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BIO-BASED EPOXY CAST RESINS FROM SOYBEAN OIL AND ORANGE PEEL DERIVATIVES

Aurora Garcia-Comer* and Maha L. Shrestha[†]

Department of Chemistry & Physics, West Texas A&M University, Canyon, TX 79016

Abstract

The current work utilizes environmental-friendly, and renewable plant based starting materials in preparation of solid cast resins. Epoxy or oxirane functional groups of soybean oil and orange-peel derived materials were copolymerized in the presence of a Lewis acidic catalyst, tris(pentaflorophenyl)borane, BCF to obtain highly crosslinked solid cast resins. Standard analytical evaluation revealed that the resulting materials are soft and rubbery in nature with high thermal resistivity. Potential applications of these epoxy cast resins include coatings, adhesives, electrical insulation, 3D printing, wearable electronics, automobiles parts, etc.

[†]Corresponding author: mshrestha@wtamu.edu *Undergraduate researcher and co-author

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Introduction

The conventional approach of preparing epoxy cast resins involves the reaction of terminal epoxy or oxirane functional group/s of petroleum-based raw materials such as bisphenol A (BPA) derivatives with amines.[1-3] However, due to severe health and environmental concerns posed by the use of petro-based materials, the scientific community has shifted the technology and focused more on developing such products from bio-based resources.[4-9] Unlike petro-based materials, agriculturally-based raw materials contain internal epoxy functional group/s which are less reactive compared to the terminal epoxy groups.[10, 11] Hence, anhydrides are commonly used to formulate epoxy cast resins while utilizing bio-based resources.[12, 13] However, the reaction with anhydrides requires very high temperatures and longer reaction times. Further, the use of anhydrides reduces the bio-based content of the resulting product. To alleviate these issues various acidic catalysts ranging from super acids to Lewis acids have been employed.[14] The reactions with superacids such as fluoroboric acid is vigorous resulting exothermic reactions, that makes the procedure unfavorable.[15] Alternatively, Lewis acids provides slower and safer conditions, therefore, gaining more popularity in obtaining cross linked cast resins from polymerization reaction. More recently Petrović et al developed a promising method for producing solid cast resins directly from natural oils such as soybean oil, linseed oil and triolien.[16, 17] However, utilization of cyclohexene oxide in obtaining copolymers also decreases the bio-based content.[18] We hereby, synthesized fully bio-based polymer network by reacting epoxidized soybean oil (ESBO) with limonene oxide (LMO, orange peel derivatives) in presence of Lewis acidic catalyst, tris(pentafluorophenyl)borane, BCF. We also focused on optimizing the reaction conditions by varying the catalyst concentration and ratios of the co-monomers. Thermal and mechanical properties of thus prepared epoxy cast resins were also studied.

Experimental Methods

Materials

Epoxidized soybean oil, Drapex 6.8 was kindly supplied by Galata Chemicals. Limonene oxide, ≥ 97.0 % was purchased from Sigma Aldrich. Toluene, anhydrous 99.8 % and tris(pentaflorophenylborane), ≥97.0 % were obtained from Fisher Scientific.

Synthesis

A Lewis acidic catalyst solution was prepared by mixing 1 mL of toluene with 0.04 g of tris(pentaflorophenylborane), BCF.[18, 19] At first, 0.04 wt. % (100 µL) of freshly prepared catalytic solution was added to 10 g of epoxidized soybean oil (ESBO) in a 20 mL glass vial. The reaction mixture was stirred vigorously for about 30 seconds and poured in a steel mold. Gelation time was recorded at room temperature followed by post curing at 100 °C in an oven for about 12 hours. Catalyst concentrations, 0.08 wt. % (200 µL), 0.12 wt. % (300 µL) and 0.16 wt. % (400 µL) were also utilized to optimize the reaction conditions.

Following the same procedure, fully bio-based new epoxy cast resins were synthesized from copolymerization of epoxidized soybean oil (ESBO) and limonene oxide (LMO) by reacting with 0.08 wt. % (200 µL) of BCF solution in a glass vial. 5 different mixtures were prepared using various ratios of ESBO and LMO to evaluate the outcome. a) 9 g ESBO and 1 g LMO (90:10), b) 8 g ESBO and 2 g LMO (80:20), c) 7 g ESBO and 3 g LMO (70:30), d) 6 g ESBO and 4 g LMO (60:40), and e) 5 g ESBO and 5 g LMO (50:50).

Analysis

Properties of the resulting materials were analyzed by Thermogravimetric analysis (TGA), Differential Scanning Calorimeter (DSC) and Attenuated Total Reflectance Infrared Spectrum (ATR-FTIR). Glass transition temperatures, T_g were measured using DSC at a heating rate of 10 °C/min from –80 °C to 200 °C with 20 mL/min flow of nitrogen. Likewise, 5 % weight loss of the materials were determined by TGA. Samples were heated at a temperature between 50 °C to 600 °C at a heating rate of 20 °C/min with 20 mL/min flow of nitrogen. Thickness and hardness of the materials were also measured.

