Carbohydrate Polymers 114 (2014) 21–26

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Effects of acid impregnated steam explosion process on xylose recovery and enzymatic conversion of cellulose in corncob

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ARTICLE INFO

Article history:
Received 4 April 2014
Received in revised form 20 June 2014
Accepted 18 July 2014
Available online 30 July 2014

Keywords:
Corncob residue
Enzymatic hydrolysis
Steam explosion
Acid impregnation

ABSTRACT

Corncob residue is a cellulose-rich byproduct obtained from industrial xylose production via dilute acid hydrolysis processes. Enzymatic hydrolysis of cellulose in acid hydrolysis residue of corncob (AHRC) is often less efficient without further pretreatment. In this work, the process characteristics of acid impregnated steam explosion were studied in conjunction with a dilute acid process, and their effects on physiochemical changes and enzymatic saccharification of corncob residue were compared. With the acid impregnated steam explosion process, both higher xylose recovery and higher cellulose conversion were obtained. The maximum conversion of cellulose in acid impregnated steam explosion residue of corncob (ASERC) reached 85.3%, which was 1.6 times higher than that of AHRC. Biomass compositional analysis showed similar cellulose and lignin content in ASERC and AHRC. XRD analysis demonstrated comparable crystallinity of ASERC and AHRC. The improved enzymatic hydrolysis efficiency was attributed to higher porosity in ASERC, measured by mercury porosimetry.

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1. Introduction

With depletion of fossil fuels and concerns over climate change, utilization of renewable and sustainable resources for the production of fuels, chemicals and materials has become a global research focus. Lignocellulosic biomass, such as agricultural residues, is recognized as one of the few significant renewable resources available worldwide and can be utilized for the production of sustainable value-added products at competitive prices (Gallezot, 2012). In China, an estimate of 20 million tons of corncob are produced every year (Gu, Zhang, & Bao, 2014). Corncob has been used in the production of xylose by dilute acid hydrolysis in addition to being traditionally used as boiler fuels (Cai, Ge, Ling, Cheng, & Ping, 2012). After converting hemicellulose into xylose, cellulose and lignin are the main components remaining in corncob residues (Yinbo, Zhu, Liu, Bao, & Lin, 2006). It is estimated that about 5–6 t of corncob residues can be obtained after 1 t of xylose is produced according to a local factory in China. Since the cost of pretreatment has been shared by xylose production, ethanol production from corncob residue with an enzymatic hydrolysis process is a promising approach to further improve the process economics (Zhang, Ma, Yu, Zhang, & Tan, 2011a).

The cost of bioethanol production from corncob residue is significantly influenced by cellulase production and enzymatic hydrolysis. Obtaining higher cellulase activity and hydrolysis efficiency is one of the most pivotal steps. Enzymatic hydrolysis of cellulose from traditional acid hydrolysis residue of corncob (AHRC) is less efficient. Therefore, AHRC has to be subjected to sulfite pretreatment to remove part of lignin and enhance its enzymatic digestibility, which in turn increases the cost of the entire operation (Bu, Xing, Yu, Gao, & Jiang, 2012; Cheng et al., 2011a,b). In addition, sulfite pretreatment also generates soluble inhibitors, especially lignin-derived phenolic compounds which hamper both enzymatic hydrolysis and glucose fermentation.

Steam explosion is one of the most extensively studied pretreatment methods, which opens the compact lignocellulosic plant cell walls, causes hemicellulose degradation and lignin transformation, thus increases the potential of cellulose hydrolysis (Chen & Qiu, 2010; Menon & Rao, 2012; Wood et al., 2014; Zeng, Tong, Wang, Zhu, & Ingram, 2014). However, the hemicellulose removal and the lignin-carbohydrate matrix disruption are often incomplete during the steam explosion process (Sun et al., 2014). As a result, a mixture sugar solution including xylose and glucose is always obtained after enzymatic hydrolysis. However the co-fermentation of xylose and glucose is still a challenge facing the cellulosic biofuel

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http://dx.doi.org/10.1016/j.carbpol.2014.07.051
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industry (Casey et al., 2013). Thus, one of the options is to develop a convenient and environmentally friendly method to minimize the hemicellulose content in the biomass residue after the pretreatment.

