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A whole stillage sieving process to recover fiber for cellulosic ethanol production

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ABSTRACT

A sieving step was developed to recover corn fiber from whole stillage of a dry grind process as a cellulosic ethanol feedstock, and to reduce fiber contents in distillers dried grains with solubles (DDGS). Several processes have been developed to recover fiber in the dry grind process, such as wet fractionation, dry fractionation and elusieve process. Wet fractionation and dry fractionation recover fiber before liquefaction, but addition of these steps to existing plants requires considerable modifications with high capital costs. The elusieve process recovers fiber from DDGS, but it requires drying of DDGS. To simply integrate fiber recovery to current dry grind plants with less operating and capital costs, sieving was applied to whole stillage. Commercial whole stillage samples were ground, incubated with protease or with a surfactant, and sieved. Sieving was effective to recover neutral detergent fiber (NDF), and NDF contents in retentate samples were increased by 45–101%. In addition, permeate samples, called enhanced DDGS, exhibited decreased fiber contents and increased oil contents. Among treatments, grinding before sieving was more effective to recover fiber with high NDF contents and low protein and oil contents. Fiber recovered from whole stillage and ground whole stillage were hydrolyzed for cellulosic sugars. Whole stillage and ground whole stillage were sieved in a scaled-up (15×) vibrator shaker. The retentate samples were pretreated with dilute acid followed by hydrolysis. Ground whole stillage showed higher sugar yields than whole stillage. After hydrolysis, glucose, xylose and arabinose yields from ground whole stillage were 90.78, 92.93 and 76.99%, respectively. Grinding before sieving produces more eDDGS, and could potentially increase downstream ethanol yields.

1. Introduction

In a conventional dry grind ethanol process, corn is milled, liquefied, and simultaneously saccharified and fermented to produce ethanol. Ethanol is distilled, and underflow from the distillation column is called whole stillage (non-fermentable components). Whole stillage consists primarily of water, fiber, protein, oil, unconverted starch and dead yeast cells. Whole stillage is centrifuged to produce thin stillage (supernatant) and wet cake (suspended solid). Thin stillage is further evaporated and concentrated to syrup. The wet cake and the syrup are blended and dried to produce distillers dried grains with solubles (DDGS).

The conventional dry grind process converts storage carbohydrate (starch) in corn kernels into ethanol. However, to increase ethanol yields, much research has been focused on utilization of structural carbohydrates from pericarp (coarse) fiber and endosperm (fine) fiber (Dien et al., 2005a). Corn fiber is composed of 11–23% starch, 12–18% cellulose, 18–28% xylan, 11–19% arabinan, 11–12% protein and 2% oil on a dry basis (Leathers, 1998). About 2.04 kg (4.5 lb) of corn fiber is obtained from 25.4 kg of corn (56 lb) with theoretical ethanol yield of 1.14 L (0.3 gal) (Saha et al., 1998). Another advantage of recovering fiber in the dry grind process is that it improves the nutritional value of DDGS by increasing protein, amino acids and fat contents (Martinez-Amezcua et al., 2007). Due to high fiber contents, DDGS produced from conventional dry grind plants is mainly used as an ingredient in ruminant

Abbreviations: ANOVA, analysis of variance; DDGS, distillers dried grains with solubles; eDDGS, enhanced distillers dried grains with solubles; E-Mill, enzymatic dry grind process; GWS, ground whole stillage; LSD, Fisher's least significant difference; NDF, neutral detergent fiber; WS, whole stillage; WSP, whole stillage incubated with protease; WSS, whole stillage incubated with surfactant.

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animal diets. However, DDGS with decreased fiber content would be suitable for nonruminant animals (Rausch and Belyea, 2006).

Several processes have been developed to recover fiber in the dry grind ethanol process. Fiber can be separated by wet fractionation (enzymatic milling (E-Mill) corn process) (Singh et al., 2005), dry fractionation (quick fiber process) (Singh et al., 1999) and elusieve process (Srinivasan et al., 2005). In the E-Mill process, germ, pericarp fiber and endosperm fiber are recovered before liquefaction. Corn kernels are soaked in water for 6–12 h followed by coarse grinding. Ground samples are incubated with protease and starch degrading enzymes for 2–4 h. Then, germ and coarse fiber are recovered by flotation. The remaining slurry is screened using a 200-mesh screen (74 μm openings), and endosperm fiber is recovered on the screen (Singh et al., 2005). In the quick fiber process, pericarp fiber is recovered before liquefaction. By increasing the density of corn slurry, pericarp fiber floats at a specific gravity of 1.090–1.098 (12–13 Beí) (Singh et al., 1999). Unlike the E-Mill and quick fiber processes, the elusieve process entails separation of fiber in DDGS by sieving and elutriation (Srinivasan et al., 2005). This process requires a low capital cost for a dry grind ethanol plant to recover fiber from DDGS, of which the payback period is less than 2 years for 287.69 million liter (76 million gallon) ethanol production per year (Srinivasan et al., 2006).

Fractionation technologies prior to fermentation, such as the E-Mill process or quick fiber process, require substantial retrofitting of a conventional dry grind ethanol plant and significant capital investment. Fractionation technologies after fermentation, such as the elusieve process, require drying of DDGS or DDG prior to separation of fiber. In this study, fiber was recovered from whole stillage by sieving prior to centrifugation. This strategy to recover fiber requires minimal change in the conventional dry grind process compared to other strategies and no drying step is required prior to separation of fiber. The objectives of this study were: (1) to investigate the effects of sieving on fiber recovery in whole stillage, (2) to determine the nutrient value of fiber-removed DDGS (enhanced DDGS), and (3) to determine sugar yields from enzymatic hydrolysis of recovered corn fiber.

