Advanced Materials from Industrial Hemp Using Deep Eutectic Solvent for Clean Water Applications

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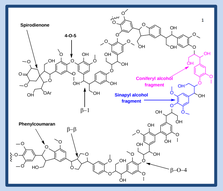
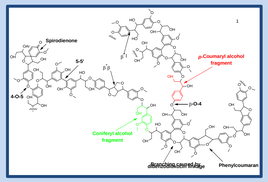
Introduction

Lignocellulosic biomass, the second largest biomass component on Earth, is composed of cellulose, hemicellulose, and lignin. Effective pretreatment technologies are essential to deconstruct the rigid plant cell wall structure to remove lignin.

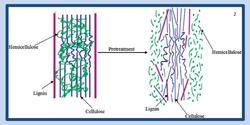


Hardwood Lignin



Softwood Lignin

Industrial hemp is known for its application in fiber, oil, and nutraceutical products, however utilizing industrial hemp as advanced material for clean water applications provides an additional avenue to utilize hemp's value. Recent advances in the application of deep eutectic solvents (DES) for biomass deconstruction and subsequent lignin extraction have brought new pathways to the biomass pretreatment process.



DES is intrinsically cheaper than many ionic liquids (ILs) due to low precursor cost, simple synthesis and improved recyclability. Nevertheless, DES can be as effective as IL towards dissolving lignin from plant materials.

Objectives

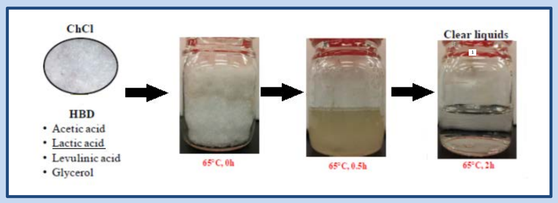
1) Extract and characterize lignin streams from DES (1:2 choline chloride: lactic acid) treated industrial hemp for applications in lignin-based carbon materials for wastewater treatment;

2) Synthesize hemp fiber supported catalysts for the accelerated removal of phenols from contaminated water.

DES Synthesis and Lignin Extraction

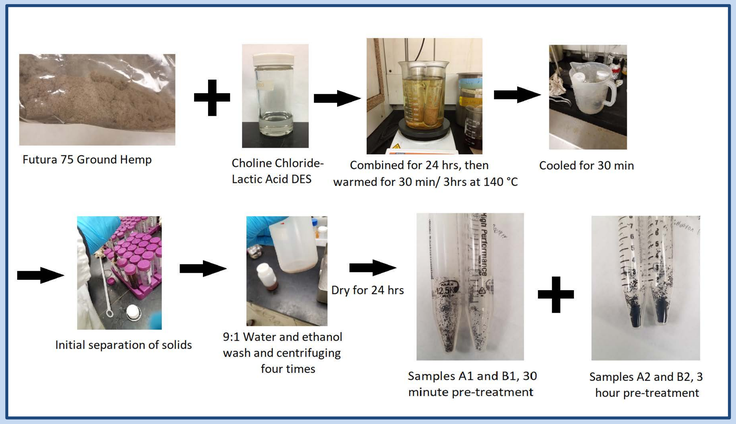
Materials: Biomass- Futura 75 Industrial Hemp, Choline-Chloride; Lactic Acid, Ethanol, and DI Water; hemp fiber from Sunstrand, LLC.

Equipment: Graduated Cylinder, Pipette, Balance, 50 mL Plastic Test Tubes, 200 mL Glass Test Tube, 500 mL Capped Bottles, Ice Bath, Spatula, Centrifuge, Hotplate, Stir Bars, Vortex, and Oil Bath.



