Characterization of the Temperature Dependent Chemical and Mechanical Properties of a Diels-Alder Based Crosslinked Polymeric Material

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A cross-linked polymeric material was formed by the Diels-Alder reaction between trisfuran and bismaleimide monomers. At elevated temperatures, chemical equilibrium shifts from the Diels-Alder reaction to the retro-Diels-Alder reaction, resulting in depolymerization. As a consequence, the material is both reversibly gelled and vitrified, existing as a liquid, polymer gel, or polymer glass, depending on the temperature and relative reaction progress. FTIR spectroscopy was used to characterize the conversion of furan and maleimide functionalities to the oxy-norbornene adduct. Equilibrium conversion of the furan and maleimide varied from 74 % at 85 degrees Celsius to 24% at 155 degrees Celsius, demonstrating significant reversion via the retro-Diels-Alder reaction. At lower temperatures vitrification and the subsequent mass transfer restrictions limit the extent of reaction rather than equilibrium. As a consequence the highest conversion realized was 87% at 45 degrees Celsius. The thermomechanical properties of the material were characterized by rheometry. Both the storage and loss moduli were found to decrease with increased temperature due to decreasing crosslink density, a result of the retro-Diels-Alder reaction. The gel point, as determined by the Chambon-Winters criterion, occurs at 92 degrees Celsius, corresponding to a 71% conversion of functional groups and consistent with the Flory-Stockmeyer equation for a stoichiometric mixture of A3 and B2 monomers.