Production of Xylose from Diluted Sulfuric Acid Hydrolysis of Wheat Straw

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The objectives of this study were to generate fermentable xylose by sulfuric acid hydrolysis of wheat straw and investigate the effect of hemicellulose removal on the physical and chemical properties of the unhydrolyzed solid residue (USR). Different reaction conditions, including concentration of sulfuric acid (COS), temperature, and time, were tested for their effects on the yield of xylose and the USR. The ideal hydrolysis conditions for xylose production were 0.5% of COS at 140 °C for 90 min, with the xylose yield of 0.185 g/g wheat straw. The Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analyses showed that the acid hydrolysis only caused slight changes in the functional groups and the crystal form of wheat straw at mild conditions, while higher temperature or higher COS exacerbated these changes.

Keywords: Wheat straw hemicellulose; Sulfuric acid; Xylose; Crystallinity

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INTRODUCTION

Lignocellulosic materials, which primarily consist of cellulose, hemicellulose, and lignin, are some of the most abundant natural resources on the earth (Zhang *et al.* 2010). They are the most promising sources of renewable raw material for various biotechnological processes, including the manufacture of chemicals and biofuels, because of their low economic cost and high availability (Himmel *et al.* 2007). In particular, hemicellulose might have the potential for industrial utilization if it could be converted into sugars efficiently (Aristidou and Penttilä 2000; Lange 2007). The main composition of hemicellulose is C5 sugars and C6 sugars (particularly xylose, mannose, arabinose, and glucose) (Ying *et al.* 2013). These sugars and their derivatives are important materials for industrial application (Krogell *et al.* 2013). For instance, xylitol produced from hydrogenation or fermentation of xylose is largely applied as an additive in the food and pharmaceutical industries (Sarrouh and Silva 2010).

Lignocellulosic materials have resilient structures that consist of a carbohydrate polymer matrix, mainly cellulose and hemicelluloses. They are cross-linked and strongly bound to lignin. This structural complexity, defined as biomass recalcitrance, severely restricts enzymatic and microbial accessibility. To hydrolyze hemicellulose for obtaining sugars, a great deal of energy is required to break down the C-O bonds interactions between adjacent sugar molecules (Dhepe and Sahu 2010). Various methods have been used for hemicellulose hydrolysis, such as mineral acids, alkali, enzymes, and subcritical/supercritical water (Yu *et al.* 2007; Wang *et al.* 2013). Although some methods increase the solubility of hemicelluloses by breaking down the sugar chains to a lower polymerization degree, most sugar units remain oligosaccharides, which are non-fermentable sugars. Interestingly, acid hydrolysis can break down the ether bond in the polysaccharide chain, resulting in sugar monomers. Meanwhile, in the process of hydrolysis, the surface structure of raw material and crystallinity of cellulose will be changed. It had been reported that hydrolysis could destroy the structure of raw material and increase accessibility of the enzyme by increasing porosity or surface area of the lignocellulosic materials (Camesasca *et al.* 2015; Barisik *et al.* 2016). Although dilute acid hydrolysis has been widely adopted in the production of xylose and furfural and in pretreating lignocellulose for cellulose hydrolysis (Liu *et al.* 2012; Encinas-Soto *et al.* 2016), there is less known about the change rule of xylose production as well as the surface structure and crystallinity of the unhydrolyzed solid residue (USR) during hydrolysis. Improved understanding about such issues can contribute to more efficient comprehensive utilization of lignocellulosic biomass.

In this study, the xylose yield and USR were systematically investigated during the dilute sulfuric acid hydrolysis of wheat straw by using single factor experiments. The effects of reaction conditions, such as the temperature, concentration of sulfuric acid (COS), and reaction time, on the yields of xylose and characteristics of USR were examined. This work optimizes the reaction conditions for obtaining the high yield of xylose and also elucidates the changes of surface structure and crystallinity of USR during hydrolysis.

EXPERIMENTAL

Materials

The wheat straw was obtained from Shandong Tranlin Group (Shandong Province, China). The wheat straw was air-dried and milled using a pulverizer (FZ102, Taisite, Tianjin, China) and screened through a mesh. Even small particle size could favor xylose production according to Khullar *et al.* (2013). While considering the energy consumption during the preparation process, particles in the size range from 0.45 mm to 1.35 mm (20-meshes to 60-meshes) were used in the experiments. The H_2SO_4 was provided by Yantai Far East Fine Chemical Co., Ltd. (Laiyang, China).

