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Optimization of the ammonia fiber explosion (AFEX) treatment parameters for enzymatic hydrolysis of corn stover

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1. Introduction

A unique physiochemical pretreatment referred to as Ammonia Fiber Explosion (AFEX) offers potential as a pretreatment for lignocellulosic material (Alizadeh et al., 2004; Gollapalli et al., 2002; Foster et al., 2001; Dale, 1999; Holtzapple et al., 1992; Dale and Moreira, 1982). In AFEX pretreatment biomass is treated with liquid anhydrous ammonia at moderate temperatures (60–100 °C) and high pressure (250–300 psi) for 5 min. Then the pressure is rapidly released. In this process the combined chemical and physical effects of lignin solubilization, hemicellulose hydrolysis, cellulose decrystallization, and increased surface area, enables near complete enzymatic conversion of cellulose and hemicellulose to fermentable sugars (Mosier et al., 2004). The AFEX treatment has some unique features that distinguish it from other biomass treatments. Some of these distinctive features are listed below:

- Nearly all of the ammonia can be recovered and reused while the remaining serves as nitrogen source for microbes, in downstream processes (Dale and Moreira, 1983).
- There is no wash stream in the process. Dry matter recovery following the AFEX treatment is essentially 100%. AFEX is basically a dry to dry process. Treated biomass is stable for long periods and can be fed at very high solids loadings in enzymatic hydrolysis or fermentation process.
- Cellulose and hemicellulose are well preserved in the AFEX process, with little or no degradation (Moniruzzaman et al., 1997).
- There is no need for neutralization prior to the enzymatic hydrolysis of AFEX treated biomass.
- Enzymatic hydrolysis of AFEX treated biomass produce clean sugar streams for subsequent fermentation process.

The goal of this study was to define the optimal conditions for AFEX treatment of corn stover to provide maximum sugar yields by enzymatic hydrolysis. AFEX pretreatment conditions (temperature, moisture content, ammonia loadings and treatment time) were varied to find an optimum. Optimal pretreatment conditions for corn stover were found to be: (1) temperature, 90 °C; (2) ammonia loading, 1.0 kg of ammonia:kg of dry corn stover; (3) moisture content of corn stover, 60% (dry weight basis (dwb)); and (4) residence time (holding at target temperature), 5 min. Approximately 100% of the theoretical glucose yield and 80% of theoretical xylose yield were obtained during enzymatic hydrolysis of the optimal treated corn stover using 60 filter paper unit (FPU) of cellulase enzyme/g of glucan. The ethanol yield of optimally AFEX-treated corn stover was increased up to 2.3 times over that of an untreated sample.

2. Methods

To carry out AFEX treatment, corn stover samples were moistened with distilled water to the desired moisture content and allowed to equilibrate for 30 min. The prewetted samples were placed in the pressure vessel. The vessel was topped up with stainless steel pellets (approximately 1 mm diameter) to occupy the void space and thus minimize transformation of the ammonia from liquid to gas during loading, and then the lid was bolted shut. Precalibrated sample cylinders were filled

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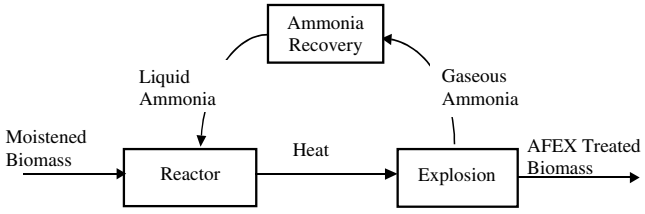

Fig. 1. Schematic diagram of AFEX process.

with the desired amount of ammonia to charge to the system. The entire reactor assembly was placed in a 400 W PARR (Moline, IL) heating mantle to warm the unit to the desired temperature. To avoid overheating, the reactor was taken out of the heater at approximately 10 °C prior to the target temperature, and, if needed, the unit was placed in a bath of cold water to maintain the system at the set temperature. After the experiment was completed the exhaust valve was rapidly opened to relieve the pressure and accomplish the explosion. The treated corn stover samples were removed and allowed to stand overnight in a fume hood to evaporate the residual ammonia. Fig. 1 illustrates the schematic diagram of AFEX process.

