



## Blending municipal solid waste with corn stover for sugar production using ionic liquid process



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### HIGHLIGHTS

- Blending in municipal solid waste (MSW) decreases the feedstock cost.
- MSW and its blends can be efficiently pretreated in certain ionic liquids.
- Blending corn stover with MSW helps to decrease the viscosity.

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### ABSTRACT

Municipal solid waste (MSW) represents an attractive cellulosic resource for sustainable fuel production. However, its heterogeneity is the major barrier to efficient conversion to biofuels. MSW paper mix was generated and blended with corn stover (CS). It has been shown that both of them can be efficiently pretreated in certain ionic liquids (ILs) with high yields of fermentable sugars. After pretreatment in 1-ethyl-3-methylimidazolium acetate ([C<sub>2</sub>C<sub>1</sub>Im][OAc]), over 80% glucose has been released with enzymatic saccharification. We have also applied an enzyme-free process by adding mineral acid and water directly into the IL/biomass slurry to induce hydrolysis. With the acidolysis process in 1-ethyl-3-methylimidazolium chloride ([C<sub>2</sub>C<sub>1</sub>Im]Cl), up to 80% glucose and 90% xylose are released. There is a correlation between the viscosity profile and hydrolysis efficiency; low viscosity of the hydrolysate generally corresponds to high sugar yields. Overall, the results indicate the feasibility of incorporating MSW as a robust blending agent for biorefineries.

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### 1. Introduction

Renewable energy technologies are being developed as new sources of fuels and power to meet our current and future energy needs. Lignocellulosic biomass is an important renewable source for production of biofuels and bio-products. Significant attention has been historically given to agriculturally-derived feedstocks; however a diverse range of wastes, including municipal solid wastes (MSW) also have potential to serve as feedstocks for the production of advanced biofuels due to its abundance and low cost (The Biomass Research and Development Board Report, 2008; Williams, 2007). Compared with the seasonal availability of

agricultural wastes, MSW has the advantage of year-round availability, an established collection infrastructure and potential availability at negative cost (Williams, 2007). An efficient use of MSW would not only benefit biofuel industry but also reduce landfill disposal (Williams, 2007). Recent reports projected that an estimated 44.5 million dry tons of MSW will be available in 2022 in the United States, among which paper mix is one of the major components, representing about 30% of total MSW (Environmental Protection Agency Report, 2010). Biomass feedstock costs remain a large contributor to biofuel production costs (Klein-Marcuschamer et al., 2010). The costs could be reduced by blending more expensive high quality feedstocks with lower cost, lower quality feedstocks such that the overall quality still meets specifications required by the biorefinery and the final costs are reduced (Thompson et al., 2014).

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Among the various options of biomass pretreatment strategies, ionic liquid (IL) pretreatment with imidazolium-based ILs has been proven to be one of the most effective ways for biomass processing, primarily due to the efficient solubilization and perturbation of the major components of the plant cell wall, which makes the biomass structure amenable for downstream processing (Li et al., 2010; Sun et al., 2013; Xu et al., 2012). The conversion to sugars can be realized biologically by using commercial enzyme mixtures, or chemically by using mineral acid as a catalyst (Binder and Raines, 2010; Li et al., 2010). Enzymatic hydrolysis is frequently used for polysaccharide hydrolysis to monosaccharides after biomass pretreatment. However, the saccharification process takes as long as 2–3 days, and enzyme cost is the second highest contributor to material costs of the biofuel conversion process after those associated with the feedstock input itself (Klein-Marcuschamer et al., 2010). Acidolysis in certain ILs has been reported as an enzyme-free process for biomass conversion (Binder and Raines, 2010). By using a direct injection of acid and water after IL pretreatment, both pentose and hexose are released from polysaccharides within 2–3 h. The significant reduction of processing time would be a great benefit for biorefineries due to the increased productivity and significant cost reduction. In addition, there is no need for ionic liquid separation or solid–liquid separation before acidolysis. Our previous study showed up to 83% of glucose and 99% of xylose liberation from switchgrass with the imidazolium chloride IL pretreatment followed by acidolysis (Sun et al., 2013). To date, there is no known published report on evaluating the performance of IL pretreatment for the processing of MSW and MSW blends. In this study, both enzymatic hydrolysis and dilute acid hydrolysis were evaluated in terms of sugar production from feedstock blends.

