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Performance and techno-economic assessment of several solid–liquid separation technologies for processing dilute-acid pretreated corn stover

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HIGHLIGHTS

- Filtration effectively separates liquor from pretreated biomass slurries.
- High solids filter cakes are difficult to produce.
- Cake washing recovers over 95% of soluble sugars from pretreated solids.
- Vacuum belt filters offer the most favorable economics.

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ABSTRACT

Solid–liquid separation of pretreated lignocellulosic biomass slurries is a critical unit operation employed in several different processes for production of fuels and chemicals. An effective separation process achieves good recovery of solute (sugars) and efficient dewatering of the biomass slurry. Dilute acid pretreated corn stover slurries were subjected to pressure and vacuum filtration and basket centrifugation to evaluate the technical and economic merits of these technologies. Experimental performance results were used to perform detailed process simulations and economic analysis using a 2000 tonne/day biorefinery model to determine differences between the various filtration methods and their process settings. The filtration processes were able to successfully separate pretreated slurries into liquor and solid fractions with estimated sugar recoveries of at least 95% using a cake washing process. A continuous vacuum belt filter produced the most favorable process economics.

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1. Introduction

Various biochemical- and thermochemical-based processes for producing liquid transportation fuels from lignocellulosic biomass are being developed throughout the world. For biochemical-based processes, biomass sugars are produced from combined thermochemical treatment and enzymatic hydrolysis. Thermochemical treatment modifies the biomass structure to enhance enzymatic conversion of cellulose to glucose. The sugars are converted to fuels and chemicals by microbial or catalytic methods (Blanch, 2011; Huber et al., 2005).

Most biochemical conversion processes employ a solid–liquid separation (SLS) step to either remove solids from pretreated slurries or produce clarified sugar-containing streams after enzymatic hydrolysis. For example, Kinnarinen et al. (2012a, 2012b) used a pressure filter to remove residual lignin solids remaining after enzymatic cellulose hydrolysis of cardboard waste and studied the extent of cellulose conversion on filtration properties of the residual solids. Removing lignin solids from sugar streams prior to microbial conversion enables processes using immobilized cell reactors or enzyme and microbial cell recycle schemes (Weiss et al., 2013). Burke et al. (2011) used polyelectrolyte flocculating agents to enhance the separation and recovery of these lignin solids. However, a solids washing step was still required to improve sugar recovery. Regardless of the separation technique used, the clarified liquor stream contains some residual solids that may negatively impact downstream processing (Kochergin and Miller, 2011).

Residual lignin solids can be removed after microbial sugar conversion and prior to product recovery. Kochergin and Miller (2011) discuss using a solids washing operation to remove ethanol from a fermentation broth prior to distillation. Humbird et al. (2011) present the techno-economic analysis of a lignocellulose-to-ethanol process that uses a pressure belt filter to recover the lignin solids, which are then burned to produce steam and electricity.

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A biochemical conversion process using dilute acid pretreatment produces high-solids slurries in which the liquor fraction contains high concentrations of soluble hemicellulosic sugars. An SLS step can split the slurry into a hemicellulosic sugar-rich (C5) stream and a high-solids cellulosic (C6) stream for separate processing (Wright, 1988). In this paper, this option is called separate C5/C6. Washing the cellulosic solids after the initial separation recovers additional hemicellulosic sugars. The solids are then subjected to an additional thermochemical conversion step (Monavari et al., 2009) or, alternatively, the solids are enzymatically hydrolyzed to glucose.

A possible advantage of the separate C5/C6 process is an improvement in enzymatic cellulose conversion due to removal of enzyme inhibiting hemicellulose sugars. Additionally, improvements in microbial conversion of glucose to product are possible since the inhibitory compounds in the liquor fraction of the slurry produce by dilute acid pretreatment are also removed (Klinke et al., 2004). Finally, diverse products could be produced from the different sugars contained in the separate streams, potentially improving process economics. An initial economic assessment of the separate C5/C6 configuration for producing ethanol from acid-pretreated corn stover showed better economic performance than the unseparated option (Dutta et al., 2009). However, the cost and performance of SLS was based on old, somewhat incomplete data and assumptions used in an earlier study (Aden et al., 2002).

Prior experience testing gravity settling on pretreated slurry indicated poor separation and the inability of this technique to wash remaining liquor from concentrated solids focused our research on filtration methods. The purpose of this study was to evaluate the performance of pressure filtration, vacuum filtration, and basket centrifugation (a type of forced filtration) in removal of liquor from dilute acid-pretreated corn stover slurries and in concentration of cellulosic solids. In addition, hemicellulosic sugar recovery from the pretreated slurry was characterized as a function of the amount of wash water used. Higher wash water use improves sugar recovery at the cost of undesirable liquor stream dilution. The performance information generated by this study, along with equipment costs, was used to estimate minimum ethanol selling price (MESP) for a separate C5/C6 process by updating a previous techno-economic model (Humbird et al., 2011).