Acid impregnation has been used in conjunction with steam explosion to enhance hemicellulose hydrolysis efficiency (Maurelli, Ionata, La Cara, & Morana, 2013; Zimbardi et al., 2007). In our previous study, an acid impregnated steam explosion process was chosen to prepare the xylose-rich hydrolysate from corncob (Wang, Fan, Tang, & Yuan, 2013). At the end of this process, the acid impregnated steam explosion residue of corncob (ASERC) consisted of mainly cellulose and lignin. It was not clear how the process characteristics would impact the xylose production and subsequent cellulose hydrolysis. In current work, the relation between hemicellulose removal (xylose and xylo-oligosaccharide production) and cellulose conversion was studied as a function of acid concentration and steam explosion severity. Since plant cell walls consist of three major components which are interrelated to each other, the exact mechanism by which hydrolysis is improved after pretreatment are not well understood (Pu, Hu, Huang, Davison, & Ragauskas, 2013). Several representative samples were chosen to understand the structural changes occurred during different pretreatment processes and their relations to enzymatic hydrolysis. The physical structures of corncob residues were characterized by XRD, SEM and mercury porosimetry analysis. These results provide useful information to the utilization of corncob for large scale xylose and glucose production.

2. Methods

2.1. Biomass material

Corncob was provided by Futaste Company in Yucheng, Shandong province of China. The air-dried corncob was mechanically milled and screened to obtain particles with size of about 1 cm, and then it was stored at room temperature. The air-dried corncob contained 41.5 ± 0.5% cellulose, 35.8 ± 0.4% hemicellulose (32.5 ± 0.3% xylan, 3.3 ± 0.1% arabinose) and 16.2 ± 0.2% lignin.

2.2. Xylose production process

2.2.1. Acid impregnated steam explosion process

An amount of 100 g air-dried corncob particles was impregnated in a 500 mL solution of different concentration of sulfuric acid at room temperature for 6 h. After impregnation, the mixture was filtrated and the wet solid was added into a 5 L batch reactor. The reactor was sealed, and quickly heated to the required temperature within 1 min by filling with high temperature water steam. The wet solid was kept at the required temperature for 5 min. Then the ball valve at the bottom of the reactor was rapidly opened to bring the reactor to atmospheric pressure. The steam-exploded sample was suspended in 1 L of water at 50 °C and stirred for 1 h before filtering. The extraction process was repeated twice, and the water extractions were combined to obtain hemicellulose hydrolysate of corncob. The solid residue (ASERC) was air dried for enzymatic hydrolysis experiment.

The severity factor of steam explosion was determined using an empirical equation (Chornet & Overend, 1991):

$$\text{Severity factor} = \log_{10} Ro = \log_{10}\left[t \times \exp\left(\frac{(T-100)}{14.75}\right)\right] \quad (1)$$

where t is the retention time (min) and T is the process temperature (°C). The process conditions were as follows: times of 5 min, and temperatures of 180–220 °C, which corresponded to severity values of 3.055–4.237. Three runs were carried out for each experimental point.

2.2.2. Acid hydrolysis process

An amount of 100 g air-dried corncob particles mixed with 2 L of 2% (w/v) sulfuric acid was added into a 2.5 L autoclave reactor. The optimal condition for xylose recovery from sulfuric acid hydrolysis was 100 °C, 120 min with a mechanical agitation (Dominguez, Cao, Gong, & Tsao, 1997). After reaction, the autoclave reactor was cooled down to 40 °C by cold water, and the liquid was collected for saccharification analysis. The solid residue (AHRC) was air dried for enzymatic hydrolysis experiment.

2.3. Enzymatic hydrolysis process

An amount of 10 g air-dried corncob residue was transferred to a 250 mL shake flask containing 150 mL of a 0.05 M acetate buffer (pH 4.8) at 15% (w/v) solid loading. The cellulase (Novozyme Cellic CTec2) activity was 206 FPU/mL, assayed by the description of IUPACA (Ghose, 1987). The digestion of the cellulose was conducted at a cellulase activity level of 18 FPU/g cellulose. The enzymatic digestion was carried out at 50 °C and 100 rpm for 72 h. All the experiments were performed in triplicate, with the average value being reported.