3. Results

3.1. Establishing the optimal AFEX pretreatment conditions

Fig. 2 shows the effects of AFEX treatment temperature on the percent conversion of corn stover glucan and xylan to monomeric glucose and xylose. Similarly Fig. 3 shows the effects of moisture content on conversion to monomeric sugars. In these figures, ammonia loading, enzyme (Spezyme CP provided by NREL) concentration, time of hydrolysis and AFEX treatment time are all held constant. As Fig. 2 depicts, increasing temperature from 90 °C to 100 °C and 110 °C improves the conversion of glucan to glucose by only about 1%. Apparently further increases in treatment temperature beyond 90 °C do not have much additional beneficial effect. Furthermore, our fermentation results showed that more ethanol was produced from biomass treated at 90 °C compared to stover treated 100 °C and 110 °C. Thus 90 °C was selected as the optimal temperature.

It is apparent from Fig. 3 that both glucan and xylan conversion increased with increasing moisture content and the maximum conversion occurred with biomass treated at 60% moisture content (dry weight basis, 60 g water per 100 g dry stover).

Experimental data showing the effects of various ammonia loadings on glucan and xylan conversion are summarized in Fig. 4. As this figure suggests, enzymatic digestibility of AFEX treated corn stover improves with

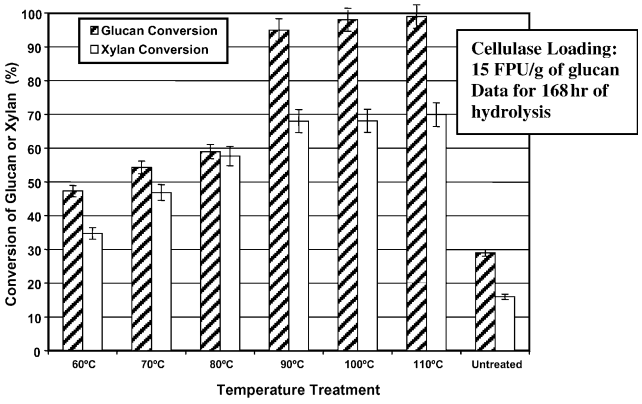

Fig. 2. Effects of treatment temperature on glucan and xylan conversion of AFEX treated corn stover at 60% moisture content (dwb) and 1:1 ammonia loading. All the runs were kept at the set temperature for 5 min. The enzymatic hydrolysis was run in duplicate and the error bars show the upper and lower measurements.

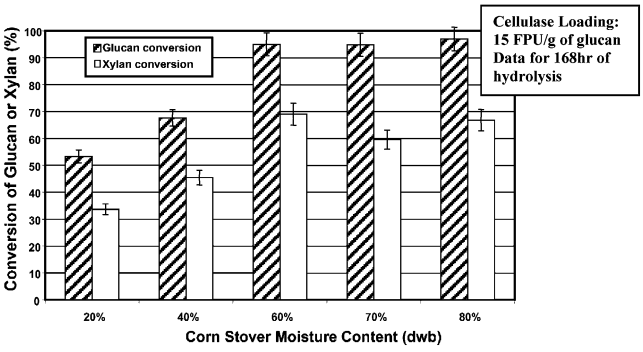

Fig. 3. Effects of moisture content on glucan and xylan conversion of AFEX treated corn stover at 90 °C and 1:1 ammonia loading. All the runs were kept at the set temperature for 5 min. The enzymatic hydrolysis was run in duplicate and the error bars show the upper and lower measurements.

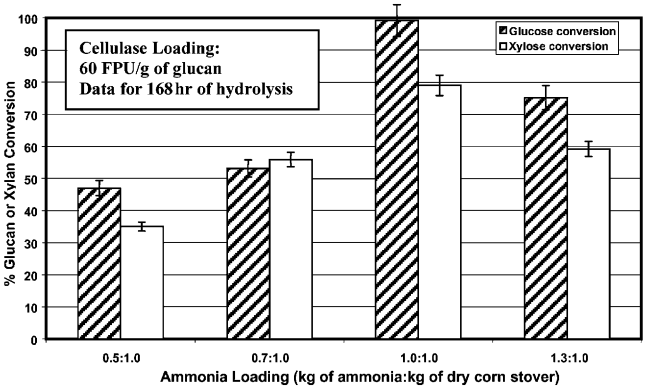

Fig. 4. Effects of ammonia loading (kg of NH_3:kg of dry biomass) on enzymatic conversion of glucan and xylan for AFEX treatment of corn stover at 90 °C and 60% moisture content (dwb). All the runs were kept at the set temperature for 5 min. The enzymatic hydrolysis was run in duplicate and the error bars show the upper and lower measurements.

increasing ammonia loadings. Both glucan and xylan conversion attained their maximum at approximately 1 kg of ammonia per 1 kg dry stover.