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LIMONENE AND LINSEED OIL AS A VERSATILE SOURCE FOR EPOXY SYSTEMS

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Abstract – Epoxy resins are a versatile class of thermosetting polymers widely used in applications such as adhesives, coatings, composites, and electronics. Among them, diglycidyl ethers of bisphenol A (DGEBA), derived from petrochemical sources, are the most commonly used. However, the growing emphasis on sustainable development and the expansion of the bioeconomy have driven increasing interest in bio-based epoxy resins derived from renewable resources such as vegetable oils, lignin, and sugars. This work reported the preparation and characterization of bio-based epoxy resins derived from limonene, a low-cost and readily available terpene, and their blends with epoxidized linseed oil (ELO). In both systems, methyl tetrahydrophthalic anhydride (MTHPA) was used as the hardener. The bioepoxy resins—limonene dioxide (LDO) and ELO—were synthesized using a potassium monopersulfate-based oxidant ($KHSO_5 \cdot 0.5KHSO_4 \cdot 0.5K_2SO_4$), acetone, sodium bicarbonate, and water with high yields, and were characterized by ^{1}H and ^{13}C NMR spectroscopy. The curing reactions were investigated by differential scanning calorimetry (DSC) and rheometry, using varying proportions of the bio-based resins (ranging from 0% to 100%). Mechanical properties were evaluated using dynamic mechanical analysis (DMA) and stress-strain testing. For the system composed entirely of LDO, DSC results indicated a two-stage curing process. The first stage, occurring at lower temperatures, is attributed to the ring-opening of terminal epoxide groups, resulting in the formation of a linear polymer. The second stage, observed at temperatures above 100 °C, involves the opening of internal epoxide rings, which are significantly less reactive due to steric hindrance. A fully cured bio-based epoxy resin was obtained with a glass transition temperature (Tg) of 104 °C, a shear modulus higher than 1 GPa, and a Young's modulus of 3.4 GPa. From multifrequency DMA data and theoretical calculations, the crosslink density was estimated at s 1.7×10^{-7} mol/cm^3, consistent with the low tenacity values observed in mechanical testing. Studies on LDO-ELO blends (ranging from 0% to 100%) demonstrated that the mechanical, thermal, and rheological properties of the resulting bio-based epoxy resins can be modulated. This tunability broadens their potential applications across various fields.

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