



The thermophilic (55 °C) microaerobic pretreatment of corn straw for anaerobic digestion



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HIGHLIGHTS

- Effects of thermophilic microaerobic pretreatment (TMP) were assessed.
- TMP obviously improved the methane yields of corn straw.
- TMP accelerated the hydrolysis and reduced the lag-phase time of fermentation.
- Structural changes could be the ground for the improvement of methane yields.

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ABSTRACT

Microaerobic process has been proven to be an alternative pretreatment for the anaerobic digestion (AD) process in several studies. In this study, the effect of thermophilic microaerobic pretreatment (TMP) on the AD of corn straw was investigated. Results indicated that TMP process obviously improved the methane yield. The maximum methane yield was obtained at the oxygen loads of 5 ml/g VS_{substrate}, which was 16.24% higher than that of untreated group. The modified first order equation analysis showed the TMP process not only accelerated the hydrolysis rates but also reduced the lag-phase time of AD process. The structural characterization analysis showed cellulosic structures of corn straw were partly disrupted during TMP process. The crystallinity indexes were also decreased. In addition, large or destroyed pores and substantial structural disruption were observed after pretreatment. The results showed that TMP is an efficient pretreatment method for the AD of corn straw.

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1. Introduction

As one of the largest agricultural countries in the world, China has abundant biomass resource. Approximately 216 million metric tons of corn straws are generated annually in China and more than half of the crop straw produced remains unused (Zhong et al., 2011). The unsuited dispose (such as burn) of corn straw not only wastes of resource but also leads to environmental problems. Anaerobic digestion (AD) has been widely employed as an efficient means of treating organic wastes (Jang et al., 2013), which is an ideal way for comprehensive utilization of corn straw. Generally, hydrolysis is regarded as the rate-limiting step in AD of cellulosic substrate such as corn straw. One way of improving the perfor-

mance of AD is to promote the hydrolysis of organic matter by pretreatment of the substrate (Cesaro and Belgiorno, 2014; Climent et al., 2007; Ferrer et al., 2008; Shen et al., 2014). Thermal, chemical, biological and mechanical process, as well as their combinations have been studied as possible pretreatment to accelerate substrate hydrolysis (Wagner et al., 2013). However, these conventional pretreatment methods often lead to increased capital costs due to the additional energy or chemicals required (Lim and Wang, 2013). Chemical treatment requires large amounts of chemicals to maintain the reaction conditions. In addition, inhibitory and biologically non-degradable compounds can be generated after thermal and chemical treatment (Jang et al., 2014).

Recently studies have demonstrated that hydrolysis can be enhanced by introducing limited amounts of oxygen (or air) directly into the anaerobic digester or during a pretreatment step (Ramos and Fdz-Polanco, 2013). Compared to other pretreatment means, microaerobic pretreatment is more environmentally

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friendly and economically, it just needs a small amount of oxygen (or air). Earlier studies have shown that microaerobic treatment could reduce the formation of toxic metabolites (e.g. lactic acid and ethanol) as well as promote the synthesis of certain lipids required for the stability of anaerobes cell membrane (Lim and Wang, 2013). There were some evidences that under microaerobic condition the activities of cellulose and protease hydrolytic enzymes are higher (Charles et al., 2009). Microaerobic pretreatment has been conducted in several studies. When treating sisal pulp, Mshandete et al. (2005) reported that compared to the sisal pulp without pretreatment, a nine hours of microaerobic pretreatment prior to AD of sisal pulp demonstrated a 26% higher methane yield. Botheju et al. (2010b) reported a 30–55% increase in methane yield in the range of oxygenation loads of 0–16% (%O₂ of COD input) when treating starch. A 10–21% higher methane yield was reported by Lim and Wang (2013) when treating the compound of brown water and food waste with an oxygen load of 37.5 ml O₂-L_R⁻¹·d⁻¹.

The thermophilic hydrolytic bacterial population has also been used to accelerate the hydrolysis process, mainly at a temperature of around 55 °C (Dumas et al., 2010). In addition, under thermophilic digestion the organic matter removal is more efficient (Ferrer et al., 2008), the rate of hydrolysis is faster and the production of volatile fatty acids (VFAs) is also more efficient (Kim et al., 2010). In practice, a thermophilic acidogenesis may start-up with mesophilic sludge inoculum because the thermophilic process is less prevalent in field-scale applications (Kim et al., 2010).

