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Research paper

Handling heterogeneous hybrid poplar particle sizes for sugar production

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ABSTRACT

Five chip sizes of freshly harvested hybrid poplar, ranging from 0.2 × 0.2 cm to 2.0 × 1.5 cm, plus an equal mixture of all the particles, were used to determine the influence of initial particle size on sugar recovery after steam explosion pretreatment and enzymatic hydrolysis. It was found that there is essentially no effect of particle size or particle size heterogeneity on sugar recovery. Enzymatic digestibility of solids from all the particles showed similar yields ranging from 78 to 82% glucan conversion after 72 h. The overall sugar recovery from all the samples ranged from 87 to 90% and 61–64% for glucose and xylose respectively and was not influenced by particle size. An unsteady heat transfer model was used to assess the intra-particle temperature profile of the wood during pretreatment. It was found that all the wood chips, regardless of size, are essentially isothermal. This supports the experimental result of no significant difference in sugar yields among different particle sizes. Steam pretreatment appears to be a robust method that can accommodate a wide range of particle sizes.

1. Introduction

The use of lignocellulosic biomass for the production of fuels and chemicals will be imperative if petroleum is to be replaced by renewable and sustainable alternatives. Production of fuels from lignocellulosic materials has attracted worldwide interest because they do not compete with food, and have significantly lower global warming potentials [1]. The United States has diverse and abundant biomass resources that can potentially be used as fuels and chemicals feedstocks. Biomass in its raw, "as-harvested", form may not, however, be a good feedstock for fuels and chemicals production [2]. It is low in energy and bulk density, and has variable physical attributes, all of which can reduce the feedstock's energy value and make supply system logistics complex and expensive [3].

Feedstock cost is a major economic factor in commercial scale biofuels production, contributing 40–50% of the operating costs in a lignocellulosic biomass based biorefinery [4]. The low selling prices of the products generated by a biorefinery do not allow a biomass conversion facility to afford 'pristine' feedstock composed of clean and homogeneous high quality raw material [5]. Low cost feedstocks will have heterogeneous chemical compositions [6] and physical characteristics. This variability results from plant physiology, agronomic and environmental conditions, as well as differences in the way biomass is harvested, stored and processed prior to conversion [7]. Heterogeneous biomass, including characteristics such as particle size, moisture content, bark content, leaf/needle content and general chemical attributes of the biomass, will affect the bioconversion process [8]. Nonetheless, the capability for a biorefinery to handle heterogeneous feedstock – including particle size – is an important criteria for economic viability.

Size reduction has been proposed as a critical operation for preparing physically heterogeneous feedstock for use in a biorefinery [9]. However, size reduction of biomass along with transportation and storage costs can make up between 13 and 28% of total feedstock costs [10]. Thus, to reduce feedstock costs, particle size heterogeneity in the feedstock may need to be tolerated. In order to make the biomass to biofuels and biochemicals process economically feasible and sustainable, a robust bioconversion system should handle different particles sizes without altering the overall sugar yields.

Most bioconversion research has been carried out using high quality, homogeneous biomass. This biomass, such as screened white wood chips or milled agricultural residues, is typically uniformly comminuted. Little attention has been paid to the efficacy of

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converting variable particle sizes biomass into fermentable sugars and fuels. The influence of particle size on bioconversion had been studied by a few researchers. Brownell et al. [11], analyzed the effect of chip size on bioconversion of aspen wood to better understand the influence of the particle size on lignocellulosic bioconversion. They concluded that smaller chip size (3.2 mm fiber direction) resulted in more rapid chip heating and more extensive decomposition during pretreatment, leading to high enzymatic hydrolysis yields. Brownell et al. [11] also concluded that larger aspen wood chips produced more of the fermentation inhibitors 5-hydroxymethylfurfural (HMF) and furfural, due to slow particle heating. Negro et al. [12] steam pretreated two poplar particle sizes, 2–5 mm and 12–15 mm, and found no effect on either enzyme digestibility of pretreated solids or on the downstream ethanol fermentation yield. The effect of using mixed particle sizes on overall sugar production via steam pretreatment and bioconversion has not been investigated.

As mentioned previously, the economic success of a potential biorefinery is directly related to the use of low cost biomass. While a more heterogeneous raw material is less expensive, the efficiency of the whole bioconversion process using mixtures of different particles sizes has not yet been investigated. In this research, the effect of feedstock particle size and particle size heterogeneity was evaluated using a single raw material, and one set of pretreatment conditions. Robust statistical analysis with replicates of all performed experiments was applied to quantitatively assess the impact of particle size on sugar recovery. The objectives of this research were twofold. Firstly, to determine the influence of particle size and particle size heterogeneity on sugar production during bioconversion of hybrid poplar via steam explosion and enzymatic hydrolysis. Secondly, to evaluate the robustness of steam explosion as a pretreatment for simultaneously processing different particle sizes ranging from 0.2 × 0.2 cm to 2.0 × 1.5 cm. A heat transfer analysis was performed to provide a theoretical underpinning for the experimental results.