Acid Hydrolysis

A total of 5.0 g (dry weight) of wheat straw was mixed with 60 mL of sulfuric acid media (0.5 wt.% to 4 wt.% of H_2SO_4) in a pressure bottle. The pressure bottle was heated at different temperatures (80 °C to 160 °C) for a given duration (30 min to 120 min) under magnetic stirring (Kumar and Wyman 2008; Akpinar *et al.* 2009). After the reaction, the pressure bottle was cooled with ice. The liquid fraction was separated by filtration, and the unhydrolyzed solid residue (USR) was washed with 300 mL of distilled water to remove residual sulfuric acid and then air dried at 105 °C for 12 h.

Characterization

Analysis of chemical composition

The chemical composition of the wheat straw and USR was determined by the NREL standard method (Sluiter *et al*. 2008), and the results are shown in Table 1.

Table 1. Chemical Composition of the Raw Material and USR

Composition	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Benzene - Alcohol Extracts (%)
Wheat straw	43.2	24.4	20.8	6.6	4.8
USR*	59.1	2.4	30.7	7.8	-
* The USR was acquired at 140℃, 90 min with 0.5 % of COS.					

Analysis of monosaccharides

The monosaccharides were determined by ion chromatography (ICS-5000$^+$, Thermo Fisher Scientific, Shanghai, China) with a pulsed amperometric detector (HPAEC-PAD). The liquid sample was first processed to secondary hydrolysis with 4% H_2SO_4 at 120 °C for 60 min as previously described (Sluiter *et al.* 2008). Monosaccharides were measured by using a Dionex CarboPac PA20 carbohydrate column (3 mm × 150 mm, Thermo Fisher Scientific, Shanghai, China) with a guard column. The elution program for monosaccharides profile of hydrolysate was 2 mmol NaOH at 30 °C with a flow rate of 0.4 mL/min.

Analysis of oligosaccharides

The oligosaccharides were determined by the same ICS-5000$^+$ system while using CarboPac PA100 carbohydrate column with a guard column (Thermo Fisher Scientific, Shanghai, China). Elution program for oligosaccharides was 50 to 450 mmol NaOAc in 100 mmol NaOH from 0 to 40 min at a flow rate of 1.0 mL/min and 25°C. The column was reconditioned using 100 mmol NaOH for 15 min after each analysis (Wang *et al.* 2015).

Analysis of the crystallinity

The X-ray diffraction (XRD) patterns of the samples were examined with a diffractometer (D2 Phaser diffractometer, Bruker, Billerica, MA, USA). The XRD patterns were calibrated with the (012) diffraction of a-alumina, which was applied as an internal standard.

Using the Lorentzian function, crystallization peaks corresponding to 101, 10, 002, and 040 planes were separated from the diffraction peak curve; the amorphous crystallization peak corresponded to the maximum value of the trough between the 101 and 002 planes. The crystallinity was calculated according to Eq. 1,

$$X_d/\% = \left[1 - \frac{S_a}{S_a + S_{cr}}\right] \times 100 \qquad (1)$$

where X_d is the crystallinity of wheat straw sample, S_a and S_{cr} are the area of amorphous peaks and crystalline peaks respectively.

Fourier transform infrared spectroscopy (FTIR) analysis

FTIR spectra of the wheat straw and the USR were recorded on a VERTEX70 FT-IR spectrophotometer (Bruker, Billerica, America) over 4000 cm^{-1} to 400 cm^{-1} using a KBr disc containing 1% finely ground samples.

Scanning electron microscope (SEM) analysis

The surface textures of the wheat straw and the USR were observed using a QUANTA 200 scanning electron microscope (SEM, FEI Instrument Co., NED, Hillsboro, OR, USA).