Previous studies have reported the effect of microaerobic pretreatment on the anaerobic digestion of sisal pulp, sludge, food waste, starch, cellulose etc. However, it is less known about the performance of AD of corn straw using the microaerobic pretreatment under thermophilic (55 °C) condition. The objective of this study is to investigate the effect of the combination of microaerobic and thermophilic (55 °C) pretreatment on the anaerobic digestion of corn straw. In addition, the structural changes of substrate during microaerobic pretreatment have never been reported in previous studies. Thus, the structures of pretreated and untreated corn straw were also characterized by scanning electron microscopy (SEM), FTIR-ART spectroscopic and XRD.

2. Methods

2.1. Substrate and inoculums

The air-dried corn straw with moisture contents of 7–8% was obtained from corn field in Pingdu (Shandong province). The corn straw was chopped to a particle size of approximately 0.5 cm and kept at ambient temperature (20–25 °C). Its total solid (TS) content is 92.44%, including 93.44% of volatile solid (VS) (TS, VS were determined according to standard methods (APHA, 2006)). The C/N and C/H ratio are 28.97 and 7.73, respectively (the element contents of corn straw were quantified with an elemental analyzer). The proportions of cellulose, hemicelluloses and lignin are 45.43%, 22.73% and 10.79% (% of TS), respectively (cellulose, hemicelluloses and lignin were determined according to the method as Goering and van Soest (1970) described).

Inoculums used in this study was biogas slurry, which was collected from a 500 m³ size of biogas plant (Qingdao, Shandong province, China), and stored in a refrigerator at 4 °C until use. The TS and VS contents of the biogas slurry are 6.64% and 70.62% (% of TS), respectively.

2.2. Thermophilic microaerobic pretreatment

The thermophilic microaerobic pretreatment was performed in duplicates, 5.77 g corn straw and 20 ml inoculums were mixed in

bottles, and then deionized water was added to reach a total volume of 0.1 L. Each bottle was flushed with N₂ for 5 min to replace the air, and then the bottles were closed with rubber stoppers. Then 0, 25, 50, 100, 150, 200 ml oxygen at atmospheric pressure was injected to each group with a syringe to reach the oxygen loads of 0, 5, 10, 20, 30, 40 ml/g VS_{substrate} (marked as R1, R2, R3, R4, R5, R6). The bottles were placed in a shaking water bath at 55 °C with 130 rpm. The oxygen levels were measured by a gas chromatograph (SP 6890, Shandong Lunan Inc., China) every 4 h until the oxygen was consumed completely.

2.3. Batch anaerobic digestion tests

Batch mesophilic (37 °C) anaerobic digestion tests were also performed in duplicates. The substrates after thermophilic microaerobic pretreatment were added with another 20 ml inoculums to reach TS_{inoculum}/TS_{substrate} of 0.5, then deionized water was added to reach a total volume of 0.2 L. Another 2 bottles (marked as A) were also employed to find out the biogas yield from corn straw without pretreatment. The bottles were placed in a shaking water bath at 37 °C with 130 rpm. In this stage, the biogas volume was measured using water replacement method. Biogas composition was measured by a gas chromatograph (SP 6890, Shandong Lunan Inc., China), equipped with a Porapak Q stainless steel column (180 cm long, 3 mm outer diameter) and a thermal conductivity detector. The temperatures of the injector, detector, and oven were 50, 100 and 80 °C, respectively. The carrier gas was argon. VFA was measured by a gas chromatograph (Varian 450-GC, USA), equipped with Innowax column (30 m × 0.25 mm × 0.25 μm) and flame ionization detectors. The operating temperatures were 220 °C, 220 °C and 50 °C, for injection port, detector and oven, respectively.

2.4. Model for data fit

In this study, the modified first order equation described as Diaz et al. (2011) was used to estimate the hydrolysis constant (d⁻¹), which was written as:

$$P(t) = P_{\infty} \exp[1 - \exp(-k_H(t - L_p))]$$

Where $P(t)$ is the cumulative methane yields (ml/g VS), P_{∞} is the total methane yields potential (ml/g VS), k_H is the hydrolysis constant (d⁻¹), L_p is the lag-phase time (d), t is the elapsed time (d).

