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Synthesis of Bio-Based Polymer Products

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Summary Abstract of the Project

Cellulose is an abundant, natural material used for multiple purposes. The material is used as a reinforcer in the formulation of polymer composites. In a typical thermoset composite formulation, at least three important components are typically present: a polymer matrix, a stiff reinforcer, and crosslinking agent. The polymer matrix, also referred to as the continuous phase, serves as the material in which the other composite components are dispersed. The reinforcer, one of the dispersed components, is distributed throughout the polymer matrix and serves to strengthen the composite by increasing its modulus. The crosslinking agent serves to chemically link separate chains of the polymer matrix, further increasing the modulus of the composite.

In recent years, cellulose has gained a great amount of attention in the scientific community. The excitement surrounding the material centers around its many useful properties, including a variety of methods possible for surface modification, highly advantageous mechanical properties, and the many uses for which it can be employed, notably in polymer composites.[1] However, in order for the material to be used as an effective component in polymer composites, its hydrophilicity must be diminished in order for sufficient dispersion in the matrix to occur. Efforts have been made to address this issue of decreasing hydrophilicity be means of different surface modification techniques.[2] With the possibility of having cellulose as the dispersed phase of a polymer composite, it is very encouraging to see techniques investigated for the further possibility of cellulose surface modification with functional groups capable of crosslinking with the polymer matrix.[3]

In the first aspect of this study, cellulose underwent surface modification in order to be used as a reinforcer in a polymer composite together with vegetable oil. The specific modifications made will allow the compounds to be used in combination as both a reinforcer as well as a crosslinker. The resulting materials were analyzed using ^{1}H NMR and FT-IR spectroscopy as well as DSC calorimetry. FT-IR spectra confirmed the surface modification of cellulose with the materials used. The synthesis and analysis of this compound was one of the subjects of this project.

In addition to the role of cellulose in the development of bio-based polymers, the use of vegetable oils in this area of polymer science has a decades-old history. The fact that vegetable oils are widely available, environmentally friendly, and cost-effective has made this area of research of particular interest in recent years.[4] Vegetable oils have already been shown to be most useful in the preparation of a wide variety of polymer materials or precursors, including alkyds, polyamides, vinyl polymers, and epoxides, among others.[5] The usefulness of vegetable oils in this regard is based on the multiple functional groups which the molecules possess, offering a variety of possibilities for chemical modification. One area of research gaining increasing attention involving vegetable oils is the production of coatings.[6] This project focused on this area of interest.

The second aspect of the research investigates the synthesis of materials for use in a vegetable oil-based UV-curable coating. Vegetable oil was functionalized with a compound synthesized in the lab, and this functionalized oil was used to conduct curing tests with various comonomers and photoinitiators. Curing tests under UV light, together with subsequent analysis, confirmed that the copolymerization of the functionalized oil and comonomers had occurred. The products were analyzed using ^{1}H NMR and FT-IR spectroscopy. ^{1}H NMR and FT-IR analysis confirmed the functionalization of the vegetable oil with the synthesized compound. Physical observation as well as FT-IR data suggests that the copolymerization of the functionalized oil with the comonomers occurred; however, further analysis is needed to confirm this.

Specific information involving particular materials, methods, and conclusions contained in the full report cannot be published at this time, as it is part of ongoing unpublished research.

Summary Conclusions

The work demonstrated a best method of surface modification to produce a useful cellulose reinforcer. It furthermore demonstrated the successful synthesis of functionalized vegetable oil for use in a coating. Ample evidence from NMR and FT-IR analysis lends credence to the fact that the material was successfully synthesized. The curing tests conducted under UV light, together with FT-IR analysis, demonstrated that the material was successfully cured. This is a promising sign for furthering the research.

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Appendix I: Safety Considerations

I. General Considerations

1.) During all in-lab work, a minimum of safety glasses, nitrile gloves, and a lab coat was worn at all times. These items were considered general lab personal protective equipment (PPE).

2.) General carefulness should be used when handling glassware and simple instruments such as stir plates or heating instruments to avoid breakage or burns.

II. Specialty Instrument Considerations

1.) **Fume Hood.** The sash of the fume hood must only be opened as far as necessary in order to keep harmful fumes within the hood, and to protect those around the hood from possible splashes, explosions, etc.

2.) **Cold Materials.** These materials, such as dry ice or liquid nitrogen, must be used in proper vessels, such as insulated containers like dewars, and caution must be exercised when using them to avoid contact with skin.

3.) **Column Chromatography.** When packing a column, containers holding silica gel must only opened under a fume hood in to avoid inhaling the silica particles.

4.) **NMR Spectrometer.** The user must remove metal objects, especially electronics and payment cards, in order to avoid interaction with the magnetic field of the instrument. The user must also be careful in loading and unloading the sample to avoid breakage or sticking of the sample tube.

5.) **FT-IR Spectrometer.** General lab PPE must be used while using this instrument, and proper cleaning of the instrument must be completed after use, typically using ethanol and a Kimwipe.

6.) **UV Lamp.** UV protective glasses must be worn while using this instrument in order to avoid eye damage.

7.) **DSC Calorimeter.** General lab PPE must be worn while using this instrument.