2.4. Analytical methods

2.4.1. Saccharification analysis

The sugar composition of the hemicellulose hydrolysate and enzymatic hydrolysate was determined by high performance liquid chromatography (HPLC) using a refractive index detector (Hitachi, Tokyo, Japan). A Sugar-pak1 column (Waters, Milford, MA, USA) was used at 80 °C with ultra-water as the eluent at a flow rate of 0.5 mL/min. Xylooligosaccharide was determined after a acid hydrolysis process (4%H_2SO_4, 121 °C, 20 min) (Gullon et al., 2010). The concentration of furfural, 5-hydroxymethylfurfural (HMF), formic acid and acetic acid were measured on an Aminex HPX-87H column (Bio-Rad, Hercules, CA,USA) at 45 °C using a refractive index detector (Hitachi, Tokyo,Japan). As the mobile phase, 5 mmol/L H_2SO_4 was supplied at a flow rate of 0.6 mL/min. The concentration of total phenols was measured by Scalbert's method (Scalbert, Monties, & Janin, 1989).

The results of saccharification process were calculated by the following equations:

$$\text{Xylose yield (\%)} = \left[\frac{\text{xylose (g) released in xylose production process}}{\text{xylan (g) in the corncob}}\right] \quad (2)$$

$$\text{Xylo-oligosaccharide yield (\%)} = \left[\frac{\text{xylo-oligosaccharide (g) released in xylose production process}}{\text{xylan (g) in the corncob}}\right] \quad (3)$$

$$\text{Cellulose conversion (\%)} = \left[\frac{\text{glucose (g) released in enzymatic hydrolysis}}{\text{cellulose (g) in the corncob residue}}\right] \quad (4)$$

2.4.2. Compositional analysis

The compositions of corncob residues were determined using ANKOM fiber analyzer instrument.

2.4.3. Morphological analysis

X-ray diffraction (XRD) was conducted using a Rigaku D/Max 2500 VB2+/PC, with 2°/min scan speed. Copper radiation (k = 0.154184 nm) was generated at a voltage of 40 kV. The scan scope was 5–50°. In order to compare the crystalline changes among different samples, an empirical equation was adopted to estimate the percentage crystallinity (CrI, Crystallinity Index) (Thygesen, Oddershede, Lilholt, Thomsen, & Ståhl, 2005):

$$\mathrm{CrI} = \frac{(I_{\mathrm{total}} - I_{\mathrm{am}})}{I_{\mathrm{total}}} \times 100 \quad (5)$$

where I_{total} is the scattered intensity at about $2\theta = 22.1°$ due to both crystalline and amorphous portions of the biomass, I_{am} is the scattered intensity at about $2\theta = 16°$ due to the amorphous portion only.

Scanning electron microscope (SEM) analysis was operated at 10–15 kV accelerated voltage using a Hitachi S-4800 instrument (Tokyo, Japan).

Mercury porosimetry was used to characterize pore structure of the granules using a PoreMasterGT 60 instrument (USA). 0.5–0.8 g samples were dried at 40 °C in vacuum oven for 48 h and placed into the sample cell which was evacuated for about 3 min (below 7 Pa) and filled with mercury in the filling apparatus. The density of each sample was determined by using a Quantachrome Ultrapycnometer 1000 instrument (USA). Determination of diameter of the pores by low-pressure porosimetry is 14–200 μm and by high-pressure porosimetry is 7 nm–14 μm (Westermarck, 2000).

3. Results and discussion

3.1. Effect of acid impregnated steam explosion process on xylose recovery and cellulose conversion

The acid impregnated steam explosion process was used to obtain high yield of xylose from corncob, and to increase the rate of enzymatic hydrolysis of cellulose from corncob residue. It was expected that under the optimum condition both high yield of xylose and high conversion of cellulose could be realized. The xylose yield and cellulose conversion obtained under different pretreatment conditions are shown in Fig. 1.