2.5. Physical and chemical analysis of solid fraction corn straw

2.5.1. FTIR-ATR spectroscopic analysis

A spectrum One FTIR system (The Nicolet iN10 IR Microscope) with a universal ATR (Attenuated Total Reflection) accessory was used to investigate chemical changes in corn straw. The samples were pressed uniformly against the germanium (Ge) surface using a spring-loaded anvil, and mid-IR spectra were obtained by averaging 16 scans from 4000 to 700 cm⁻¹ at 2 cm⁻¹ resolution. Baseline and ATR corrections for penetration depth and frequency variations were applied using Spectrum One software supplied with the equipment.

2.5.2. Crystallinity measurements

Wide angle X-ray diffraction (Bruker D8 Advance) was used to determine the crystallinity of untreated and pretreated corn straw at 40 kV and 40 mA in the scanning angle of 5–60° 2θ at a scanning speed of 1.0°/min. the crystallinity of the corn straw was determined on the basis of the crystallinity index (CrI), which is a measure of crystalline material in biomass and is given by:

$$\text{CrI} = [(I_{002} - I_m)/I_{002}] \times 100$$

where I_{002} is the intensity for the crystalline portion of biomass at about $2\theta = 22$ and I_m is the peak for the amorphous portion at about $2\theta = 16.2$ (Dhabhai et al., 2013; Kumar et al., 2009).

2.5.3. Scanning electron microscopy (SEM)

SEM pictures were taken of untreated and pretreated corn straw using Hitachi S-4800 scanning microscope at 3 kV. Samples were mounted onto aluminium pin stubs and sputter-coated with a thin layer of gold.

3. Results and discussion

3.1. The effects of TMP process on the performance of anaerobic digestion of corn straw

3.1.1. Effect on methane yield

The ultimate aim of pretreatment is to increase the methane yields and accelerate the AD process. The cumulative methane

yields were calculated from the first day of AD until no obvious gas productions were detected. At the initial stage of fermentation, the methane yields increased sharply. The methane yields reached the highest at about the fifth day. The daily methane yields of pretreated groups had always been greater than that of untreated group during the AD process. The cumulative methane yields during AD are shown in Fig. 1. The maximum cumulative methane yield was achieved at the oxygen load of 5 ml/g $VS_{\text{substrate}}$, which was 16.24% higher than that of untreated group. It was followed by the oxygen loads of 10, 20, 30, 40 and 0 ml/g $VS_{\text{substrate}}$, they were 15.85%, 10.46%, 8.49%, 5.96% and 5.21% higher than that of untreated group, respectively. When using the oxygen as the pretreatment method, the amount of oxygen used is very important, since the excessive oxygen may inhibit the activities of methane-forming bacteria and decrease the methane yield (Xu et al., 2014). On the other hand, the excessive oxygen may lead to the oxidation of readily available substrates or methane consumption by aerobic methanotrophs. Decreased amounts of methane

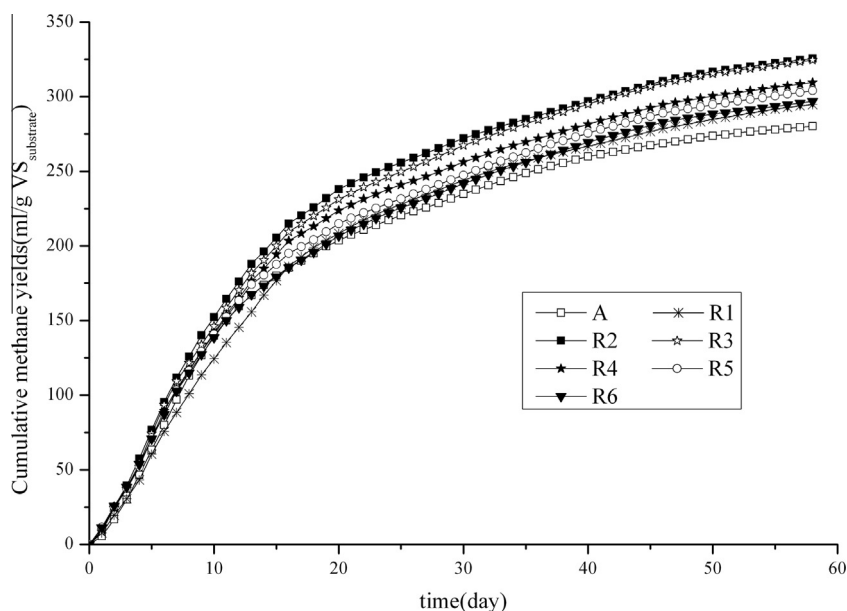


Fig. 1. Cumulative methane yields during anaerobic digestion of corn straw.

