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Post-treatment mechanical refining as a method to improve overall sugar recovery of steam pretreated hybrid poplar

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HIGHLIGHTS

- Refining reduces the particle size of steam pretreated biomass.
- Refining greatly improves hydrolyzability for samples pretreated at low severity.
- High sugar yield is achievable with low severity, SO_2-free pretreatment via refining.
- Refining may mitigate process disturbances and secure stable operation of biorefinery.

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ABSTRACT

This study investigates the effect of mechanical refining to improve the sugar yield from biomass processed under a wide range of steam pretreatment conditions. Hybrid poplar chips were steam pretreated using six different conditions with or without SO_2. The resulting water insoluble fractions were subjected to mechanical refining. After refining, poplar pretreated at 205 °C for 10 min without SO_2 obtained a 32% improvement in enzymatic hydrolysis and achieved similar overall monomeric sugar recovery (539 kg/tonne) to samples pretreated with SO_2. Refining did not improve hydrolyzability of samples pretreated at more severe conditions, nor did it improve the overall sugar recovery. By maximizing overall sugar recovery, refining could partially decouple the pretreatment from other unit operations, and enable the use of low temperature, non-sulfur pretreatment conditions. The study demonstrates the possibility of using post-treatment refining to accommodate potential pretreatment process upsets without sacrificing sugar yields.

1. Introduction

Significant progress has been made towards the development of an economically feasible biorefinery. Pioneer commercial biorefineries are now operating all over the world. Most previous research has investigated scenarios where the feedstock to the biorefinery was of consistent quality and the bioconversion processes were optimized for that feedstock. In reality, biorefineries will have to deal with feedstock variability (Kenney et al., 2013) and process upsets that are inevitable in large scale biomass processing facilities – both of which will significantly impact the final product yields.

Biorefineries require a stable, year-round, and large scale supply of feedstock (Humbird et al., 2011; Kenney et al., 2013). To meet these requirements, biorefineries must use feedstock with variable compositions and properties. This can be a challenge, however, as different feedstocks have unique characteristics which affect their utilization. As the initial step of bioconversion process, pretreatment determines the sugar recovery and subsequent fuel/chemical yield of the biorefinery. Steam explosion pretreatment has been adopted by many biorefineries because of its robust nature and ability to process a variety of feedstocks (De Bari et al., 2007). By manipulating temperature, time and pH, steam pretreatment can efficiently fractionate lignocellulosic materials with short reaction time, limited or no use of chemicals, and low energy consumption (Chandra et al., 2007). High severity steam pretreatment generally fractionates lignocellulose more effectively and improves the enzymatic digestibility of pretreated biomass. However, more severe pretreatment conditions result in greater sugar and lignin degradation, forming more furans and phenolic compounds which inhibit

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fermentation. Less severe pretreatment conditions have limited sugar degradation, but may not open the lignocellulosic matrix sufficiently for enzymatic hydrolysis (Ramos, 2003).

Previous research has focused on determining unique steam pretreatment conditions for individual types of biomass to efficiently fractionate the biomass and recover the sugars. No single pretreatment condition is suitable for all feedstocks. The optimal pretreatment is generally a compromise between enzymatic hydrolysis efficiency, overall sugar recovery, and fermentation yield (Bura et al., 2009; Chandra et al., 2007). Agricultural residuals typically have lower optimal pretreatment severity compared to woody feedstocks (Bura et al., 2009; Sassner et al., 2005). Instead of continuously varying processing conditions to accommodate variable feedstocks, a robust and stable pretreatment would be preferable for future biorefineries.

Sulfuric acid or sulfur dioxide (SO_2) is routinely applied prior to steam pretreatment, particularly to more recalcitrant woody biomass. The use of acid catalysts enables shorter reaction time and lower pretreatment temperature (Sassner et al., 2005). Nevertheless, acid catalysts do have three major drawbacks: strongly acidic environments can lead to equipment corrosion and increased maintenance costs (Alvira et al., 2010); Acid catalyzed pretreated substrates require additional neutralization prior to enzymatic hydrolysis and fermentation, meaning greater expense for neutralizing reagents and subsequent wastewater treatment (Humbird et al., 2011); finally, sulfur is poisonous in many chemical catalytic reactions (Bartholomew et al., 1982; Dunleavy, 2006; Oudar, 1980). Since some biorefineries may also produce chemicals, such as glycols, by catalysis, the presence of sulfur may prohibit production of higher value co-products. For these three reasons, it would be desirable for future biorefineries to have the flexibility to operate under sulfur free conditions.