RESULTS AND DISCUSSION

Effects of Conditions on the Xylose Yield

To investigate hemicellulose hydrolysis, the experiments were performed in a temperature range of 80 °C to 160 °C with the concentration of sulfuric acid (COS) of 0.5% to 4%. Figure 1 shows the effect of the COS and the reaction temperature on the xylose yield at a constant time of 60 min. In all experimental conditions, the xylose yield increased with reaction temperature in the initial stages of hydrolysis, and then it declined with further increases of temperature after the xylose yield reached its maximum value at around 120 °C to 140 °C. This result might be explained by the conversion of xylose into furfural at higher temperature (Zhong *et al.* 2015). Moreover, as the COS increased, an obvious increase of the xylose yield was detected from the hemicellulose hydrolysis at temperatures of 80 °C to 120 °C. This result is consistent with the studies of Lenihan *et al.* (2010) and Hernández *et al*. (2012). However, the trend was reversed at temperatures of 140 °C to 160 °C. This might be due to the higher COS, which accelerated the depolymerization of xylose (Arslan *et al.* 2012). Hence, within a certain range, increasing the temperature and COS favors the increase in xylose yield. But excessive temperature and COS could aggravate the conversion of xylose to furfural, which eventually reduced the xylose yield. Figure 1 also shows that even 4.0% of COS at 120 °C had the highest xylose yield. While considering the cost of sulfuric acid and the subsequent neutralization process, 0.5% of COS at 140 °C was recommended, which had a compatible yield.

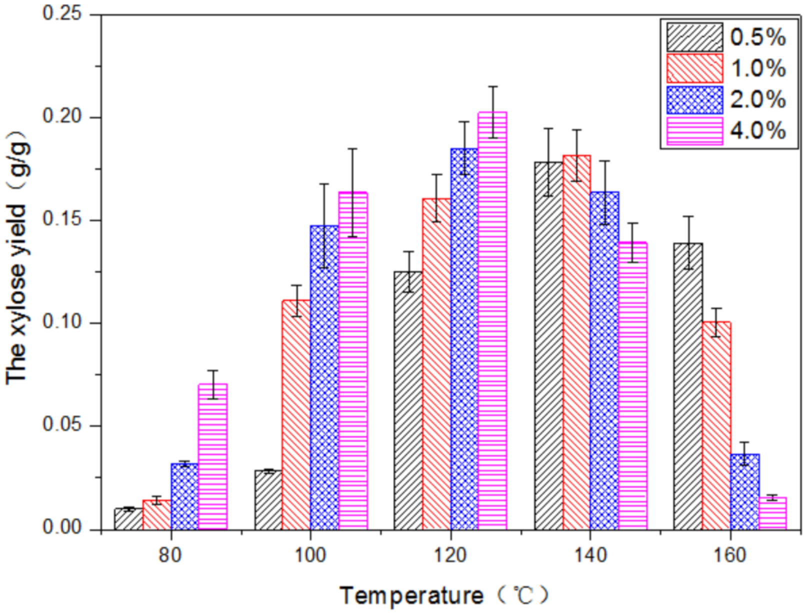


Fig. 1. The effect of reaction temperature and COS on the xylose yield at 60 min

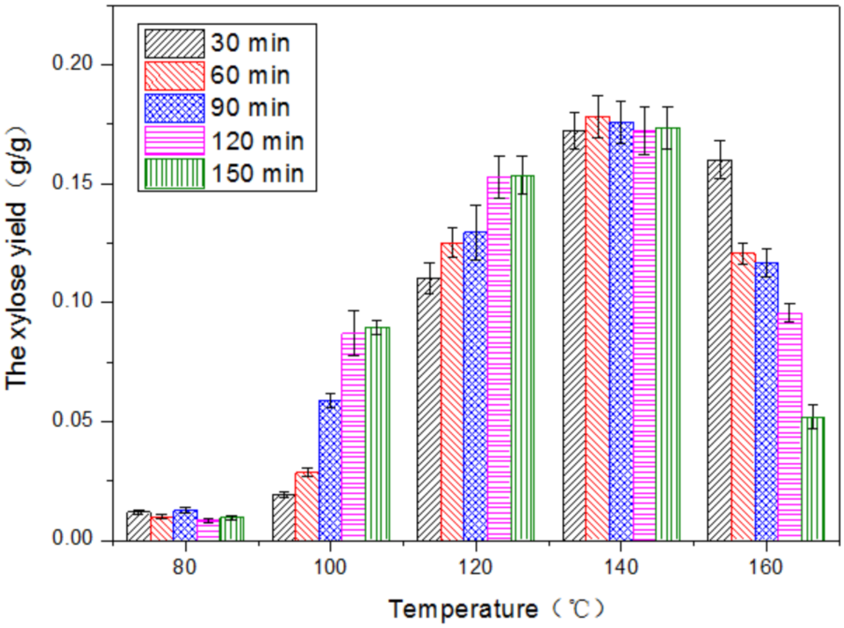


Fig. 2. The effect of reaction temperature and time on the xylose yield with 0.5 % of COS