2. Methods

In this study, five particle sizes ranging from 0.2 × 0.2 cm to 2.0 × 1.5 cm, plus an equal weighted mixture of all particles, were used to determine the influence of particle size on sugar production following steam pretreatment and enzymatic hydrolysis. The steam pretreated particles were then chemically characterized and saccharifies. A complete mass balance of carbohydrates was determined to assess overall sugar recovery. Fig. 1 shows process flow for the bioconversion experiments.

2.1. Material

Freshly harvested 18-year-old hybrid poplar, *Populus deltoides x Populus nigra*, from Puyallup, WA was used for this study. The biomass was comminuted using two different processes to obtain five different size fractions: S1 (length: 0.2× width: 0.2× thickness: 0.1 cm), S2 (0.4 × 0.4 × 0.4 cm) and S3 (1.2 × 0.4 × 0.4 cm) (referred to as Crumbles™) were made using a patented technology developed by Forest Concepts, LLC and previously described by Dooley et al. [13]. In the Forest Concepts process, Crumbles™ particles are produced by feeding wood veneer in a direction normal to the grain through a counter rotating pair of intermeshing cutting discs aligned axially perpendicular to the direction of veneer travel. S4 (0.7 × 1.0 × 0.2 cm) and S5 (2.0 × 1.5 × 0.4 cm) (chips), were made using a slant disc chipper (Acrowood, Everett, WA). In addition, a mixture was prepared of all the particles in equal proportions. The particles had a moisture content of 60–65% and were stored at −20 °C until use.

2.2. Calculation of sugar recovery

Material balances for each particle size were closed for steam pretreatment and enzymatic hydrolysis by measuring the composition and total mass of each liquid and solid stream leaving pretreatment and saccharification and converting this data to amount of sugars recovered. The calculations were based on Wyman et al. [14]. Recoveries were then calculated based on glucose and xylose available in the raw material fed to the systems. Thus, based on hybrid poplar composition and the appropriate increase in mass with hydrolysis, for S1, a maximum of 54 and 15 mass units of glucose and xylose respectively could be produced from 100 mass units of hybrid poplar. After pretreatment and saccharification a monomeric sugar recovery of 87.3 and 62.1% of glucose and xylose were observed for S1. Thus, 47.1 and 9.3 mass units of glucose and xylose were recovered after bioconversion. Identical procedures were completed for the rest of the samples.

2.3. Pretreatment and processing conditions

The bioconversion process flow for this research is shown in Fig. 1. Prior to pretreatment, 800 g of dry biomass of each of the six feedstocks was impregnated with gaseous sulfur dioxide (3% w/w) and sealed in airtight plastic bags. Specifically, 24 g of SO_2 was added to the 800 g of dry biomass from a gas cylinder into a plastic bag containing the biomass. After 12 h, the weight of the biomass was monitored to determine the gas retention for each chip size. Samples were then subdivided into 400 g samples and pretreated using a 2.7 L batch steam gun manufactured by Aurora Technical (Savona, BC, Canada) at 195 °C for 5 min. The pretreatment conditions were chosen for previous work done by Ewanick [8]. At the end of the reaction time, a pneumatic valve was opened between the pressurized reaction tank and the collection tank, causing the explosion of the biomass, which was discharged into the collection tank. The resulting slurry was vacuum filtered to separate the liquid fraction from the pretreated solids. Both fractions were analyzed as described below and used to construct a mass balance of carbohydrates and lignin. Solids were washed with deionized water equal to 20 times the mass of solids prior to analysis and hydrolysis. Each steam explosion was performed 3 times for each different size fraction and the mixture, resulting in a total of 18 substrates, all of which were analyzed as described below.

2.4. Instrumental analysis

2.4.1. High pressure liquid chromatography (HPLC)

Carbohydrates were measured on a Dionex (Sunnyvale, CA) HPLC (ICS-3000) system equipped with an AS autosampler, ED electrochemical detector, dual pumps, and anion exchange column (Dionex, CarboPac PA1). Deionized water at 1 mL/min was used as an eluent, and post-column addition of 0.2 M NaOH at a flow rate of 0.5 mL/min ensured optimization of baseline stability and detector sensitivity. After each analysis, the column was reconditioned with 0.25 M NaOH. Twenty microliters of each sample were injected after filtration through a 0.22 μm syringe filter (Restek Corp., Bellefonte, PA). Samples were measured against standards consisting of arabinose, galactose, glucose, xylose, and mannose. In addition, fucose was used as an internal standard.

Acetic acid, furfural and 5-hydromethylfurfural were measured using refractive index detection on a Shimadzu Prominence LC. Separation of these compounds was achieved by an anion exchange column (REZEX RHM-Mono-saccharide H^+ (8%), Phenomenex, Inc., Torrance, CA) with an isocratic mobile phase that of 5 mM H_2SO_4 at a flow rate of 0.6 mL/min. The column oven temperature was maintained at 63 °C. Twenty microliters of each sample were