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# Xylose production from corn stover biomass by steam explosion combined with enzymatic digestibility

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#### HIGHLIGHTS

• A novel conversion process of steam explosion and digestibility was exploited.

• Steam explosion increased the recovery of sugars and reduced the formation of DPs.

• Periodic peristalsis enhanced the digestibility of SECS at high solid loading.

• The highest yield of xylose and total sugar reached to 62.8% and 72.3%, respectively.

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## ABSTRACT

A novel conversion process using steam explosion combined with enzymatic digestibility was exploited to increase sugar yield. Results showed that glucan and xylan recovery decreased with the increase of holding temperature and residence time in SE, respectively, while glucan and xylan conversion exhibited an opposite trend. The optimal conditions of steam explosion were 160 °C and 48 min, under which glucan and xylan recovery was 93.4% and 71.6%, respectively. Glucan and xylan conversion at 18% solid loading by periodic peristalsis increased by 3.4–5.8% and 4.5–6.2%, respectively, compared with that by water baths shaker. In the whole process, glucose, xylose and total sugar yield to 77.3%, 62.8% and 72.3%, respectively. The yield of hydroxymethyl furfural, furfural and lignin-derived products was  $6.3 \times 10^{-2}$ ,  $7.5 \times 10^{-2}$  and less than  $3.7 \times 10^{-2}$  g/100 g feedstock, respectively. This novel conversion process increased sugar recovery, reduced degradation products formation, improved digestibility efficiency, and hence increased sugar yield.

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#### 1. Introduction

Energy security and environmental concern have strengthened the interests in alternative and nonpetroleum-based sources of energy (Jonker et al., 2015; Chen et al., 2014; Himmel et al., 2007). Lignocellulosic biomass, which can be converted into alternative fuels such as ethanol, should be the primary energy resource due to its high annual production worldwide ( $1 \times 10^{10}$  MT/year) (Alvira et al., 2010; Chen and Qiu, 2010). It can be obtained as the wastes of the agriculture and forestry industries, or from energy crops especially cultivated for the purpose (Chen and Liu, 2015; Ko et al., 2015). Corn stover is a potential feedstock for ethanol production due to high sugar content and low price of

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preparation (Ou et al., 2014; Luo et al., 2009). Converting low value corn stover into ethanol is also beneficial for agricultural economy.

Ethanol production from lignocellulosic biomass mainly comprises three unit operations: pretreatment, digestibility of carbohydrates and fermentation of sugars. Pretreatment is an essential step for obtaining potentially fermentable sugars in digestibility and it represents one of the main economic costs (Mood et al., 2013; Chen and Liu, 2007). The goal of pretreatment is to change the physicochemical factors hindering the digestibility of carbohydrates and thus increase the accessibility of enzymes to the carbohydrates (Alvira et al., 2010; Mood et al., 2013). Steam explosion (SE) has attracted more attention due to less hazardous chemicals use, lower environmental impact, and larger chip size using compared with other pretreatments (Chen and Liu, 2014; Agbor et al., 2011; Alvira et al., 2010).

However, SE of lignocellulosic biomass to improve the conversion efficiency is still technically problematic because of the degradation of hemicellulose (mainly xylan) and the generation of





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various degradation products (Sharma et al., 2015; Mood et al., 2013; Agbor et al., 2011). Corn stover, corncob, and other agricultural stalks are rich in hemicellulose, which is the second most abundant polysaccharide in nature (Mood et al., 2013; Chen and Liu, 2015). The ultimate goal of biomass refinery is 'zero waste', meaning that all sugars or carbons fixed in lignocellulosic biomass must be utilized efficiently (Zeng et al., 2014). Too many losses of hemicellulose should reduce the utilization efficiency of sugars. Worse still, the degradation of hemicellulose also leads to more generation of degradation products (DPs) concomitantly, such as furfural. The pretreated liquor of SE contains, in addition to fermentable sugars and furan derivatives, weak acids and phenolic compounds generating from lignin. Some of these DPs significantly present the inhibitory effects on the enzymes and are toxic to the microorganisms used in the fermentation (Zhao and Chen, 2014; Cantarella et al., 2004). Because digestibility is catalyzed by enzymes and also accounts for most economic costs of the conversion process, pretreatment research should also focus on identifying, evaluating, and support the subsequent digestibility of pretreated solid with lower enzyme dosage, shorter conversion time and higher solid loading (Mood et al., 2013; Alvira et al., 2010). Therefore, in order to break through these bottlenecks that exist in SE and digestibility, an effective conversion process should be exploited.

In this study, a novel conversion process using SE combined with enzymatic digestibility was exploited to increase glucose and xylose yield. Sugar recovery, sugar conversion, and sugar yield as well as DPs yield were used as key metrics for the assessment of SE performance. The enzymatic digestibility of steam exploded corn stover (SECS) was evaluated with different enzyme and solid loadings. The digestibility kinetics was also carried out. Periodic peristalsis was used to enhance the digestibility efficiency at high solid loading compared with water baths shaker. The water holding capacity and specific surface area of SECS and their correlations with digestibility were analyzed to get insight into the mechanism of the novel SE strategy.

