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Effects of ionic liquid/water mixture pretreatment on the composition, the structure and the enzymatic hydrolysis of corn stalk

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ABSTRACT

Recently, pretreatment of lignocellulose by ionic liquids have attracted considerable attention due to their excellent pretreatment effects and their environmental sustainable properties. However, because of the high cost and viscosity, their industrial application are restricted. In this study, water was introduced to 1-butyl-3-methylimidazolium tetrafluoroborate ([Bmim]BF_4) to reduce both the viscosity and the cost of using pure ionic liquid. The [Bmim]BF_4/water system was proved to be effective for improving the enzymatic hydrolysis of corn stalk, and the enzymatic hydrolysis efficiency (81.68%) obtained was much higher than that found with the pure ionic liquid under the same conditions (14.46%). The findings obtained from the characterization studies were in accordance with the enhancement of the hydrolysis of the lignocellulosic biomass. After pretreated by [Bmim]BF_4-H_2O, the formation of a porous structure, destruction of the lignin-polysaccharide interactions, and deacetylation of hemicellulose were achieved as suggested by SEM, FTIR and ^{13}C NMR, respectively, but the crystalline structure was not damaged indicated by XRD results.

1. Introduction

Lignocellulose, the most earth-abundant biomass resource (David and Ragauskas, 2010), is an ideal substitute for fossil resources due to its low cost, reproducibility and potential to be converted to biofuels and other value-added chemicals. Lignocellulose is composed of three main biopolymers, cellulose, hemicellulose, and lignin. The rigid structure and the complex chemical composition of lignocellulose contributes to strong resistance to biological and chemical degradation (Agbor et al., 2011; Sannigrahi et al., 2011). Many methods have been used to break the tight structure to make cellulose available to enzymatic hydrolysis. Chemical pretreatments, such as dilute acid hydrolysis, alkaline and organosolv process, are the most promising method currently but with the disadvantages of environmental pollution and production of degradation products (Fu et al., 2010). Therefore, developing novel green and effective pretreatment techniques remains an ongoing and meaningful endeavor (Pu et al., 2007; Zhu et al., 2008).

Recently, the lignocellulose pretreatment using ionic liquids as promising green solvents has attracted considerable attention. Ionic liquids are organic salts that usually melt below 100 °C. Ionic liquids reveal many advantages like thermal and chemical stability, low vapor pressure and easy to reuse (Fort et al., 2007). In addition, ionic liquids have excellent dissolvability of polar and non-polar organic materials, and have been frequently used in material separations and chemical conversions (Lee and Lee, 2005) Previous studies show that ionic liquid possess excellent solubility for cellulose and lignocellulose (Carvalho et al., 2015; Hyvärinen et al., 2014). Therefore, the application of ionic liquids in biomass dissolution and pretreatment has attracted considerable attention.

Cellulose has shown excellent solubility in several ionic liquids including 1-allyl-3-methylimidazolium chloride ([Amim]Cl), 1-butyl-3-methylimidazolium chloride ([Bmim] Cl), and 1-ethyl-3-methylimidazolium acetate ([Emim] [Ac]) (Remsing et al., 2008; Xu et al., 2010; Zavrel et al., 2009). Typical ionic liquid are applied for dissolving cellulose containing an anion of chloride, acetate or alkylphosphonate, which can generate strong hydrogen bonds with cellulose (Zhao et al., 2009). Additionally, several research groups studied the dissolution of lignin in ionic liquids and found that [Bmim]$MeSO_4$ was the most effective to dissolve lignin, while [Bmim]BF_4 and [Bmim]PF_6 were unsuitable to dissolve lignin, and the solubility of lignin was affected by anions types (Pu et al., 2007). More recently, the dissolution of lignocellulose such as bagasse, rice straw and pine wood in ionic liquid was studied (Fort et al., 2007; Kilpeläinen et al., 2007; Lee et al., 2009; Pu et al., 2007). After dissolution, adding anti solvent like water,

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Table 1
The constituents and enzymatic hydrolysis efficiency of corn stalk before and after pretreatment.

pretreatment	Residue recovery (%)	Composition (%)						Enzymatic hydrolysis efficiency (%)
		Cellulose	Lignin	Xylan	Arabinan	Acetyl groups	Ash	
Raw	100	30.89 ± 0.60	23.49 ± 0.25	15.77 ± 0.47	2.25 ± 0.21	1.97 ± 0.11	3.89 ± 0.33	19.56 ± 0.33
Water	58.92 ± 1.14	50.73 ± 0.77	29.54 ± 0.21	14.17 ± 0.36	2.64 ± 0.18	2.82 ± 0.16	1.90 ± 0.23	46.88 ± 0.32
10%IL	54.48 ± 1.18	53.94 ± 0.56	31.84 ± 0.26	5.49 ± 0.19	3.76 ± 0.23	2.74 ± 0.19	2.32 ± 0.19	49.54 ± 0.24
30%IL	52.17 ± 1.15	55.77 ± 0.33	29.52 ± 0.19	4.38 ± 0.32	3.24 ± 0.19	1.87 ± 0.16	2.47 ± 0.20	66.52 ± 0.51
50%IL	52.08 ± 1.23	56.62 ± 0.81	26.73 ± 0.23	7.29 ± 0.28	4.01 ± 0.26	1.04 ± 0.09	2.35 ± 0.15	81.68 ± 0.47
70%IL	56.99 ± 1.25	52.58 ± 0.45	20.82 ± 0.25	15.32 ± 0.23	3.98 ± 0.30	3.03 ± 0.21	2.02 ± 0.12	43.34 ± 0.34
90%IL	68.33 ± 2.19	44.38 ± 0.32	19.28 ± 0.31	19.88 ± 0.29	3.57 ± 0.27	2.98 ± 0.23	2.38 ± 0.20	27.19 ± 0.38
Pure IL	80.39 ± 2.21	43.15 ± 0.27	22.39 ± 0.27	21.68 ± 0.34	2.86 ± 0.23	2.32 ± 0.15	1.67 ± 0.15	14.46 ± 0.47