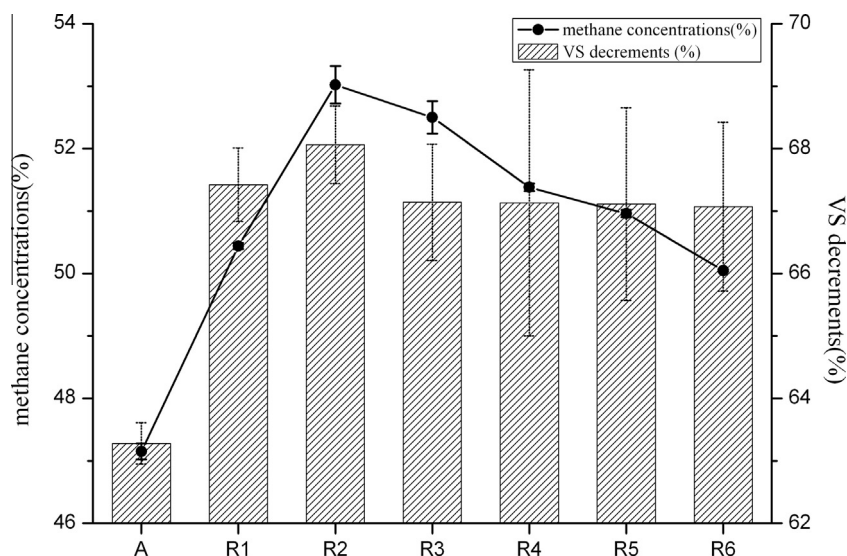


Fig. 2. Methane concentrations and VS decrements.

generation under increased aeration conditions has also been reported by [Botheju et al. \(2010a\)](#), who reported a linear reduction in methane generation under increasing aeration conditions within the oxygenation range 0–10.1% (as % of feed COD).

3.1.2. Effects on methane concentrations and VS decrements

The average methane concentrations and VS decrements are shown in [Fig. 2](#). The average methane concentrations and VS decrements were calculated as following:

$$\text{The average methane concentrations} = \frac{\text{Total cumulative methane yields}}{\text{Total cumulative biogas yields}}$$

$$\text{VS decrements} = \frac{\text{Initial VS (VS of substrate)} - \text{Remaining VS (VS of substrate)}}{\text{Initial VS (VS of substrate)}}$$

The average methane concentrations of pretreated groups were higher than that of untreated group, which would be beneficial for the followed biogas upgrading process of biogas plant. The VS decrement was also an important parameter to evaluate the AD process. A higher VS decrement means that more substrate was utilized during AD process. It would be beneficial for the fermentation residue reduction. All of the VS decrements in pretreated groups were greater than that of untreated group. The highest VS decrement was getting at the oxygen loads of 5 ml/g $\text{VS}_{\text{substrate}}$, which was 11.1% higher than that of untreated group. The present work showed that the TMP process can enhance the substrate utilization efficiency.

Table 1
Parameters of first order equation fitting experimental data.

Group	P_{∞} (ml/g VS)	k_H (d ⁻¹)	L_p (d)	R^2
A	281.65 ± 1.86	0.066 ± 0.002	0.601 ± 0.143	0.995
R1	300.35 ± 1.67	0.059 ± 0.001	0.739 ± 0.111	0.997
R2	327.03 ± 1.81	0.064 ± 0.001	0.469 ± 0.121	0.997
R3	328.88 ± 1.95	0.060 ± 0.001	0.433 ± 0.125	0.997
R4	311.68 ± 1.86	0.062 ± 0.001	0.433 ± 0.128	0.996
R5	307.29 ± 2.12	0.059 ± 0.001	0.240 ± 0.149	0.996
R6	302.38 ± 1.98	0.057 ± 0.001	0.081 ± 0.142	0.996