2. Materials and methods

2.1. Whole stillage, enzymes and chemicals

Whole stillage sample was obtained from One Earth Energy, LLC in Gibson City, IL and stored at 4 °C prior to testing. Protease (Fermgen) was obtained from Genencor International (Palo Alto, CA). Fermgen is an acid proteolytic enzyme obtained by controlled fermentation of a selected strain of *Trichoderma reesei*. Protease activity is 1000 SAPU/g (SAPU = Spectrophotometer Acid Protease Units). The surfactant, polyethylene sorbitol ester (TWEEN 80), was purchased from Fisher Scientific (Fair Lawn, NJ).

2.2. Whole stillage treatments

Whole stillage (200 g, wet basis) was either ground (GWS), incubated with protease (WSP) or incubated with surfactant (WSS). For GWS, the whole stillage sample was ground using a Quaker City Mill (Model 4-E, The Straub Co., Hatboro, PA) (Eckhoff et al., 1996). For WSP, the pH of the whole stillage was adjusted to 4.0 using sulfuric acid. Then, 30 μL protease was added to the whole stillage with incubation for 2 h at 48 °C (Singh et al., 2005). WSS was prepared following a similar procedure to WSP. WSS was incubated at pH 4.0 for 2 h at 48 °C with 1% surfactant. WSP and WSS were mixed every 30 min during incubation. For the scale-up test, whole stillage and ground whole stillage were chosen.

2.3. Sieving

The sieving method was adapted from Eckhoff et al. (1996). Control and treatment samples were transferred to a sieve placed on top of a bucket and shaken for 15 min using a sieve shaker (RX-86, W. S. Tyler Co., Cleveland, OH). During sieving, samples were continuously dispersed by spatula and washed using 500 mL of water. Three different sieve sizes were used: U.S. No.100-mesh (149 μm), U.S. No. 200-mesh (74 μm) and U.S. No. 325-mesh (44 μm). The material that passed through the sieve was collected in the bucket and named as enhanced distillers dried grains with solubles (eDDGS). For the mass balance, the dry weight of the retentate and eDDGS were determined by measuring moisture contents by drying samples at 135 °C for 2 h.

For the scale-up study, 3 kg whole stillage and ground whole stillage were sieved using a 91.4 cm vibratory sieve screener (LS18S33, SWECO, Los Angeles, CA) equipped with U.S. No. 270-mesh (53 μm) (Somavat et al., 2016). During sieving, 2 L of water was used to wash the samples. After sieving, samples retained on the sieve were recovered, and moisture content was measured by incubating at 135 °C for 2 h. The recovered samples were then subjected to pretreatment and hydrolysis steps. Samples that passed through the sieve were discarded.

For samples retained on the sieve, recovery yields were determined by dividing the dry weight of the recovered sample by the dry weight of the starting sample. The sieving was performed in duplicate, and each replicate was analyzed for neutral detergent fiber (NDF) content (Van Soest et al., 1991), crude protein (AOAC 2003, Method: 990.03) and crude fat (AOAC 2003, Method: 920.39) in duplicate. Analysis of variance (ANOVA) and Fisher's least significant difference (LSD) with $p < 0.05$ was used to compare recovery yields and NDF recovery across treatments and sieve sizes.

2.4. Pretreatment and hydrolysis from recovered scale-up test sample

Scale-up test samples that were recovered on the sieve underwent pretreatment and hydrolysis. The samples were pretreated in stainless steel pipe reactors in a fluidized sand bath. The reactors were made of 316 stainless steel with 10.48 cm (4.125″) length × 1.91 cm (0.75″) outer diameter × 0.165 cm (0.065″) wall thickness tubing (SS-T12-S-065-20, Swagelok, Chicago Fluid System Technologies, Chicago, IL), with caps on both ends (SS-1210-C, Swagelok, Chicago Fluid System Technologies, Chicago, IL). To measure the internal temperature of the reactors, one tube reactor was assembled with a thermocouple (39105K212, Penetration/Immersion Thermocouple Probe Mini Conn (Pointed-Tip, Type K, −418 to 1652 °F), McMaster-Carr, Robbinsville, NJ). A datalogger (HH306/306A, Datalogger Thermometer, Omega, Stamford, CT) was used to record the temperature of the thermocouple. Pretreatment tubes were capped and placed in the fluidized sand bath (IFB-51 Industrial Fluidized Bath, Techne Inc., Burlington, NJ) along with the tube reactor fitted with the thermocouple. The sand bath was set 20 °C higher than the desired temperature to achieve quick heat up times. The thermocouple was used to determine when the desired internal temperature in the tubes was achieved. The pretreatment conditions were modified from previous reports (Dien et al., 2005b; Singh et al., 2003). Pretreatment was conducted at 20% solid content by mixing 12 mL of 0.5% v/v sulfuric acid with 3 g of dry solids biomass. Pretreatment was performed at 150 °C for 20 min. After 20 min operation time, the reactors were cooled down in cold water. It took about 3 min to heat up to 150 °C and 20 min to cool down the reactors to room temperature. The pretreated sample was transferred with minimal losses to a pre-weighed conical tube for composition analysis or to a 250 mL bottle for hydrolysis.