The effect of reaction temperature and time on the xylose yield at a constant COS (0.5%) is illustrated in Fig. 2. Hydrolysis at 80 °C could only obtain the xylose yield of 0.01 g/g, and the xylose yield showed no changes with extended reaction time. As the temperature increased to 120 °C, the xylose yield had a noticeable increase as time increased, which was similar to previously published results (Camesasca *et al.* 2015). This may be because the main chain of the hemicellulose began to degrade when the reaction temperature exceeded a certain threshold, but at lower temperatures, only the branches of hemicellulose were degraded. At 140 °C, the xylose yield did not increase with extended time. This result might be attributed to the full degradation of hemicellulose at the relatively high temperature of 140 °C, and with increased time, no more hemicellulose was converted into sugar. However, the xylose yield decreased with longer reaction times when the reaction temperature was 160 °C, which showed that 160 °C was severe for production of xylose from diluted sulfuric acid hydrolysis of wheat straw. Increasing time at excessive reaction temperature will result in drastic degradation of xylose to form furfural. Therefore, the temperature and time had important impacts on the hemicellulose degradation (Neureiter *et al.* 2002). Notably, the xylose yield did not increase with extended time at 140 °C. However, the oligosaccharides analysis showed that the oligosaccharides content decreased with extended time, and it was almost absent in hydrolysate after 90 min as shown in Table 2. The absence of oligosaccharides and high content of monosaccharides will benefit for the subsequent fermentation. Therefore, when the COS was optimized at 0.5%, 90 min was the optimized time at 140 °C to extract xylose by acid hydrolysis, under which the xylose yield was 0.185 g/g of wheat straw, *i.e.* 91.5% of xylose based on the content of raw material could be achieved, with the corresponding xylose concentration was 15.4 mg/mL hydrolysate. This is higher than hydrolysis of hemicellulose by solid acid (Zhong *et al*. 2015) and similar with that by organic acid (Barisik *et al* 2016).

Table 2. The Content of Oligosaccharide in Hydrolysate Obtained at 140 °C and 0.5% of COS with Varied Time

Times	Xylobiose (mg/g)	Xylotriose (mg/g)	Xylotetraose (mg/g)	Xylopentaose (mg/g)
30 min	12.3	8.9	2.3	-*
60 min	7.2	4.3	-	-
90 min	0.2	-	-	-
120 min	-	-	-	-

* Not detected.

Effects of Conditions on the Glucose Yield

Figure 3 shows the effect of the reaction temperature and the COS on the glucose yield at a constant time (60 min). Under all experimental conditions, the glucose yield increased with the increasing temperature and COS. Especially at 160 °C, the glucose yield increased rapidly when the COS was 2.0% and 4.0%. As reported by Arslan *et al.* (2012), the glucose mainly originated from heteropolymers of hemicellulosic fractions at a temperature less than 120 °C, while more intense reaction conditions resulted in cellulose hydrolysis. Figure 4 shows the effect of reaction temperature and time on the glucose yield with a constant COS (0.5%). The glucose yield did not change by further increasing time at each given temperature. This result suggests that the reaction temperature and the COS have important effects on the glucose yield, while time has little effect (Liu *et al.* 2012). Compared with xylose production, therefore, the glucose production required more intense reaction conditions.