Fig. 1(a) shows a positive correlation between xylose production and cellulose conversion. The steam explosion severity was varied from 2.706 to 4.516 with a fixed acid concentration of 0.3 wt%. Since xylan is the major component present in the hemicellulose of corncob, the presence of xylose and xylo-oligosaccharide in the water-extraction was used as an indicator of degradation degree of hemicellulose. When the severity level ranged from 2.760 to 3.054, the yield of xylo-oligosaccharide produced by partial breakdown of xylan was appreciable, and the yield of xylose was below 70%. Meanwhile, the cellulose conversion was also low because of incomplete removal of hemicelluose and the xylo-oligosaccharide residue in ASERC which inhibits cellulases (Qing, Yang, & Wyman, 2010). When the severity level was increased beyond 3.938, the xylose yield decreased again because of the degradation of xylose to furfural. Meanwhile, the cellulose conversion also decreased since some of the cellulose converted into hydroxymethylfurfural during the xylose production process (Horn, Nguyen, Westereng, Nilsen, & Eijsink, 2011). Both the xylose yield and cellulose conversion reached a maximum value of 87.19% and 85.40%, respectively, at a severity factor of 3.643.

Fig. 1(b) also presents a positive correlation between xylose production and cellulose conversion. The impregnated acid concentration was varied from 0 wt% to 1.5 wt%, and the steam explosion severity was fixed at 3.643. Under the condition of 0% acid

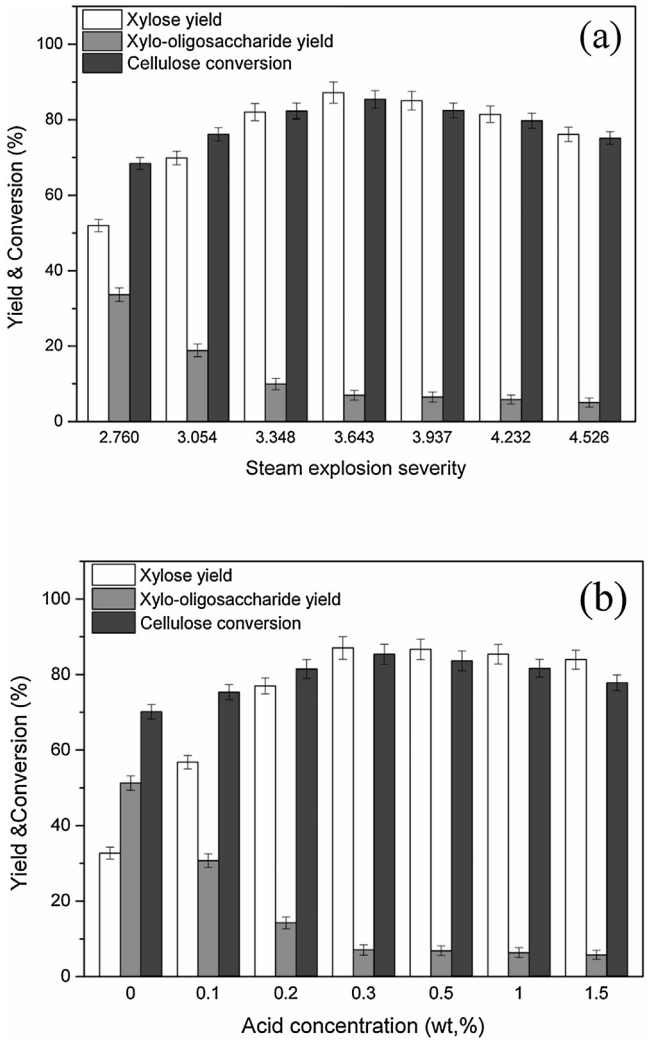

Fig. 1. Effect of acid impregnated steam explosion process on xylose recovery and cellulose conversion. Results of yields of xylose and xylo-oligosaccharide and cellulose conversion were investigated at (a) different steam explosion severity of 2.760–4.526 and impregnated acid concentration of 0.3 wt%; (b) different impregnated acid concentration of 0–1.5 wt% and steam explosion severity of 3.643.