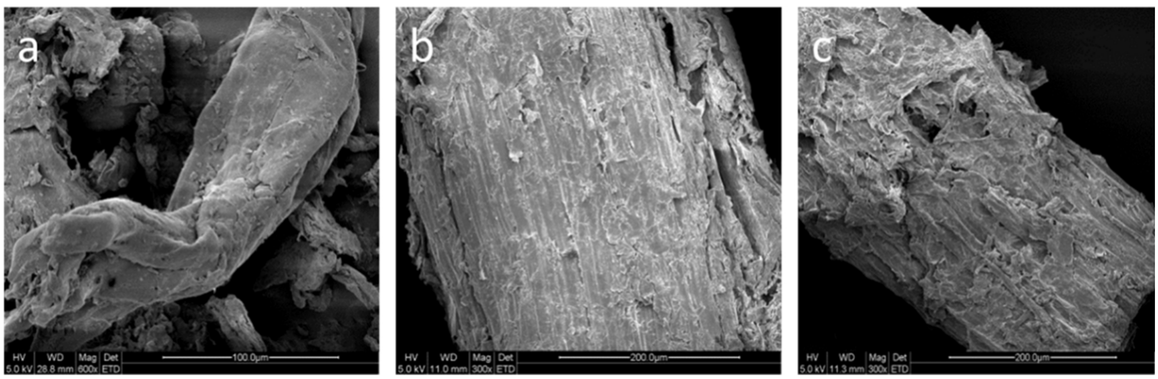

Fig. 1. SEM images of corn stalk before and after pretreatment (1200 ×). Note a: Raw sample; b: $[Bmim]BF_4$; c: $[Bmim]BF_4$-H_2O.

Table 2
The specific surface area of corn stalk before and after pretreatment.

Sample	Specific surface area (m^2/g)
Raw	0.614
$[Bmim]BF_4$	0.754
$[Bmim]BF_4$-H_2O	2.717

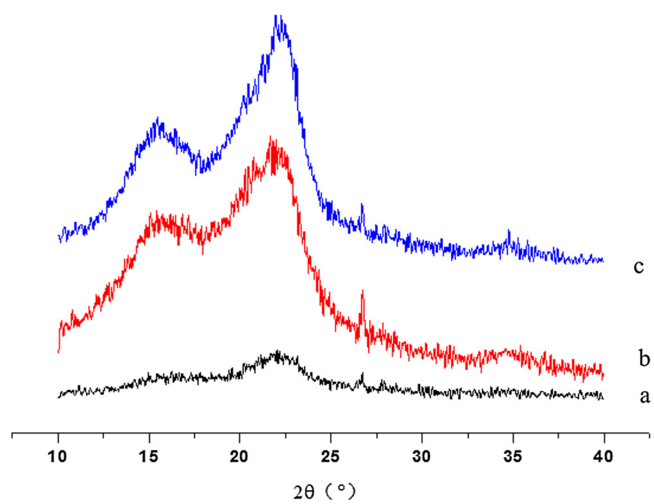

Fig. 2. XRD images of corn stalk before and after pretreatment.

Table 3
The cellulose crystallinity index of corn stalk before and after pretreatment.

Sample	Cellulose crystallinity index (CrI)
Raw	37
$[Bmim]BF_4$	59
$[Bmim]BF_4$-H_2O	65

acetone and acetonitrile to the reaction system can regenerate the biomass. The regenerated biomass showed more amorphous and porous structure than native biomass, and lignin was partially extracted during pretreatment, which resulted in higher enzymatic efficiency than native biomass (Lee et al., 2009; Sun et al., 2009).

Although ionic liquids are promising in the pretreatment of lignocellulose, the requirement of large amounts of expensive ionic liquids and the extremely viscous of solution during pretreatment seriously affect the industrial application of ionic liquids (Fu and Mazza, 2011). To address these problems, other solvents were added to the ionic liquid to reduce both the amount of the ionic liquid and the viscosity of reaction system. Previous studies suggested that the $[Bmim]BF_4$ based ionic liquid showed no obvious effect on both the lignocellulose structure and the improvement of enzymatic hydrolysis (Pu et al., 2007; Zhu et al., 2006). However, during our previous screening experiment of ionic liquid mixing system, it was found that the enzymatic hydrolysis efficiency could be greatly improved when 50% (w/w) of water was added to $[Bmim] BF_4$. Additionally, the mechanism was studied by comparing the composition, the structure and the physicochemical properties of the samples prepared via pretreatment with both pure $[Bmim]BF_4$ and $[Bmim]BF_4$-H_2O mixtures.

2. Materials and methods

2.1. Materials and reagents

Corn stalks were provided by Dacheng Corn Development Co., Ltd., (Changchun, China). After milling, the fraction between 40 and 60 meshes was selected and dried for 24 h at 45 °C. $[Bmim]BF_4$ was purchased from Chengjie Chemical Co., Ltd. (Shanghai, China). Cellulase (ATCC 26921, 45FPU ml^{-1}) was obtained from Sigma-Aldrich. Sulfuric acid, calcium carbonate, citric acid and sodium citrate are all analytically pure reagents and are derived from Sinopharm Group Chemical

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