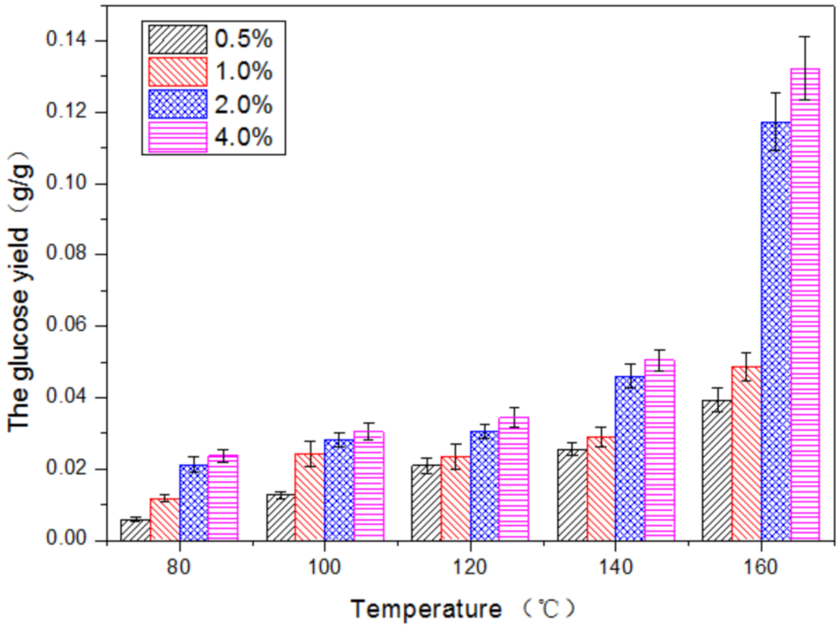


Fig. 3. The effect of reaction temperature and COS on the glucose yield at 60 min

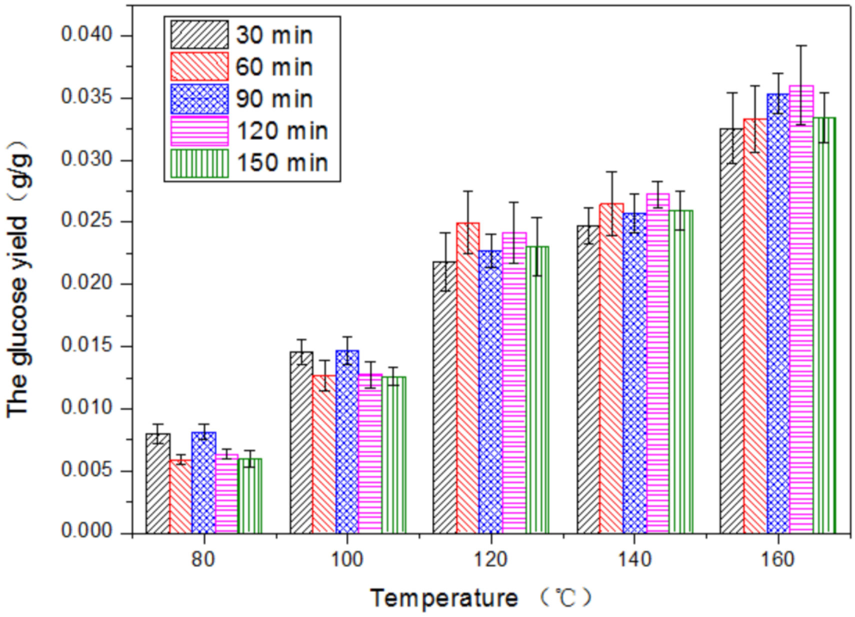


Fig. 4. The effect of reaction temperature and time on the glucose yield with 0.5 % of COS

Compositional Analysis of USR

The chemical composition of the USR acquired under the ideal hydrolysis condition (0.5 % of COS at 140 °C for 90 min) and the original wheat straw are shown in Table 1. It could be seen that after diluted sulfuric acid hydrolysis, as the hemicellulose was hydrolyzed to produce xylose, the USR then mainly consisted of cellulose and lignin. Because a large amount of hemicellulose was dissolved during hydrolysis, the cellulose and lignin content in the USR increased remarkably compared with that of the original wheat straw.

XRD Analysis

XRD was conducted to evaluate the crystalline behavior of wheat straw and the USR. As shown in Fig. 5, the diffractograms of wheat straw and the USR obtained at 120 °C for 60 min with 4.0 % of sulfuric acid hydrolysis exhibited similar patterns, indicating that the crystallization structure of cellulose did not change during hydrolysis. The crystallinity index of wheat straw and the USR at different hydrolysis conditions was calculated based on Eq. 1, and the results are presented in Tables 2 to 4.

