Characterization and Modification of Asphalt With Epoxy Resins Synthesized From Pyrolysis Oil, a Derivative of Lignocellulosic Biomass

Kyle Thompson¹, Junna Xin², Jinwen Zhang²

1. Biomedical, Chemical, and Materials Engineering, Charles W. Davidson College of Engineering, San Jose State University, San Jose, California
2. Composite Materials and Engineering Center, Washington State University, Pullman, Washington

Introduction

Asphalt is an important commodity that has a vital purpose in civic infrastructure, both in urban and rural communities alike. Polymer-modified asphalt has become a popular topic of interest due to the advantageous property modifications provided by its polymer components. Such alterations include increased moisture resistance, higher tensility, better resistance to long-lasting deformation, greater elasticity, and lower temperature susceptibility. In this study, asphalt was modified with an epoxy resin synthesized from pyrolysis oil (a derivative of lignocellulosic biomass). Typically, in commercial and industrial settings, epoxy resins are formed by treating bisphenol-A with chloromethylxiran (epichlorohydrin), Lignocellulosic biomass, namely lignin, houses similar hydroxyl environments like those found in bisphenol-A, and thus can be used as a substitute.

Experimental Methods

Three separate samples were obtained from extraction and separation processes performed on the starting pyrolysis oil solution. The phenolic and hydroxyxylethyl functional groups belonging to the pyrolysis oil fractions in these samples were reacted with epichlorohydrin as follows: 350 mg of sample, 10 mL of epichlorohydrin, and 10 mg of benzyltrimethylammonium chloride were combined and set to reflux for 4 hours at 117°C. The mixture was then cooled to 60°C, 500 mg of sodium hydroxide was added, and stirring occurred for 3 hours. The remaining epichlorohydrin was evaporated, leaving the desired epoxy monomer. The epoxy monomer was then mixed with the Diels-Alder adduct produced from the reaction between dipentene and maleic anhydride (DMPA) using 1 wt% 2-ethyl-4-methylimidazole as a catalyst. The mixture was then combined with neat asphalt at different concentrations: 7.5 wt%, 15 wt%, and 22.5 wt%, respectively. The epoxy-modified asphalt was then cured at 150°C for 2 hours and then 200°C for 1 hour. Note: The glycidylization reaction was performed on all of the samples in the same numerical ratios as described.

Analytical Methods and Results

**Gas Chromatography & Mass Spectroscopy**

A ThermoScientific Focus GC was used in concordance with a ThermoScientific ITQ 1100 to identify the highest probable fractionations in the pyrolysis oil samples.

**Differential Scanning Calorimetry**

A non-isothermal TA Instruments 2920 Modulated DSC was used under a nitrogen atmosphere to test if the epoxy monomers could be successfully cured.

**Thermal Gravimetric Analysis**

A TA Instruments SDT Q600 was used to evaluate the thermal stability and degradation behavior of the pyrolysis oil samples.

**Fourier Transform Infrared Spectroscopy**

A ThermoScientific Nicolet Nexus 670 FT-IR was employed to test samples before and after epoxidation in order to determine if the glycidylization reaction was successful.

**Rheology**

Figure 10. DSC curves for Sample 2 at 5 different heating rates.

A TA Instruments Discovery HR-2 Hybrid Rheometer was used to test the viscoelastic properties of the epoxy-modified asphalt.

Figure 11-16. Rheology test results for both angular frequency and temperature.

Conclusions

According to the rheology test results, the epoxy-modified asphalt exhibits greater viscoelastic properties, specifically asphalt containing 22.5 wt.% of the epoxy/DMPA mixture.

Acknowledgements

I would like to thank:
- Ran Li and Xiaoxu Teng for taking the time to help me get acquainted with the lab equipment and facilities
- Maika Bui for her assistance in the lab
- Michelle White for coming to the lab to take photographs of the equipment

References