concentration, the obtained residue was called "H_2O impregnated steam explosion residue of corncob (HSERC)". It was found that both the xylose yield and cellulose conversion increased with increasing acid concentration and reached a maximum value of 87.03% and 85.36%, respectively, at an acid concentration of 0.3%. As the acid concentration increased from 0 wt% to 1.5 wt%, the xylo-oligosaccharide yield decreased from 51.25% to 5.75%. The results showed that diluted acid impregnation could promote the conversion of xylo-oligosaccharide to xylose during steam explosion process. A similar trend was found by Kumar that the selectivity of monomeric xylose formation from oligomers increased with the acid concentration during dilute sulfuric acid pretreatment (Kumar & Wyman, 2008). Moreover, appropriate amount of impregnated acid could enhance the enzymatic digestion of corncob residue. With 0.2 to 1.0 wt% acid impregnation, the cellulose conversion of corncob residue was over 80%, which was about 10% higher than that of HSERC. However, when the acid concentration was increased over 1.0 wt%, minor reductions of xylose yield and cellulose conversion were observed because of increased sugar degradation (Chen, Tsai, Lin, Tsai, & Hwang, 2012).

Table 1
Compositions of three hemicellulose hydrolysates.

Methods	Components								
	Sugar products recovery (g/100 g corncob)				Degradation products recovery (g/100 g corncob)				
	Xylose	Xylooligosaccharide	Arabinose	Glucose	Formic acid	Acetic acid	Furfural	5-HMF	Total phenols
AI-SE[a]	27.8 ± 0.53	2.34 ± 0.09	3.76 ± 0.27	3.91 ± 0.21	1.38 ± 0.04	3.15 ± 0.28	0.8 ± 0.04	0.14 ± 0.02	2.15 ± 0.20
HI-SE[b]	10.5 ± 0.23	14.38 ± 0.30	2.42 ± 0.18	1.87 ± 0.15	0.81 ± 0.02	2.26 ± 0.16	0.45 ± 0.02	0.09 ± 0.01	1.5 ± 0.12
AH[c]	28.2 ± 0.42	1.82 ± 0.05	3.73 ± 0.24	3.56 ± 0.16	0.95 ± 0.02	3.09 ± 0.25	0.64 ± 0.03	0.1 ± 0.03	0.92 ± 0.06

[a] AI-SE, hemicellulose hydrolysate obtained from acid impregnated steam explosion.
[b] HI-SE, hemicellulose hydrolysate obtained from H_2O impregnated steam explosion.
[c] AH, hemicellulose hydrolysate obtained from acid hydrolysis.

Table 2
Compositions and enzymatic hydrolysis of three corncob residues.

	Components (%)				Enzymatic hydrolysis			
	Cellulose	Hemicellulose	Lignin	Others[d]	Glucose concentration (g/L)	Xylose concentration (g/L)	Cellulose conversion (%)	Total sugar yield (%)[e]
ASERC[a]	64.4 ± 1.8	5.2 ± 0.3	26.5 ± 0.6	2.9 ± 0.1	82.3 ± 2.7	2.1 ± 0.2	85.3 ± 2.7	56.2 ± 2.5
HSERC[b]	58.3 ± 1.6	11.4 ± 0.5	27.3 ± 0.8	2.2 ± 0.2	60.8 ± 1.9	4.9 ± 0.4	69.5 ± 1.9	43.8 ± 1.6
AHRC[c]	62.3 ± 1.7	5.4 ± 0.2	28.9 ± 0.8	2.6 ± 0.2	49.2 ± 1.5	2.2 ± 0.3	52.6 ± 1.5	34.3 ± 1.4

[a] ASERC, acid impregnated steam explosion residue of corncob.
[b] HSERC, H_2O impregnated steam explosion residue of corncob.
[c] AHRC, acid hydrolysis residue of corncob.
[d] Others, neutral detergent solute and ash content.
[e] Total sugar yield is based on the recovered sugars (glucose and xylose) during enzymatic hydrolysis per dry weight of corncob residue.