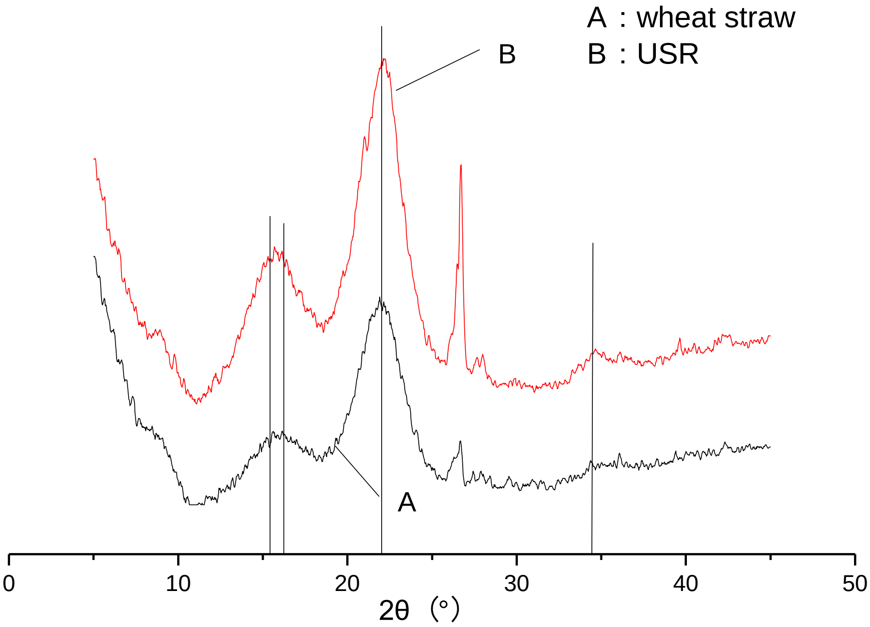


Fig. 5. XRD pattern of wheat straw and the USR obtained at 120 °C for 60 min with 4.0 % of sulfuric acid

Table 2 lists the crystallinity indices of wheat straw and the USR obtained under 0.5% of COS at 120 °C with varied times. The crystallinity index of the USR was higher than that of wheat straw, and it had a slight increase with extended time. This result could be interpreted as the removal of a substantial part of the amorphous noncellulosic constituents (hemicellulose and lignin) during hydrolysis (Ibbett *et al.* 2008). Table 3 shows the crystallinity indices of wheat straw and the USR obtained with various COS. The crystallinity index of the USR decreased with increasing COS, which might indicate that the crystalline domains were destroyed in higher COS. Table 4 indicates the crystallinity indices of wheat straw and the USR obtained at different temperatures. The crystallinity index of the USR decreased slightly with increasing temperature. Thus, the temperature and the COS had important impacts on destroying the crystalline region.

Table 2. Crystalline Coefficients of Raw Material and the USR at Different Times

Time (min)	Raw	30	60	90	120
Crystallization coefficient (%)	70.2	75.9	76.2	76.3	76.8
* The temperature was 120 °C, and the COS was 0.5%					

Table 3. Crystalline Coefficients of Raw Material and the USR at Different COS

COS (%)	Raw	0.5	1.0	2.0	4.0
Crystallization coefficient (%)	70.2	76.6	73.4	69.6	68.7
* The temperature was 120 °C, and the time was 60 min					

Table 4. Crystalline Coefficients of Raw Material and the USR at Different Temperature

Temperature (°C)	Raw	100	120	140
Crystallization coefficient (%)	70.2	73.3	69.6	69.1
* The COS was 2.0%, and the time was 60 min				

FTIR Analysis

FTIR spectroscopy is an appropriate technique to monitor the chemical transformation of the samples in response to different treatments. The FTIR spectra of the tested samples are shown in Figs. 6 and 7. All samples exhibited two major absorbance regions, including regions of 500 cm^{-1} to 1700 cm^{-1} and 2800 cm^{-1} to 3500 cm^{-1}. The peak at 3344 cm^{-1} is ascribed to OH stretching vibration (Liao *et al.* 2015). The peak at 1735 cm^{-1} is associated with the presence of the acetyl and/or uronic ester groups of the hemicellulose (Sain and Panthapulakkal 2006).

Figure 6 shows the FTIR spectra of wheat straw and the USR obtained with different concentration of sulfuric acid at 120 °C for 60 min. The peak intensity of the USR at 1735 cm^{-1} was reduced as the COS increased, and the peak disappeared when the COS was 4.0%, indicating that most hemicellulose was hydrolyzed effectively during the sulfuric acid treatment. The findings were similar to the previous study by Kargarzadeh *et al*. (2012). The FTIR spectra of wheat straw and the USR obtained at different times by using 0.5% of COS at 120 °C are shown in Fig. 7. The peak of the USR at 1735 cm^{-1} was weaker than that of wheat straw, but it did not disappear with extended time, which meant that part of the hemicellulose could not be completely hydrolyzed by 0.5% of sulfuric acid at 120 °C, even with the time extended to 120 min.

