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Intensification of sodium hydroxide pretreatment of corn stalk using magnetic field in a fluidic system

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HIGHLIGHTS

- This method shortened CS NaOH pretreatment time by 33.3%.
- Sugar release rose by 23 mg in 2–6 Hz and fell by 18 mg from 1 g dry CS in 6–10 Hz.
- High flow rate is conducive to the increase of sugar yield.
- RSM was used to optimize key processing parameters for maximal sugar yield.
- Optimal conditions were 6.71 Hz, 0.50 L/min and 1.02% NaOH for CS pretreatment.

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ABSTRACT

To promote NaOH pretreatment of corn stalk (CS), a continuous processing system uniting magnetic field and millimeter-scaled channel flow was established. First, four comparative pretreatments were conducted: (I) CS was pretreated with NaOH under traditional agitation; (II) CS was pretreated with NaOH in a flowing state inside the millimeter-scaled channel; (III) CS was pretreated with NaOH in a flowing state and under a static magnetic field; or (IV) CS was pretreated with NaOH in a flowing state and under a rotating magnetic field. By comparison, the highest pentose (121.22 mg/g dry CS) and hexose (287.04 mg/g dry CS) yields were obtained in the shortest pretreatment time with Pretreatment IV (8 h). Accordingly, the key parameters of Pretreatment IV were optimized as 6.71 Hz frequency, 0.50 L/min flow rate, and 1.02% NaOH concentration. Under these conditions, 439.24 mg sugars were released by 1 g dry CS during pretreatment and enzymatic hydrolysis.

1. Introduction

In regards to the rapidly growing global demand for energy, and the climate change caused by overdependence on fossil fuels, it is a crucial – and urgent – endeavor to find innovative, sustainable, and environmentally friendly alternative fuel resources (Cheng et al., 2011; Wahlströem and Suurnäekki, 2015). Lignocellulosic biomass is considered a tremendously promising feedstock for this purpose due to its renewability, biodegradability, carbon neutrality, abundance, and local accessibility in most areas (Perlack et al., 2005; Ren et al., 2008; Bhaumik and Dhepe, 2015). Among various lignocellulosic biomasses, corn stalk (CS) represents ready accessibility and simple utilization due to the fact that corn is ubiquitously planted across the globe. To this effect, CS has attracted extensive research interest as far as its potential for biofuel conversion.

The CS cell wall is composed of close-linking lignin, hemicellulose, and cellulose. This compact network structure is resistant to biomass saccharification (Kumar et al., 2009; Ding et al., 2012). In order to alter the complex structure, pretreatment is necessary prior to enzyme hydrolysis and subsequent fermentation of released sugars (Correia et al., 2013; Li et al., 2014). Pretreatments increase the degree of porosity, enlarge the interaction area between the enzyme and cellulose substrates, and ultimately improve the CS saccharification rate (Alvira et al., 2010; Zhang et al., 2013).

Alkali or alkali-based pretreatments have been extensively studied due to the effective lignin removal and sugar conversion enhancement (Xu et al., 2010; Zhang et al., 2013; Kim et al., 2013). As reported by Xu et al. (2010), 77.8% lignin in 'Performer' switchgrass was removed after treating the biomass with 1% sodium hydroxide at 50 °C for 12 h. This delignification increases

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the amount of total reducing sugar yield released during enzymatic hydrolysis 2.78 times as compared to the untreated switchgrass. According to Macdonald et al. (1983), the saccharification of CS was improved by 32% after a NaOH pretreatment at 2% concentration and 150 °C for 5 min. Zheng et al. (2009) pretreated the CS containing 88% moisture with 2%–6% NaOH at 20 °C for 0–5 day in order to promote anaerobic biogasification; they found the biogas production increased by 72.9% at the optimal conditions of 2% NaOH dosage and 3-day residence time.

NaOH, as a strong alkali, is commonly used in alkali or alkali-based pretreatment accompanying mild conditions. On one hand, it can break lignin-carbohydrate bonds, unfold recalcitrant structures, enhance the enzyme hydrolysis, and finally improve the saccharification of biomass (Cabrera-Rodríquez et al., 2012; Sambusiti et al., 2013; Yan et al., 2015). On the other hand, NaOH pretreatment is typically applied under conditions of low concentration (commonly 0.5–4%), low temperature (about 50–55 °C), or brief treatment duration (nearly 2–6 h) (Cabrera-Rodríquez et al., 2012; Wang et al., 2012; Xu et al., 2010). However, it is difficult to achieve high biomass saccharification rate under all three of these conditions. In this study, we attempted to establish an alternative pretreatment method to improve pretreatment efficiency at mild conditions.