3.2. Comparative studies of the processes of acid hydrolysis and acid impregnated steam explosion

To reveal the process characteristics of acid impregnated steam explosion, corncob residues recovered after two different processes, acid hydrolysis and acid impregnated steam explosion were used for glucose production via cellulase hydrolysis. Three samples prepared as ASERC, HSERC and AHRC were subjected to structural analysis and hydrolysis. AHRC was prepared under the condition described in Section 2, where a maximum xylose yield was reported before (Dominguez et al., 1997). ASERC was prepared with an acid concentration of 0.3 wt% and a steam explosion severity of 3.643. HSERC was prepared with a steam explosion severity of 3.643.

3.2.1. Effects of the processes on xylose recovery and cellulose conversion

The compositions of different hemicellulose hydrolysates are shown in Table 1. The monosaccharide (xyloase, arobinose and glucose) recovery of acid impregnated steam explosion process was 35.47%, which was similar to that of acid hydrolysis process, 35.49%. However, for the steam explosion process without addition of sulfuric acid, only 14.79% of monosaccharide was obtained, and as a result, the xylooligosaccharide recovery was higher than those two processes. These results suggest that addition of acid could enhance the degradation of hemicellulose and monosaccharide recovery during steam explosion process. Moreover, the amount of phenolic compounds formed during steam explosion process was higher than that formed during acid hydrolysis process. It indicates that steam explosion led to more lignin degradation than acid hydrolysis during xylose production process.

These three corncob residues were hydrolyzed by cellulase under same condition given in Section 2. The composition and results of enzymatic hydrolysis of the three corncob residues are shown in Table 2. For all corncob residues, the content of hemicellulose was much lower than that of untreated corncob (35.8%). Meanwhile, the content of hemicellulose in ASERC and AHRC decreased significantly to approximately 5%, an half of that in HSERC. The lignin content of AHRC was slightly higher than that of ASERC and HSERC.

The kinetics of enzymatic hydrolysis of three corncob residues is shown in Fig. 2. The initial slope indicates that the cellulose conversion rate of ASERC was significantly higher than the others. At 72 h, the maximum cellulose conversion of ASERC was 85.3%, which was 1.2 and 1.6 times higher than that of HSERC and AHRC, respectively. In previous studies, sulfite pretreatment was applied to AHRC to improve enzymatic hydrolysis efficiency (Bu et al., 2012; Cheng et al., 2011a,b). The cellulose conversion of pretreated AHRC at 72 h was however lower than that of the current study. Additionally, a separate pretreatment step increases the overall operation cost, which makes the acid impregnated steam explosion process for xylose production and cellulose conversion more promising.

3.2.2. Impact of biomass crystallinity on its digestibility

XRD has been frequently used to determine the relative CrI of biomass by comparing the ratio of the main peak intensity

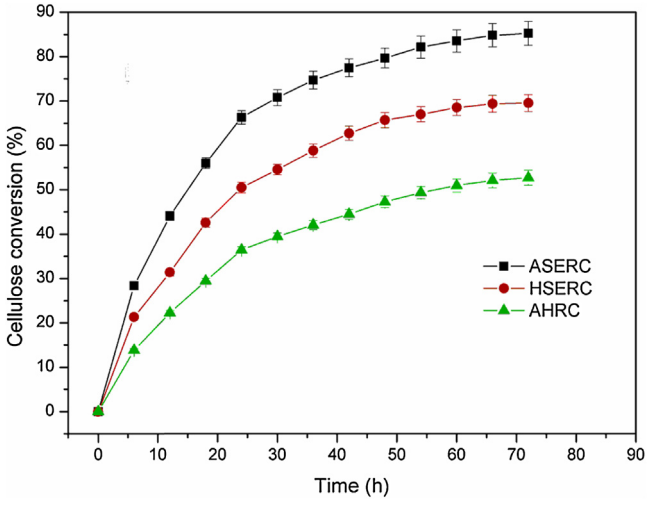

Fig. 2. The kinetics of enzymatic hydrolysis of cellulose conversion of different corncob residues. (a) ASERC, acid impregnated steam explosion residue of corncob; (b) HSERC, H_2O impregnated steam explosion residue of corncob; (c) AHRC, acid hydrolysis residue of corncob