Results and Discussion

The synthesis of a new epoxy solid cast resins utilized easily accessible, commercially available bio-based starting materials, i.e. epoxidized soybean oil (ESBO) derived from soybean oil, and limonene oxide (LMO) extracted from orange peel (Figure 1)

The process involved a simple and efficient procedure, i.e. mixing comonomers in presence of a Lewis acidic catalyst solution, tris(pentafluorophenyl)borane, BCF, 0.08 wt. %, prior to pouring in a steel mold at room temperature. The polymerization reaction initiates *via* cationic ring opening of the epoxy monomer which subsequently acts as a nucleophile and reacts to another monomer (Scheme 1).[14, 17, 20] The reaction continues until all the monomers react completely, forming a crosslinked network. The reaction mechanism is however complex, producing a mixture of linear, branched and cyclic polymers.

Progress of the reactions was first monitored by gelation time at room temperature. The epoxy cast resin obtained by using higher amounts of ESBO, i.e. 90 % ESBO and 10 % LMO reacted the fastest, gelation time being 13 minutes. Lowering the amount of ESBO or increasing the concentration of LMO, also increased the gelation time. Soft and transparent solid epoxy cast resins, as shown below, were obtained after curing at 100 °C in an oven (Figure 2).

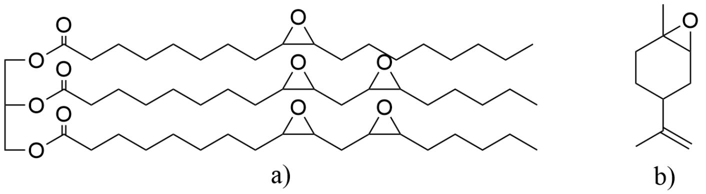
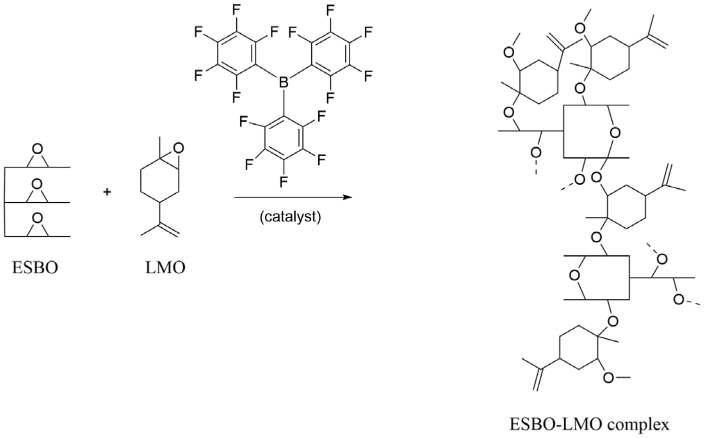


Figure 1. Chemical structures of a) epoxidized soybean oil and b) limonene oxide



Scheme 1. A Schematic diagram of ESBO-LMO complex by copolymerization

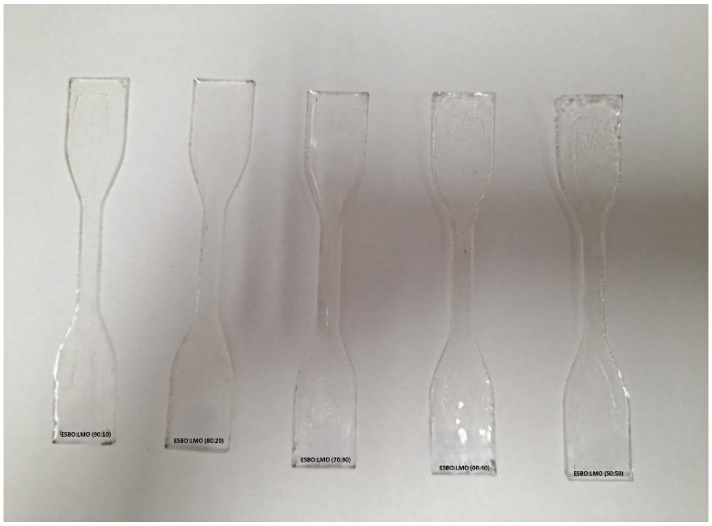


Figure 2. Epoxy cast resins obtained from copolymerization of ESBO and LMO

0.08 wt. % of the catalyst concentration was chosen in obtaining these copolymerized networks from ESBO and LMO, as our catalyst optimization study suggested no significant difference in gelation time while reacting ESBO with various concentrations of the catalyst, ranging from 0.08 wt. % to 0.16 wt. %. The gelation point at room temperature were achieved in about 20 minutes of time with 0.16 wt. % and 0.12 wt. % of the catalyst concentration, whereas gelation occurred within 25 minutes when 0.08 wt. % of the catalyst solution was used with ESBO. However, using 0.04 wt. % of the catalyst concentration with ESBO did not result in crosslinking even after monitoring at room temperature for about 8 hours.

Analysis of mechanical properties of the epoxy cast resins prepared from ESBO and LMO, suggests that the decreasing the concentration of ESBO and increasing the concentration of LMO, reduces the hardness of the resulting materials (Table 1). Our attempts to obtain tensile properties were, however, not successful due to the soft and brittle nature of the materials.