2. Methods

2.1. Pretreated corn stover

Pioneer maize variety 34M95 whole stover harvested from a farm near Wray, CO, USA was thermochemically pretreated with dilute sulfuric acid in a 900 dry kg/d vertical pretreatment reactor using procedures discussed in Schell et al. (2003). Operating conditions were 1% sulfuric acid in solution, 30% total solids, 190 °C, and an estimated residence time of one minute. The pretreated corn stover (PCS) slurry contained 29.7% (w/w) total solids, 17.4% insoluble solids, and 57.6 g/L xylose and 16.8 g/L glucose in the liquor fraction. All total and insoluble solids are reported in this paper on weight percent basis, unless noted otherwise. Insoluble solids mean particle diameter was measured at 215 μm via laser diffraction by Hazen Research, Inc. (Arvada, CO, USA), with 10% (v/v) particles at or below 12 μm and 90% (v/v) particles at or below 770 μm.

2.2. Pressure filter

An Outotec (Jessup, MD, USA) pressure filter test skid consisted of a 0.1 m^2 round stainless steel filter chamber, slurry tank, slurry pump, and hydraulic system. 1.3 mm thick Tamfelt (Helsinki, Finland) type S2106L1 twill woven multiple-filament polypropylene filter cloth was selected by the Outotec test engineer based on previous experience with biomass filtration using this pressure filter. Tamfelt does not provide a characteristic aperture size for this cloth, however air permeability is listed as 2.7 m^3/min-m^2 at 200 Pa. PCS was diluted with water to 11.5% insoluble solids to facilitate pumping the slurry into the filtration chamber. The slurry was also heated to approximately 70 °C to simulate the expected temperature of the slurry directly downstream of pretreatment. Diluted slurry was pumped at 300 kPa into the filter chamber and then further dewatered by pressing the cake against the filter cloth with a rubber diaphragm. The back side of the diaphragm was pressurized to 400 kPa. Then a measured volume of ambient (25 °C) or 60 °C water varying between 2.0 and 6.0 L was pumped at 400 kPa through the filter cake, and a second dewatering press using the diaphragm with 1300 kPa air pressure removed the remaining wash liquid. Wash times varied between 2 and 16 min, depending on quantity of water used. Finally, 600 kPa air was applied to the chamber to displace liquid from the cake before discharge. The feed system did not permit measurement, but average cake weight was 3.3 kg.

2.3. Vacuum filter

A Büchner filter test kit, also from Outotec, consisting of a 100 cm^2 filter funnel, flask, and vacuum pump, was used to simulate processing on a full-scale vacuum belt filter. 1.3 mm thick Tamfelt type S11M51-L2K2 twill woven multi-filament polypropylene filter cloth with air permeability of 25 m^3/min-m^2 at 200 Pa was chosen by the Outotec test engineer based on previous experience. PCS slurry was diluted to 12.6% insoluble solids and heated to approximately 70 °C to facilitate spreading of solids onto the filter cloth. For each test, 150 mL of diluted slurry was poured onto the filter cloth to form a cake using 70 kPa vacuum to draw liquid from the cake. The cake was washed by pouring a measured volume of ambient (25 °C) or 60 °C water varying between 30 and 240 mL over the cake while still under vacuum, which required between 19 and 78 s to pass through. The wash liquid was collected separately from the initial filtrate. Vacuum was sustained for 10 s following washing to displace additional liquid from the cake.

2.4. Basket centrifuge

Testing was also performed on a Western States Machine Co. (Hamilton, OH, USA) model STM-2000 basket centrifuge fitted with a 0.2 mm thick Sefar (Buffalo, NY, USA) type 3A05-0105-115-00 square weave mono-filament polypropylene filter bag with open apertures of 105 μm nominal size). The centrifuge bowl diameter was 36 cm by 15 cm tall and was powered by a variable speed 3 hp motor. PCS slurry was diluted to 11.5% insoluble solids to facilitate pumping into the centrifuge. Since experimental work with the pressure and vacuum filters showed no significant filtration rate differences between hot and cold processing, the slurry was processed at ambient temperature (25 °C) slurry and ambient temperature water was used for washing. With the basket rotating at 400 rpm, approximately 9.5 kg of slurry was pumped into the centrifuge over 1.5 min. The slurry was dewatered at 2000 rpm (795 G) for 5 min and the filtrate liquor was separately collected. Water was sprayed on the cake at a rate of 0.5 L/min while the centrifuge was spinning at 2000 rpm, varied between 4 and 22 min. The cake was dewatered by spinning the centrifuge at 2000 rpm for 5 min.

The time required to complete each phase of the filtration cycle was measured and used for capacity calculations (mass of dry solids processed per unit of filtration area over time). Experimental errors were estimated using at least four replicates for each type

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