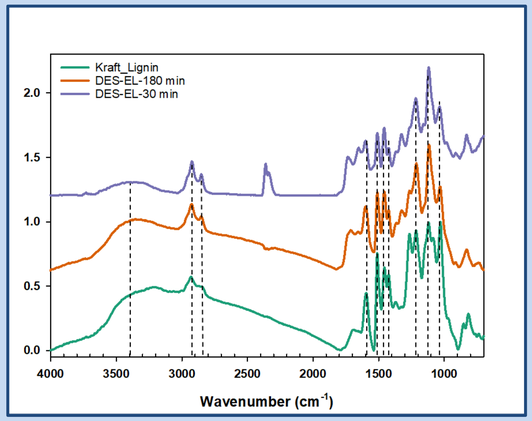
Choline Chloride and Lactic Acid were combined in a 1:2 molar ratio to produce 300 g of DES for use in lignin separation.

Lignin Extraction Procedure



Lignin Extraction Results

FT-IR Lignin Characterization



- Key lignin identifying peaks occur at the following wavelengths: 3400 cm^{-1}, 2930 cm^{-1}, 2840 cm^{-1}, 1595 cm^{-1}, 1510 cm^{-1} 1460 cm^{-1}, 1420 cm^{-1}, 1220 cm^{-1}, 1110 cm^{-1}, 1030 cm^{-1}.
- Additional peaks and variations are typically indicative of impurities in the lignin sample. The most notable of these occur within 2300-2400 and 1600-1700 cm^{-1}. Results suggest that 180 minute DES extracted lignin sample (DES-EL) had higher purity than the 30 minute DES-EL sample.

Lignin Yield

The total mass of lignin retrieved from 30-minute trials from the original 3 g of ground hemp averaged .0165 g of hemp. The 3-hour trials averaged .0545 g of lignin from the original 3 g of hemp. Given that hemp is composed of approximately 18% lignin, there was .5 g of lignin in total which is much larger that the recorded sample values. Longer reaction times improved the yield, however the values are still an order of magnitude lower than theoretical probably due to loss during sample transferring.

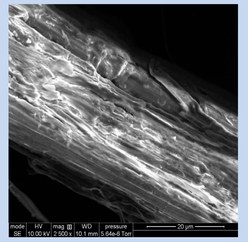
Hemp Fiber Impregnated Catalyst Synthesis

Materials: Biomass- Futura 75 Industrial Hemp, KOH, DI Water, and Copper Chloride

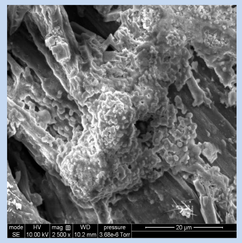
Equipment: Graduated Cylinder, Balance, Spatula, 250 ml Beaker, 400 m Beaker, and Thomas Wiley Laboratory Mill (1 mm grains)

Process: Full Sunstrand hemp fibers were submerged in KOH solution for 24 hrs. A second sample of biomass was ground to 1 mm grains, treated with copper chloride solution, and allowed to rest for 24 hours. Both samples were then dried and scanning electron micrographs were obtained for surface analysis.

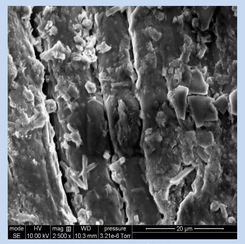
Analysis of Hemp Fiber Catalyst Synthesis



Raw Hemp Strand



CuCl-Coated Hemp



KOH Coated Hemp

These scanning electron microscopy (SEM) images demonstrate the ability of hemp fiber absorbing metal catalysts. The copper chloride had a greater affinity for coating the hemp fiber surface. The coating was more complete, increasing the catalytic surface area desired for water treatment applications.

Conclusions

Improvements to the experimental methodology and continued refinement of DES pretreatment methods will greatly improve both yield and purity of retrieved lignin to fully utilize this emerging technology. In regard to the second objective, both catalysts successfully impregnated in hemp fiber. CuCl appeared superior to KOH in coating the hemp fiber as shown in the SEM images. This implies that CuCl catalyst would be superior for phenol removal as opposed to KOH when using hemp fiber as a supporting structure.

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