## 2. Methods

### 2.1. Raw materials

The paper waste materials, consisting of 15% glossy paper, 25% non-glossy paper, 31% non-glossy cardboard, and 28% glossy cardboard, were collected over the course of 2 weeks from one of the Idaho National Laboratory (INL) buildings and utilized to represent the MSW material in this study. The MSW paper material was shredded through a conventional office shredder and the cardboard material was cut into pieces with scissors. Each paper type was ground to 2 mm using a Thomas Scientific Model 4 Laboratory Wiley Mill (Thomas Scientific, Swedesboro, NJ). The corn stover was grown near Emmitsburgh (IA, USA) and was harvested in September 2010. Harvested corn stover was ground using a Vermeer BG480 grinder (Vermeer, IA, USA) designed for processing up to 4 × 4 ft bales. A 1-inch screen was used for these grinds. The MSW paper materials were then mixed with previously ground corn stover (CS) in different ratios. The IL [C<sub>2</sub>C<sub>1</sub>Im][OAc] (>95% purity) was purchased from BASF (Ludwigshafen, Germany). [C<sub>2</sub>C<sub>1</sub>Im]Cl (>97% purity) and 6 N hydrochloric acid were purchased from Sigma–Aldrich.

### 2.2. Feedstock cost determination

DOE has set a cost target of \$80/ton for feedstock delivered to the biorefinery. This target was developed to address barriers involved with commercializing logistics systems to be cost competitive with petroleum fuels. INL has developed several feedstock logistics models that calculate the costs associated with harvest and collection, storage, preprocessing, handling and transportation of feedstocks. The Biomass Logistics Model (BLM) simulating the flow of biomass throughout the entire supply chain and accounting

for cost as different unit operations are applied. This model is used to evaluate supply chain designs in order to meet DOE targets. The BLM is an integrated model whose analytic engine is developed in the system dynamic software package Powersim™. Additionally, the Least Cost Formulation Model (LCF) combines grower payment/access cost from the Billion Ton Update (BT2) with logistics costs from the BLM and feedstock quality characteristics from the Biomass Resource Library to estimate the total cost of feedstock to the throat of the biorefinery. The LCF model is developed in a simulation software package AnyLogic™. The concept behind LCF is similar to the animal feed industry where the goal is to provide the least expensive combination of feed constituents (soybean meal, corn meal, etc.) while meeting nutrient requirements for desired animal growth. For example, the University of Georgia Athen's Windows User-Friendly Feed Formulation for Poultry and Swine (WUFFDA) model generates least cost animal feed formulations for desired feed ingredients. Where the feed industry pursues desired yield (animal weight gain) the biofuel industry's targets fuel production, both trying to minimize cost while maintaining performance. The LCF joins output of models and databases (BLM, BT2, Biomass Resource Library) to generate the delivered costs of feedstock formulations to direct research to help meet the \$80/ton target.

### 2.3. Pretreatment in [C<sub>2</sub>C<sub>1</sub>Im][OAc] followed by enzymatic saccharification

#### 2.3.1. Pretreatment

A 10% (w/w) biomass solution was prepared by combining 2 g of MSW or MSW/CS blends with 18 g of [C<sub>2</sub>C<sub>1</sub>Im][OAc] in a 50 mL Globe reactor (Syrris, UK). The reactor was heated to the desired temperature (140 °C, ramp time: 40 min) and stirred at 300 rpm with a Teflon overhead stirrer. Following pretreatment, 60 mL of 95% ethanol was slowly added to the biomass/IL slurry with continued stirring. The mixture was transferred into 50 mL Falcon tubes and centrifuged at high speed (14,000 rpm) to separate the solids. Additional solids were collected from the supernatant by nylon mesh filtration (1 micron pore size), and the combined pretreated biomass was washed two additional times with 60 mL DI water to remove any residual IL. The solids were again filtered through 1 micron nylon mesh and stored at 4 °C for analysis.

#### 2.3.2. Enzymatic saccharification

Enzymatic saccharification of pretreated and untreated biomass was carried out at 50 °C and pH 5.5 at 150 rpm in a rotary incubator (Enviro-Genie, Scientific Industries, Inc.) using commercial enzyme mixtures, Cellic® CTec2 (batch number VCN10001) and HTec2 (batch number VHN00001), obtained as a gift from Novozymes. The protein content of enzymes was determined by bicinchoninic acid (BCA) assay with a Pierce BCA Protein Assay Kit (Thermo Scientific) using BSA as protein standard. CTec2 has a protein content of 186.6 ± 2.0 mg/mL, and protein content of HTec2 is 180.1 ± 1.8 mg/mL protein (Socha et al., 2014). All reactions were conducted at 10% biomass loading by placing 500 mg of biomass (dry weight) in a 25 mL centrifuge tube. The pH of the mixture was adjusted with 50 mM sodium citrate buffer (pH 4.8) supplemented with 0.02% (v/v) Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> to prevent microbial contamination. The total volume of 5 mL included a total protein content of 20 mg protein/g glucan as determined by compositional analysis, with the volumetric ratio of CTec2:HTec2 = 9:1. Reactions were monitored by centrifuging 50 µL aliquots of supernatant (5 min, 10,000×g) in spin-filter centrifugal tubes with 0.45 µm nylon filter at specific time intervals and measuring monomeric sugar concentrations by HPLC.

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