3.1.3. Mathematical model analysis

The data fitted to the modified first order equation is shown in [Table 1](#). Results showed that the whole cumulative methane yields of pretreated groups were higher than that of untreated group, this result was quiet coincident with experimental data. All the R^2 of the fitted data were above 0.995, this meant these fitted data might explain 99.5% (and over) of total variations in the data. The parameter of hydrolysis constant was lower in pretreated groups compared with the untreated group, which stood for a faster translation of complex structure to simple organic compounds in pretreated groups. The result was in agreement with the results reported by [Zhu et al. \(2009\)](#), [Lim and Wang \(2013\)](#). Lag-phase time interpreted as the time elapsed until a significant production

of methane was found in the batch assays. In this study, the highest lag-phase time was obtained in R1 and followed by A. The groups after TMP had lower lag-phase time. In the pretreated groups, the lag-phase time was lower with the rise in oxygen loads. Thus, it can be concluded that the thermophilic microaerobic pretreatment reduced the lag-phase time of AD, which was coinciding with the found of [Diaz et al. \(2011\)](#) in treating cellulose.

3.2. VFA concentrations and compositions

VFAs are the main products of the acidogenesis of organic matter and used as substrate for the methanation process, which means that a certain amount of VFAs is necessary for the biogas production ([Li et al., 2014](#)). The compositions and concentrations of VFAs are shown in [Fig. 3](#) (all of the VFAs data were obtained after 1-day anaerobic digestion). The highest concentration of VFAs was attained at the oxygen loads of 5 ml/g $\text{VS}_{\text{substrate}}$, which was 424.96 mg/L and 63.25% higher than that of untreated group. The VFAs concentration of R1, R3, R4, R5 and R6 was 316.09, 332.64, 337.10, 324.84 and 315.64 mg/L, respectively. They were 21.48%, 27.84%, 29.55%, 24.84% and 21.31% higher than that of untreated group, respectively. Acetate and butyrate are the desirable VFAs in the acidogenic phase of AD to maximize methane production.

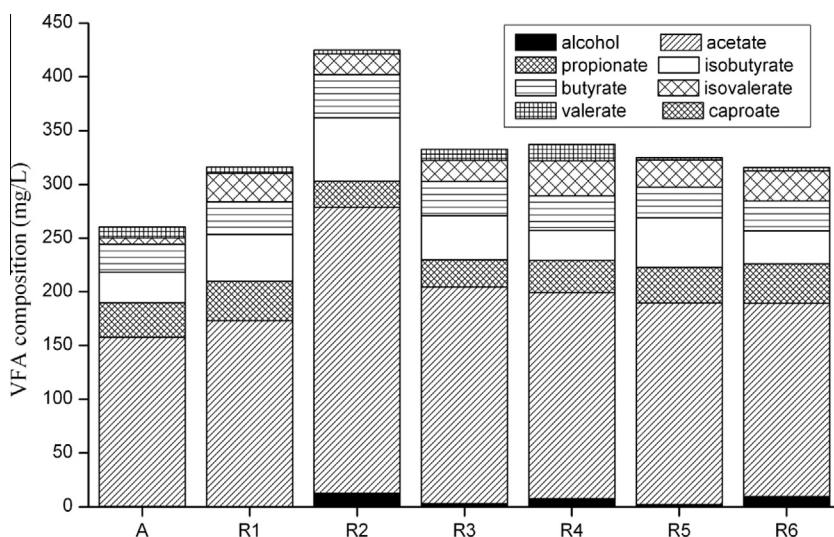


Fig. 3. Concentrations and composition of VFAs after TMP process.

In contrast, accumulations of propionate and valerate would be limiting factors for methane generation (Xu et al., 2014). The proportion of acetate in VFAs exceeded 55% and the proportion of propionate after TMP process was lower than that of untreated group. This would be beneficial for subsequent methanogenesis. The higher VFAs concentrations in pretreated groups could be due to the higher hydrolysis under microaerobic condition. The results obtained in this study were in accordance with what Lim and Wang (2013) reported when treating brown water and food waste. The increasing oxygen loads during TMP did not lead to the increase in the VFAs concentration. This could ascribe to that the exceeded oxygen consumed some readily available organic substrates (e.g. volatile fatty acids (VFAs)) during TMP.

