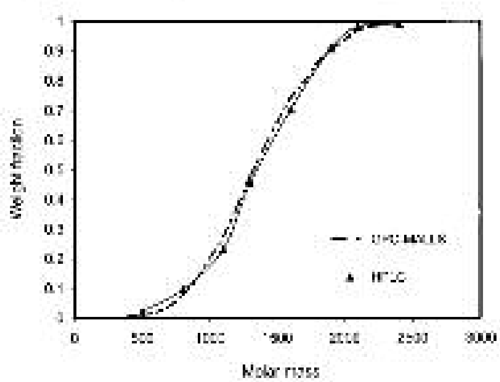

Figure 5. Comparison of cumulative weight fractions by HPLC and GPC–MALLS (PGE/NMA/BDMA 240 min, 80 °C, initiator/anhydride ratio 0.15).

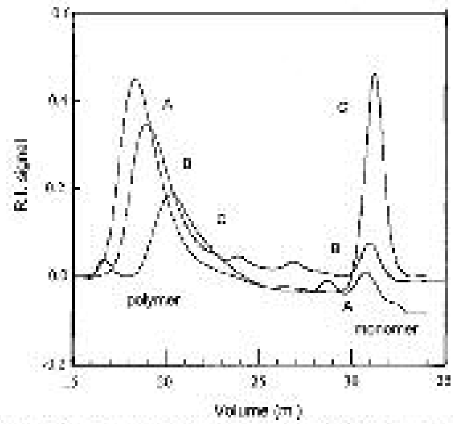

Figure 6. Chromatograms of PGE/NMA/BDMA reaction mixture (80 °C, initiator/anhydride ratio 0.01): (A) 240 min; (B) 160 min; (C) 60 min.

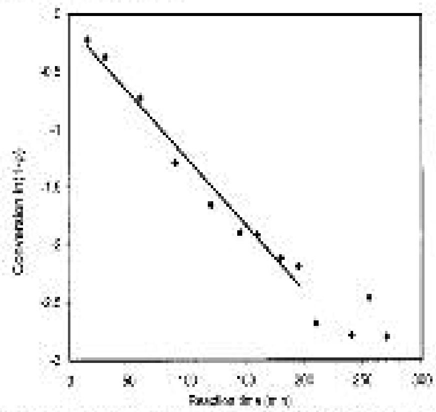

Figure 7. Monomer decay for PGE/NMA/BDMA at 80 °C (initiator/anhydride ratio 0.01).

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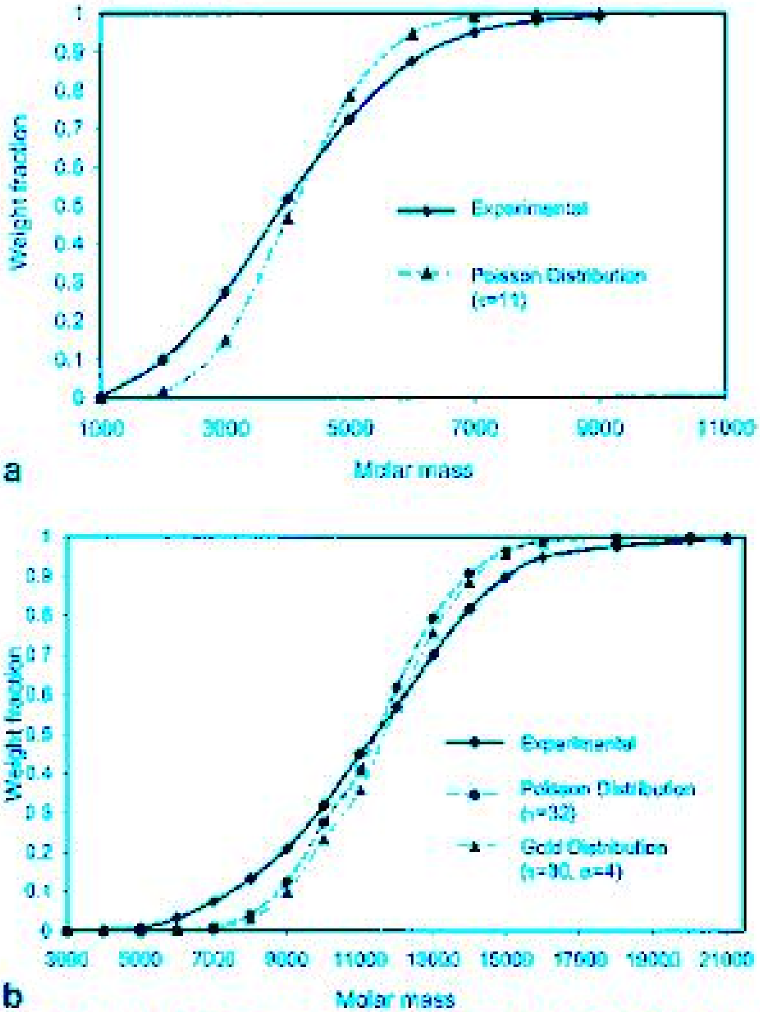

Figure 8. Molecular weight distribution of (a) PGE/NMA/BDMA (60 min, 90 °C, initiator/anhydride ratio 0.01) and (b) PGE/NMA/BDMA.