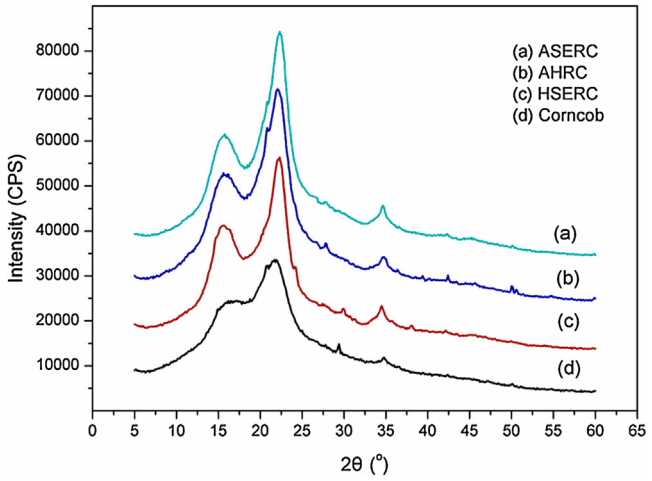

Fig. 3. XRD patterns of different corncob residues and corncob. (a) ASERC, acid impregnated steam explosion residue of corncob; (b) AHRC, acid hydrolysis residue of corncob; (c) HSERC, H_2O impregnated steam explosion residue of corncob; (d) corncob

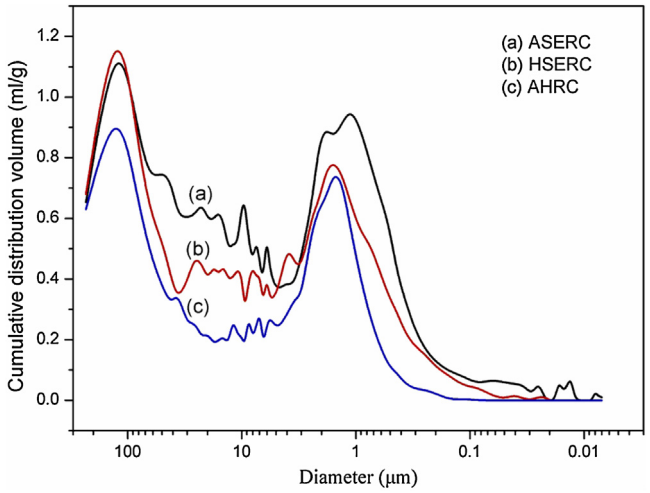

Fig. 4. Pore size distributions of different corncob residues. (a) ASERC, acid impregnated steam explosion residue of corncob; (b) HSERC, H_2O impregnated steam explosion residue of corncob; (c) AHRC, acid hydrolysis residue of corncob

with the intensity at the minimum between the main peak and the secondary peak (Eq. (5)). In native state, cellulose exists as a semi crystalline polymer with crystalline structure of cellulose I (O'sullivan, 1997). With this structure, three peaks are often observed in diffraction patterns (Li et al., 2011). The main peak position varies slightly with species (22.1° for corncob), and it is indicative of the distance between hydrogen-bonded sheets in cellulose I lattice. The second broad peak at about 16° is known to be a composite of several peaks (Cheng et al., 2011a,b). The third small peak at 34.5° corresponds to 1/4 of the length of one cellobiose unit and arises from ordering along the fiber direction (Cheng et al., 2011a,b). Fig. 3 shows that the crystalline structure of the samples was consistent with cellulose I. The CrI value of raw corncob was estimated to be 37.2%. A CrI of 39.2% was reported previously for the raw corncob using a similar method (Sahare, Singh, Laxman, & Rao, 2012). After xylose production, the CrI values of three corncob residues were significantly higher than that of raw corncob due to removal of amorphous hemicellulose. The value of the CrI of ASERC, AHRC and HSERC was 52.1%, 48.8% and 42.5%, respectively. The results in Table 2 suggest that there was no clear correlation between CrI value and enzymatic digestibility.

