



STEF GREEN

**MASTERS FINAL
EXAM DEFENSE
CHEMICAL ENGINEERING**

Selective Production of Phenolic Aldehydes with Acetosolv Lignin Extracted from Corn Field Leftovers

Abstract

Annual U.S. production of lignocellulose from crop residues such as corn cobs and stover is nearly 400 million tons. Yet, it is a vastly underutilized resource. Global sustainability and biorefinery profitability will benefit from innovative technologies which valorize all lignocellulosic fractions to produce diverse chemical products alongside fuels. To this end, a valorization technology where phenolic aldehydes are selectively produced from lignin extracted from corn residues was devised. Lignin was extracted from lignocellulose in aqueous acetic acid and a sulfuric acid catalyst. Under agitated reflux conditions at 110 °C, corn residues were mildly hydrolyzed to cleave lignin-carbohydrate complexes (LCCs) and other inter-unit linkages. After separation from the cellulosic pulp and hemicellulose, the estimated yield of lignin is 14-17% by dry mass of corn residue. As revealed by 2D-NMR (¹H-¹³C HSQC), lignin from corn cobs (CL) and corn stover (SL) are identical in the aromatic region and rich in phenyl-alkyl linkages. To produce phenolic aldehydes, the isolated lignins (SL and CL) were dissolved in a suitable protic solvent such as acetic acid then sprayed into a continuous phase of gaseous ozone at ambient temperature and pressure. The spray aerosolizes into fine droplets thus maximizing the gas-liquid interfacial area. This enhanced mass transfer area enables ozone to easily penetrate the droplets to preferentially cleave lignin's pendant C=C bonds according to the Criegee mechanism. Such spray ozonolysis occurs rapidly with a short residence of 5-8 s. Analysis with Gas Chromatography/Flame Ionization Detector (GC-FID) confirms the production of phenolic aldehydes, vanillin and *p*-hydroxybenzaldehyde (*p*HB). The cumulative yield of these products is ca. 10 wt.% of the initial lignin mass. Gel Permeation Chromatography (GPC) and HSQC of the remaining ozonized lignin reveals a largely intact macrostructure, suggesting that the ozonized lignin may be further valorized. Demonstration of these scalable concepts for lignin isolation and ozonolysis paves the way for further development towards potential commercialization with several value-added product streams.

Friday,
May 13,
2022

Starts @
10:30AM

CEBC,
Bldg. A
Conference
Room

Zoom Meeting
Details:
[MEETING
HYPERLINK](#)

Meeting ID:
929 7032 9494

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2022

Research Advisor
**Distinguished
Professor Bala
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