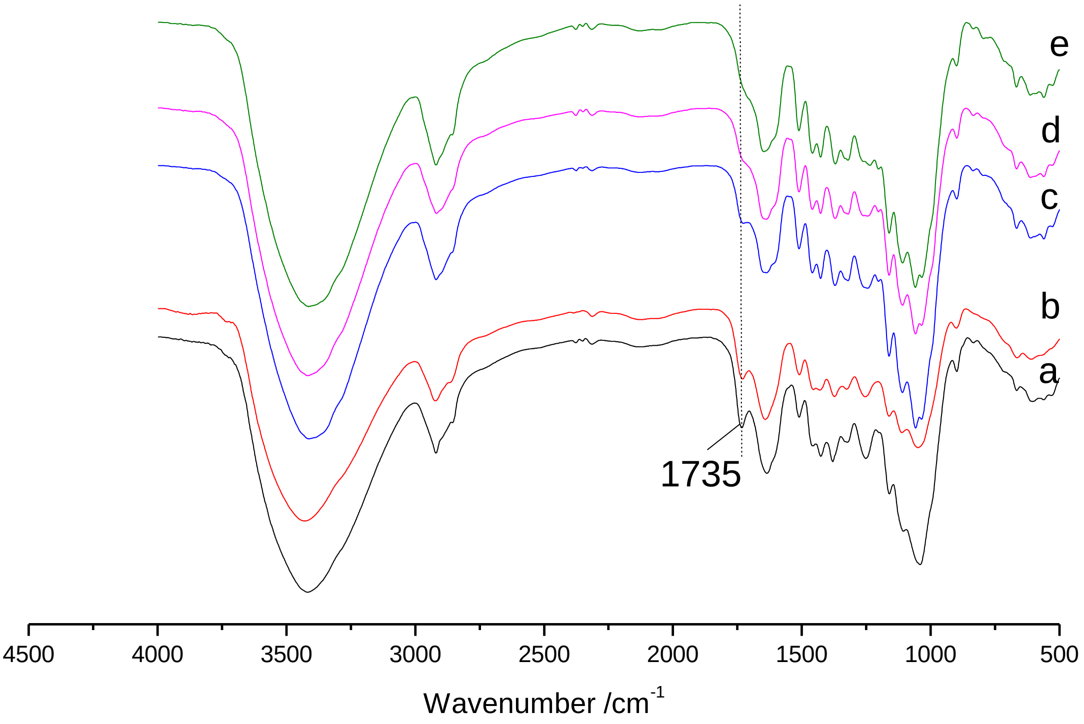


Fig. 6. The FTIR spectrum of (a) wheat straw and the USR obtained with (b) 0.5%, (c) 1.0%, (d) 2.0%, and (e) 4.0% of sulfuric acid at 120 °C for 60 min