3.3. Structural characterization analysis of corn straw

3.3.1. FT-IR analysis

In this study, the chemical structure of untreated and pretreated corn straw was analyzed by ATR FT-IR spectroscopy in the region of 700–4000 cm^{-1} (Supplemental material, Fig. S1). The peak near 3348 cm^{-1} and 2900 cm^{-1} represented wagging vibration in C–H and the O–H stretching of the hydrogen bonds of cellulose (Ang et al., 2012; Hsu et al., 2010; Kumar et al., 2009). The intensity of this two absorption peaks in the pretreated corn straw was smaller than untreated corn straw, which means that the crystalline cellulose in the corn straw was partly disrupted by the thermophilic microaerobic pretreatment. The band at 1595 cm^{-1} is attributed to aromatic ring stretching, which is associated with lignin removal. The intensity of this absorption peak was lower after pretreatment, which indicating that part of lignin was destroyed during pretreatment. The band at 1245 cm^{-1} is attributed to C–O adsorption and has been proposed to be associated with the acetyl group in hemicelluloses. The intensity of this absorption peak was also lower after pretreatment, which was due to the disruption of hemicelluloses during the pretreatment process. Spectra of all the corn straw showed the strongest absorption bands at about 1059 cm^{-1} . This band corresponds to the C–O stretching vibration in both cellulose/hemicellulose and lignin (Ang et al., 2012), and it explains the lignocellulosic nature of corn straw. The intensity of this absorption peak after pretreatment was smaller than that of untreated sample, which indicates that the lignocellulosic component had been degraded partially in pretreated groups. The intensity of the 900 cm^{-1} is very sensitive to the amount of crystalline versus amorphous structure of cellulose (Proniewicz et al., 2001). The intensity of this band after pretreatment was lower than untreated corn straw, which reflected higher amount of disordered structure being obtained after pretreatment.

3.3.2. XRD analysis

It is broadly accepted that highly crystalline cellulose is less accessible to cellulase attack than amorphous cellulose. Therefore, crystallinity negatively affects the efficiency of enzyme contact with cellulose (Zhu et al., 2008). In this study, the crystallinity of corn straw was analyzed by XRD (Supplemental material,

Fig. S2). The results are shown in Table 2. The XRD analysis result was quiet confirmed with the results of ATR FT-IR analysis reported previously. After thermophilic microaerobic pretreatment, the crystallinities of pretreated corn straw were lower than that of untreated group. The lower crystallinity index indicates a higher amount of amorphous cellulose present in the lignocellulosic substrate (Kuo and Lee, 2009), such it is more feasible for cellulase to breakdown the cellulose. Hence it could be concluded that thermophilic microaerobic pretreatment is beneficial for the anaerobic digestion of corn straw.

3.3.3. SEM analysis

To investigate the microscopic change of corn straw, SEM images of untreated and pretreated corn straw samples were taken at 500 and 3000 magnification (Supplemental material, Fig. S3). The untreated corn straw was intact and had a rather smooth surface. The pores of the untreated corn straw were smaller and less than the pretreated samples. After thermophilic microaerobic pretreatment, large pores and substantial surface disruption were observed and many pores structure were also demolished. The results were coincided with the ATR FI-IR analysis. Part of the lignocellulosic component of corn straw was destroyed during pretreatment process, which may enhance the cellulase contact area during enzymolysis of lignocellulosic component. Thus, it may be beneficial for the subsequent anaerobic digestion of corn straw.

4. Conclusions

TMP is an efficient pretreatment means for the anaerobic digestion of corn straw, which obviously improved the methane yields. The maximum methane yield was obtained at the oxygen loads of 5 $\text{ml/g VS}_{\text{substrate}}$, which was 325.7 ml/g VS and 16.24% higher than that of untreated group. TMP process not only reduced the lag-phase time but also improved the hydrolysis efficiency. The cellulosic structures and pores of corn straw were partly disrupted and the crystallinity index was also decreased during TMP process. The structural change during the TMP process may be the grounds for the improvement of methane yields in pretreated groups.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.biortech.2014.10.072>.

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Table 2

Crystallinity indexes of untreated and pretreated corn straw.

Groups	Crystallinity index	Relative change (% relative to A)
A	37.6	0
R1	37.6	0
R2	35.9	−4.6
R3	34.7	−7.7
R4	34.5	−8.2
R5	34.3	−8.9
R6	32.9	−12.5

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