Thermal evaluation of these materials by Thermogravimetric analysis (TGA) suggested that the epoxy cast resin prepared by using higher concentration of ESBO provided the materials with the highest thermal stability (Figure 3). Nevertheless, all these copolymerized materials possess very high thermal resistivity, i.e. above 300 °C. Glass transition temperature, T_g of all these samples measured by Differential Scanning Calorimeter (DSC) were below room temperature which indicates these cast resins are soft and rubbery in nature.

Table 1. Properties of solid epoxy cast resins prepared from ESBO and LMO

Epoxy Cast Resins	Gelation time, mins	Thickness, mm	Hardness, Shore A	Temperature °C, 5 % wt. loss	Temperature °C, T_g
ESBO:LMO (90:10)	13	1.825	84.6±1.9	380	-41.11
ESBO:LMO (80:20)	16	1.814	86.6±2.3	374	-44.72
ESBO:LMO (70:30)	29	1.714	77.6±2.3	355	-44.9
ESBO:LMO (60:40)	35	1.914	75.6±2.7	324	-40.33
ESBO:LMO (50:50)	35	1.770	69.6±2.0	301	-39.30

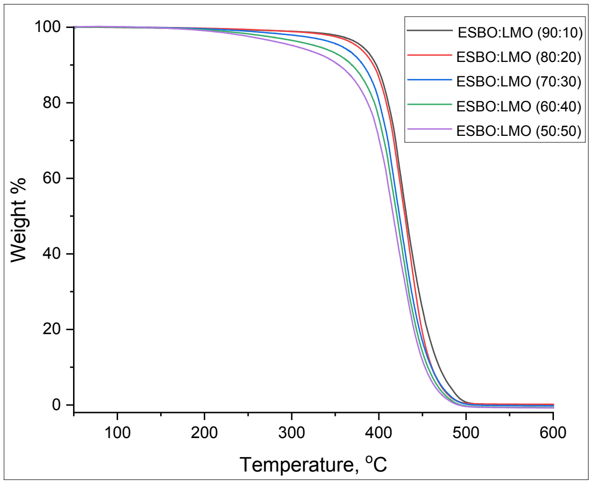


Figure 3. TGA overlay of the epoxy cast resins prepared from copolymerization of ESBO and LMO

FTIR spectrum of ESBO shows the presence of peaks at around 2922 cm^{-1}, 1740 cm^{-1} and 825 cm^{-1} corresponding to -CH stretch, -C=O and -C-O-C epoxy functional group respectively (Figure 4). Similarly, FTIR of LMO contains peaks at 2930 cm^{-1} for -CH stretch (1433 cm^{-1} for -CH_3) and 3082 cm^{-1}, 1644 cm^{-1} for -C=CH. The spectrum of LMO also shows the presence of characteristic epoxy peaks at 830 cm^{-1} and 882 cm^{-1}. The -C=O of ESBO at 1740 cm^{-1} is still present in all the cast resins obtained after the reaction. However, epoxy functional peaks of ESBO and LMO were not observed which suggests both starting materials reacted completely to produce crosslinked copolymers. Furthermore, formation of a new peak at around 1078 cm^{-1} suggests a linear -C-O-C resulting from the ring opening reaction.

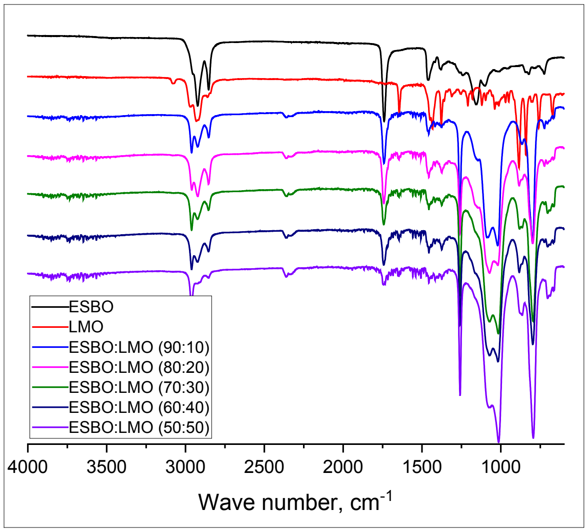


Figure 4. FTIR overlay of the epoxy cast resins prepared from copolymerization of ESBO and LMO

Hence, we obtained several batches of epoxy cast resins with different thermo-mechanical properties. Additionally, we optimized the reaction condition by varying the concentration of the catalyst and also the comonomers.

Conclusion

The significance of our study was that we successfully prepared new epoxy cast resins from 100 % bio-based starting materials, i.e. raw materials derived from soybean oil and orange peels. Since many of the epoxy cast resins available in the market are either partially or fully petroleum based, our work contributed to reducing carbon footprint, utilizing renewable, plant-based resources. Properties of all the epoxy cast resins synthesized were also found to be satisfactory. These materials could be used in various commercial applications such as electrical insulation, automobile industries, wearable electronics and 3 D printing.

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