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SUMMARY REPORT

Objective

Collaboration between Illium Technologies LLC and the Advanced Process Demonstration Unit (ABPDU) at Lawrence Berkeley National Laboratory (LBNL) was developed to investigate oxidative lignin depolymerization and synthesis of lignin-derived ionic liquids for biomass processing.

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Devlierables

1. Procurement of lignin

The ArbomassTM lignin (prepared using dilute acid, steam pretreatment of hardwood, mostly Birch and Beech trees) was procured from collaborators at Cedar Creek P&T.

2. Lignin extraction, solids separation and depolymerization

Lignin was isolated from pulverized ArbomassTM using alkali extraction followed by solid liquid separation and precipitation by adjusting the pH. The extraction process was scaled up to kg scale in ABPDU with similar lignin yield (37%). A series of small scale experiments were conducted to optimize the vanillin and syringaldehyde yields from the extracted lignin. Optimal conditions were obtained by reacting for 60 min at 170-190oC under air at 120 psi with 800 RPM stirring. The lignin depolymerization process was scaled up (150x) using the ABPDU's 10 L Parr vessel and different solid loadings were attempted. It was found that higher solids loading decreases the overall aldehyde yields. The larger sample amounts allowed for the comparative studies to be performed between extracting solvents of different polarities. The more polar solvent ethyl acteate (EtOAc) showed greater extracting power. Optimal pH for extraction yield was observed as pH 7 > pH 5 > pH 9.

3. Analysis of lignin depolymerization reaction products and preparation of ILs

Using benzyltrimethylammonium as a model cation for lignin-derived ILs, a series of compounds were prepared for biomass pretreatment studies. Both aqueous IL solutions (10%, w/w) and the neat (pure) ILs were tested for pretreatment efficiency.

4. Small-scale pretreatment performance validation using ILs

The most promising IL was identified to be benzyltrimethylammonium thiosalicylate (TMBnA TSL), which showed up to 85% glucose yield with 10% aqueous IL as the pretreatment solvent. The results suggest that benzyltrimethylammonium ILs derived from lignin are potentially more sustainable for biomass pretreatment than petroleum-based ILs.

Summary

The polymers found in plant cell walls represent the largest reserves of renewable carbon on Earth - it has been estimated that the United States has approximately 1.3 billion tons of plant biomass available per year. Ionic liquids (ILs) are salts with melting points less than 100oC, typically containing organic cations, that possess a wide range of extraordinary properties as compared to traditional organic solvents. The unusual catalytic and solvation properties of ionic liquids have attracted the biofuel and biomass communities because IL-pretreatment provides higher fermentable sugar yields than other methods such as dilute acid or ammonia fiber expansion (AFEX) under similar severities. This research used the by-product (lignin) from lignocellulosic biofuel production process as the starting material for ILs production to lower down the cost of the ILs and enable a closed-loop biorefinery process. The lignin is depolymerized, extracted, and used to prepare the renewable ILs. The application of the ILs is to pretreat biomass or serve growing IL markets such as those in process chemistry, biotechnology and materials recycling. We have shown a comparable sugar yields with the published report by using both neat and aqueous lignin-derived ILs.