Consistently maintaining optimal process conditions year round may prove difficult for an industrial scale biorefinery. Pretreatment, like most chemical reactions, is sensitive to process upsets. Three variables control steam pretreatment performance: temperature, residence time, and pH (controlled by the addition of acid catalyst). Process disturbances may move any of these critical variables off their optimal setpoints. Disturbances in steam supply can impact temperature control. Variability in biomass packing density can affect residence time depending on the pretreatment reactor configuration. pH control can be particularly challenging due to variable chemical uptake by different biomass feedstocks (De Bari et al., 2007). Instability in any of these parameters may alter the final yield. The presence of a unit operation, after pretreatment but before the expensive hydrolysis and fermentation processes, that could mitigate the impact of these disturbances would significantly improve the operability of the biorefinery and enable a more stable operation.

Mechanical refining is one potential unit operation that could serve to mitigate such disturbances in the pretreatment process. As a mature technology in the papermaking industry, refining is commonly applied to develop pulp fiber qualities and enhance paper properties (Paulapuro, 2000). The main goal of refining for papermaking is to improve the bonding among fibers by exposing internal and external microfibrils. Refining also severs fibers and shortens the overall average fiber length (Paulapuro, 2000).

Previous research demonstrated that mechanical comminution by milling or grinding of raw materials before pretreatment is energy-intensive and not economically feasible (Alvira et al., 2010). Post-treatment refining, however, has been proposed to improve the enzymatic hydrolysis efficiency, allowing reduced enzyme dosage (Jones et al., 2013; Koo et al., 2011), and has been reported to be cost-effective by offsetting energy consumption (Tao et al., 2012; Zhu and Pan, 2010). A number of studies in mechanical refining at laboratory, demonstration and commercial scales have shown comparable outcomes in improving enzymatic hydrolysis of feedstocks pretreated with different methods (Chen et al., 2013; Jones et al., 2014; Zhu et al., 2010). In addition, considerable work has been done in the last 5 years on the mechanisms whereby refining enhances pretreatment effectiveness (Chen et al., 2013; Hoeger et al., 2013; Jones et al., 2013). Previous work, however, only focused on the impact of refining on digestibility of the water insoluble fraction, and did not assess how refining could improve the overall pretreatment performance by allowing milder pretreatment conditions and eliminating the need for sulfur based acid catalysts. A more holistic approach that measures total sugar recovery is needed to completely assess the benefits of mechanical refining.

This research investigates the effect of refining on the hydrolyzability of the water insoluble fraction of steam pretreated biomass. In addition, the effects of refining on overall sugar recovery are explored to better assess the use of mechanical refining to decouple pretreatment from enzymatic hydrolysis and fermentation. The specific objectives of this work are threefold. First, to determine whether refining changes the chemical composition and physical properties of steam pretreated hybrid poplar. Second, to study the influence of refining on cellulose to glucose conversion in enzymatic hydrolysis for hybrid poplar steam pretreated at a range of conditions. Finally, to investigate the effect of refining on the overall sugar recovery of hybrid poplar steam pretreated at different conditions.

2. Methods

Hybrid poplar chips were steam pretreated using 6 different conditions with or without SO_2 catalysis, shown in Fig. 1. After each pretreatment, the water soluble fraction (WSF) was analyzed to determine chemical composition. Half of the water insoluble fractions (WIF) were refined in a valley beater refiner for 30 min. All 12 samples, including refined and unrefined solids, were enzymatically hydrolyzed at 5% consistency with 10 FPU/g cellulose enzyme loading. The particle length and width for all the refined and unrefined solids were measured. The overall sugar recovery, based on monomeric glucose and xylose, was determined after steam pretreatment and enzymatic hydrolysis. Since arabinose, galactose, and mannose made up only a minor contribution to total sugars, only the sugar recovery of glucose (glucan) and xylose (xylan) were calculated.

2.1. Feedstock

Hybrid poplar chips used in this research were from fresh 18-year-old hybrid poplar obtained from Forest Concepts (Auburn, WA). Trees were debarked, chipped and screened into 1.0 cm × 2.0 cm at 5 mm thickness. Chips (50% moisture content) were stored at −20 °C until use. This poplar was composed of 0.3% arabinan, 1.1% galactan, 46.2% glucan, 15.1% xylan, 2.0% mannan, 22.6% lignin, 3.4% acetate, and 0.5% ash.

2.2. Pretreatment and processing conditions

In experiments utilizing SO_2 catalyst, samples of 800 g oven-dried weight hybrid poplar were pre-impregnated overnight with anhydrous SO_2 in plastic bags at atmospheric pressure. The amount of SO_2 added to the bag corresponded to 3% (w/w) loading of the samples, and was determined by weighing the bag before and after the addition of gas.

Steam pretreatment was performed in a 2.7 liter batch reactor (Aurora Technical, Savona, BC, Canada). Briefly, samples were subdivided into 400 g oven-dried weight batches and loaded into the