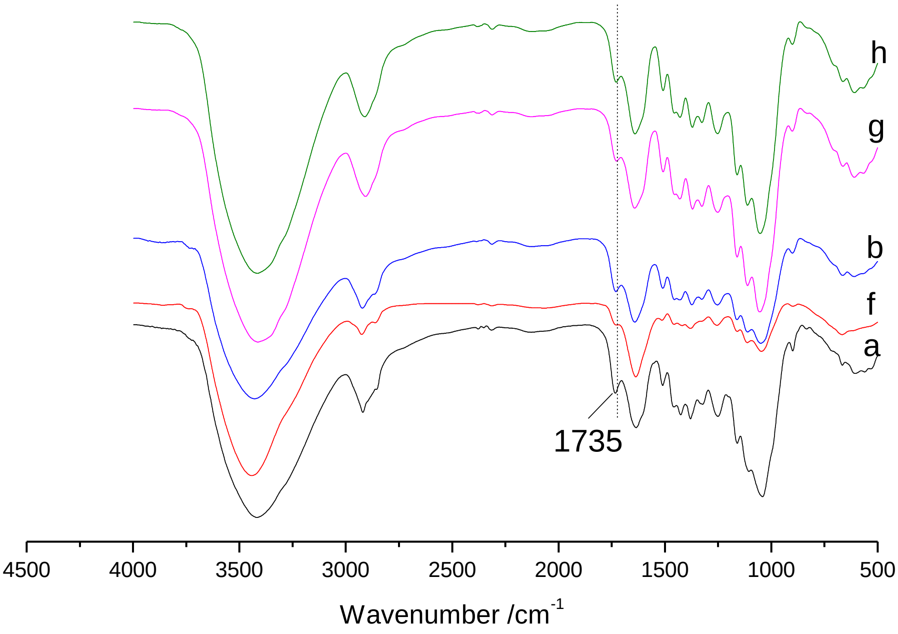
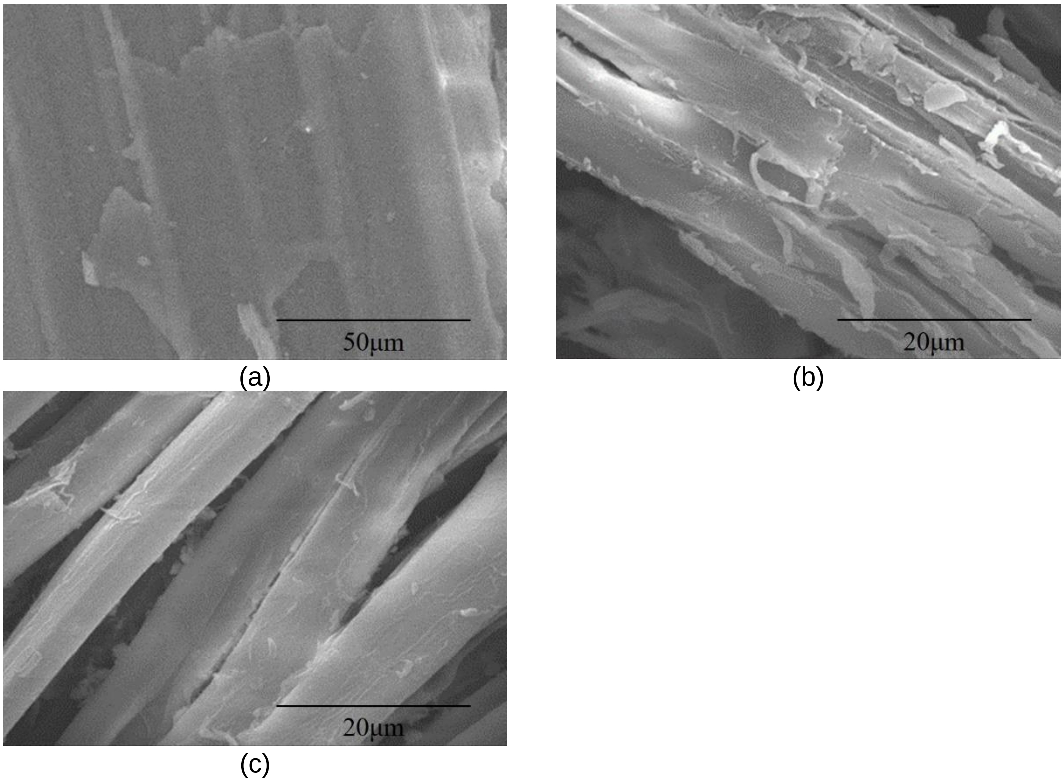


Fig. 7. The FTIR spectrum of (a) wheat straw and the USR obtained with 0.5 % of sulfuric acid at 120 °C for (f) 30 min, (b) 60 min, (g) 90 min, and (h) 120 min

SEM Analysis

The morphology of the samples was investigated by scanning electron microscopy, as shown in Fig. 8. The surface of wheat straw was smooth and tight. The USR was broken after acid-hydrolysis, and the wraps around the fibers had almost disappeared when the COS was 4.0%, suggesting that hemicellulose was almost completely hydrolyzed.



(a) (b) (c)

Fig. 8. SEM micrographs of (a) wheat straw, (b) the USR obtained with 0.5 % of COS at 120 °C for 60 min, and (c) the USR obtained with 4.0 % of COS at 120 °C for 60 min

CONCLUSIONS

1. The main factors affecting the xylose yield from the acid hydrolysis of wheat straw were temperature and acid concentration, followed by reaction time. The ideal hydrolysis conditions for xylose production were 0.5% of concentration of sulfuric acid (COS) at 140 °C for 90 min, under which the xylose yield was 0.185 g/g of wheat straw, *i.e.* 91.5% of the initial xylose in raw material could be obtained.
2. The XRD and FTIR analysis showed that acid hydrolysis did not greatly change the crystal form and the functional groups of wheat straw at relatively mild conditions, while higher temperature and COS reduced the crystallinity index of the USR.

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