

Standard Operating Procedure (SOP): Synthesis of Quadruple-Function Modified Hemp Lignin (QF-MHL)

1.0 Objective

To chemically modify unmodified hemp lignin to perform four functions: cross-linking with the Epoxidized Hemp Seed Oil (EHSO) matrix, acting as an interfacial compatibilizer, and serving as both the aldehyde source and the amine co-reactant for catalyst-free vitrimer chemistry.

2.0 Required Materials

- Isolated hemp hurd lignin.
- Maleic anhydride.
- Acetone (reagent grade).
- Formaldehyde solution (37%).
- Diethylamine (reagent grade).
- High-shear mixer, temperature-controlled reflux apparatus, vacuum filter.

3.0 Tentative Procedure

3.1 Maleinization Step

1. Add 10 g of isolated hemp lignin to a reflux apparatus with a 10% solution of maleic anhydride in 200 mL of acetone.
2. Heat the mixture to reflux at approximately $60 \pm 2^\circ\text{C}$ for 7 hours under magnetic stirring. This step grafts maleic anhydride groups onto the lignin to act as a compatibilizer.

3.2 Purification

1. After reflux, remove excess acetone via rotary evaporation.
2. Vacuum filter the product and wash it multiple times with distilled water to remove impurities and unreacted maleic anhydride.

3.3 Amination Step (Mannich Reaction)

1. Disperse the purified, maleated lignin in a suitable solvent.
2. Add an excess of formaldehyde and diethylamine. The molar ratio of reagents must be experimentally optimized, but a starting point could be a 1:1:1 molar ratio of lignin to formaldehyde to diethylamine.
3. Heat the mixture to $70\text{--}80^\circ\text{C}$ for several hours to promote the Mannich reaction.

3.4 Final Purification

1. The resulting QF-MHL must be filtered and thoroughly washed to remove unreacted reagents before use in the final composite.

4.0 Validation

- **FTIR Spectroscopy:** Use FTIR to confirm the presence of new functional groups from both maleinization (C=O stretch) and amination (N-H stretch).
- **NMR Spectroscopy:** Use ^1H and ^{13}C NMR to quantify the degree of modification and confirm that the original aldehyde and hydroxyl groups, necessary for the other functions, are still present.
- **Titration:** Perform acid-base titration to quantify the number of accessible amine groups.

Additional Readings & References

For further details and the theoretical framework behind this process, you can refer to the following documents found in your Google Drive:

- "A Proposal for the Experimental Validation of a 6-Component Hempoxies Platform: A Streamlined, Catalyst-Free Vitrimer Bionanocomposite System"

- "Hempoxies - A Bionanocomposite Material Class Made with Seven Hemp Components...".