3.2.3. *Impact of biomass porosity on its digestibility*

In addition to cellulose crystallinity, the structure of pores present in biomass is another important aspect, as the access of cellulases to cellulose in plant cell walls is limited by the available channels for their penetration. Steam explosion pretreatment of hybrid poplar wood resulted in a considerable increase in pore volume, which improved cellulases accessibility to cellulose (Grous, Converse, & Grethlein, 1986). In this work, mercury porosimetry was utilized to determine the pore size distribution (Fig. 4). The pore size distribution in all three samples was characterized by two populations with their sizes centered about 1 and 100 μm, respectively. The pores with a diameter of ~100 μm was probably due to inter-particle space of granules (Vilar, Botelho, & Boaventura, 2007). Except for those pores, the major part of macropores has diameters of about 50–500 nm, and their distribution of ASERC was broader than that of HSERC and AHRC. Moreover, small volumes of mesopores (7 nm $\leq\varphi\leq$50 nm) were also observed on the distribution curves of ASERC, while no pores with diameter lower than 50 nm are found on the distribution curves of HSERC and AHRC. The specific surface area of ASERC, HSERC and AHRC was 6.49, 2.23 and 1.60 m^2/g, respectively. Meanwhile, the specific pore volume of ASERC, HSERC and AHRC was 2.09, 1.80, and 1.23 mL/g, respectively. A representative SEM image of ASERC (Fig. S1) proved that the plant cell walls were shattered by the pretreatment. It is interesting to note micro-holes were present on the surface of AHRC (Fig. S1), which was reported before for samples after the dilute acid pretreatment (Zhang, Li, Li, & Liu, 2011b). The larger surface area and pore volume resulted in higher cellulose conversion, indicating that acid impregnated steam explosion process could not only remove most of hemicellulose, but also improve the porosity, which enhanced the enzymatic digestibility of corncob residue.

3.3. *Correlation between biomass digestibility and physiochemical changes*

Both the dilute acid and steam explosion pretreatment can lead to hemicelluloses removal and lignin's structural alteration (Menon & Rao, 2012). In general, the lignin–carbohydrate complex cannot be completely destructed by steam explosion pretreatment alone (Menon & Rao, 2012). Therefore, the use of acid catalyst during steam explosion increases the recovery of hemicelluloses sugars and improves the enzymatic hydrolysis of the solid residue (Agbor, Cicek, Sparling, Berlin, & Levin, 2011). The different digestibility of the three corncob residues in current study was analyzed by comparing biomass crystallinity, lignin content and porosity. There is no clear correlation between CrI and digestibility in current study. ASERC has higher value of CrI than those of AHRC and HSERC; however it has higher hydrolysis rate and cellulose conversion. On the other hand, HSERC possesses lower value of CrI and shows higher hydrolysis rate and cellulose conversion than those of AHRC. All three samples have comparable lignin content, which is higher than that of untreated corncob due to hemicelluloses removal. An increase in lignin content after dilute acid pretreatment was reported before (Samuel, Pu, Raman, & Ragauskas, 2010). Lignin migration and relocation during dilute acid and hydrothermal pretreatment were also documented in previous studies; it was argued that the lignin redistribution may affect the porosity of the substrate (Samuel et al., 2010). In current work, it is not clear whether lignin relocation took place or not. The specific surface area and pore volume of the three samples correlate nicely with their digestibility. It is thus concluded that the higher accessibility of ASERC determines its efficient enzymatic hydrolysis among the three samples.

4. Conclusions

Acid impregnated steam explosion processes were investigated for the production of xylose and subsequent cellulose conversion from corncob. It was found that higher xylose recovery was accompanied with higher cellulose conversion. The maximum cellulose conversion of ASERC reached 85.3%, which was 1.6 times higher than that of AHRC. In this work, the porosity of biomass samples was found to be the most important factor affecting their enzymatic digestibility, while the other two commonly studied parameters: cellulose crystallinity and delignification were less significant. The results suggest that the contributions of substrate-related properties such as crystallinity, lignifications and porosity to biomass recalcitrance could vary among different types of biomass and pretreatment conditions. The integrated process of acid impregnated steam explosion and enzymatic hydrolysis could be applied to the commercial utilization of corncob for large scale xylose and glucose production.

Acknowledgments

The authors are indebted to Novozymes for the gift of the enzyme cocktail used in this work. This work was supported by the National High-tech Research and Development Program (2011AA02A207 & 2014AA021903).

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at http://dx.doi.org/10.1016/j.carbpol.2014.07.051.

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