### 2. Methods

#### 2.1. Biomass resource

Corn stover biomass was kindly provided by Chinese Academy of Agriculture Sciences (CAAS) in Beijing, China. It was air dried to 5–10% (w/w) moisture content based on dry weight (DW). The composition analysis was conducted according to the laboratory analysis protocol (LAP) of National Renewable Energy Laboratory, Colorado, USA (Hames et al., 2008).

#### 2.2. Steam explosion

Prior to SE, corn stover biomass was manually cut into 2.0 cm particles. The moisture content (DW) of corn stover biomass was then adjusted to 35% (w/w). SE was carried out in a 20-l steam explosion reactor connected with a steam generator (Weihai Automatic Control Co. Ltd., China). During SE, 1.0 kg corn stover biomass (DW) was added into the steam explosion reactor. High temperature steam supplied by the steam generator was rapidly injected into the reactor until holding temperature reached to desired values (150, 160, 170 and 180 °C). The reaction system was then maintained for a certain residence time (12, 24, 36 and 48 min). As the desired residence time being reached, the pressure of reaction system was then exploded into a reception tank. Time used for the explosion step before the corn stover biomass was exposed to atmospheric pressure was less than 2 s. The process that corn

stover biomass was firstly exposed at a low holding temperature for a long residence time and then exploded at a high exploding pressure was named low holding temperature and high exploding pressure steam explosion (LHT-HEP-SE). The control experiment of SE was conducted at 200 °C for 6 min. After SE, the water insoluble fraction was washed using 15-1 water. The SECS and pretreated liquor was collected for further analysis.

Pretreatment severity (log  $R_0$ ) is calculated as follows (Overend and Chornet, 1987):

$$\log R_0 = t \times \exp\left[(T - 100)/14.75\right]$$
(1)

where *t* is residence time, min; *T* is holding temperature, °C.

#### 2.3. Enzymatic digestibility

Cellulase preparation Cellic CTEC2 was a generous gift from Novzymes (Beijing, China). Filter paper activity (FPU) of cellulase was 108 FPU/mL, while cellobiase activity of  $\beta$ -glucosidase was 1290 CBU/mL. For the experiments of solid loading, the digestibility was carried out at a certain solid loading of 1%, 6%, 12% and 18%, respectively, in citrate buffer solution (50 mM, pH 4.8) with an enzyme loading of 10 FPU/g solid. For the experiments of enzyme loading, the digestibility was conducted at 12% solid loading with an enzyme loading of 5, 10, 15 and 20 FPU/g solid, respectively. The above experiments of digestibility were carried out in an water baths shaker at 50 °C with 200 rpm for 120 h. Compared with that by water baths shaker, high solid enzymatic digestibility (18% solid loading) by periodic peristalsis was carried out at 50 °C with 20 rpm for 120 h in the periodic peristalsis enzymatic digestibility reactor system, which had been developed in our previous study.

Enzymatic digestibility kinetics was conducted at 6% solid loading with an enzyme loading of 10 FPU/g solid. 1.0 mL supernatant was sampled at different digestibility time points, respectively, for sugars analysis.

Sugar conversion and sugar conversion rate were calculated as follows:

Glucan conversion (%)

$$= (glucose in hydrolyzate \times 162/180)/glucan in$$
  
steam exploded corn stover biomass  $\times 100\%$  (2)

Xylan conversion (%)

= (xylose in hydrolyzate 
$$\times$$
 132/150)/xylan in steam  
exploded corn stover biomass  $\times$  100%

(3)

Sugar conversion rate (SCR) = 
$$(SC_i - SC_j) \times TSM / [V \times (i - j)]$$
  
(4)

where SCR is sugar conversion rate, g L<sup>-1</sup> h<sup>-1</sup>; SC is sugar conversion, %; TSM is total sugar mass in SECS, g; *V* is volume of the enzymatic digestibility mixture, L; *i* = 6, 12, 24, 48, 72, 96, 120 h and *j* = 0, 6, 12, 24, 48, 72, 96 h is digestibility time, h.

#### 2.4. Characterizations of corn stover biomass

The water holding capacity of untreated corn stover (UCS) and SECS was determined by centrifuge separation method. 1.0 g sample (DW) was mixed with 10 mL water in 50 ml beaker for 1.0 h. The mixture was centrifuged at a certain separation factor (F = 1,000) for 5 min. Separation factor (F) was defined as the ratio of rotational acceleration and gravity acceleration and calculated as follows:

$$F = \omega^2 R/g = (2\pi Rn/60)^2/(Rg) = Rn^2/895$$
(5)

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