As inspired by the Hall Effect theory, we designed a continuous processing system that unites magnetic field and millimeter-scaled channel flow. According to the Hall Effect, when an electroconductive medium passes through alternating magnetic flux, electrolyte ions are inclined to depart their initial movement trajectory and move in the orientation perpendicular to both the magnetic and the induced electric fields. The induced voltage and the orientational ions' motion are defined as Hall voltage and Hall current, respectively (Sawaya et al., 2002). Within the chemical industry, reactions of raw reagents typically take place in a long channel; the reaction dynamic is significantly influenced by the diameter and geometry of the channel. In the proper range, narrowed diameters or irregular geometries improve the reaction rate by accelerating mass transport, heat transfer and fortifying the effective collision of reactive particles (Aamo et al., 2003; Liu et al., 2000; Kim et al., 2013). The magnetic field also exerts a synergistic effect with the channel flow system to enhance reactive dynamic. According to Kenis et al. (2000), when an 2-Tesla magnetic field is applied in the photochemical reaction of 10-methylphenothiazine and 2,2,6,6-tetramethylpiperidin-1-oxyl-linke d electron acceptors in a fluidic 2-propanol system, the intermediate products (free ions) are produced at a higher rate.

Charge-carrying ions such as Na^+ and OH^- existing in NaOH-CS solution can respond to the applied magnetic field, which may influence the interaction between flowing free ions and the lignocellulose substrate. To the best of our knowledge, there has been little research on the application of magnetic field combined with millimeter-scaled channel flow in enhancing the alkali pretreatment of biomass materials so far. Accordingly, we established a continuous processing system uniting magnetic field and millimeter-scaled channel flow to enhance the NaOH pretreatment of CS. Initially, four different NaOH pretreatments of CS were compared in terms of pentose and hexose yields as an indicator to determine the most effective of the four. The key parameters (rotational frequency of the rotating magnetic field, flow rate of reactant fluid, and NaOH concentration) of the selected pretreatment method were then optimized for maximum saccharification of CS via response surface methodology (RSM).

2. Materials and methods

2.1. CS preparation

CS was provided by Changchun Dacheng Corn Development Co., Ltd. (Jilin, China). The biomass was sieved to collect fractions with an average particle diameter of 0.4 mm. The collected portions were then oven-dried at 50 °C for 48 h, sealed in polyethylene bags, and stored in a desiccator until pretreatment. The compositional analysis of CS was conducted according to the NREL method (Sluiter et al., 2005a,b, Sluiter et al., 2012a, Sluiter et al., 2012b). As determined, 100 g of the dry bases contain 35.8 g cellulose, 19.5 g hemicellulose, 25.4 g lignin, 6.9 g ash, 8.1 g water-extractives, and 2.9 g ethanol-extractives, respectively.

2.2. Experimental setup

Fig. 1 shows a diagram of the uniting continuous processing system. As described in detail in our previous study (Jin et al., 2016), the system is comprised of a toroidal magnet, customized glass chamber, servo motor, peristaltic pump, vinyl pipe, circular water bath, sample container, and heating magnetic stirrer. The toroidal magnet was assembled with eight NdFeB magnetic tiles each with inner diameter of 70 mm, external diameter of 90 mm, and length of 80 mm. A 3-Tesla magnetic field was generated inside the toroidal magnet. Driven by the servo motor, the magnet rotated with a certain frequency to form a rotating magnetic field. The customized glass chamber was divided into interior and exterior parts; the interior was a 13-turn helical channel connected to the vinyl pipe for reactant fluid to flow through, and the exterior was connected to the circular water bath for holding circulating water at the desired temperature. The helical and vinyl pipe constituted of the three-millimeter channel and the reactant fluid (homogeneous mixed solution of NaOH and CS) were propelled by the peristaltic pump to continuously flow in the channel. To prevent the reactant fluid from precipitating, the sample container (a 500 ml flask) was placed on a magnetic stirrer for agitation (300 rpm).

2.3. Determination of the most effective method and pretreatment time

We preliminarily set out to confirm the effectiveness of the proposed processing system and determine the most effective pretreatment method out of four different pretreatments: (I) CS was pretreated with NaOH under agitation (magnetic stirring, 300 rpm); (II) CS was pretreated with NaOH in a flowing state inside the 3-mm channel (flow rate, 0.3 L/min); (III) CS was pretreated with NaOH in a flowing state and with a static magnetic field applied (flow rate, 0.3 L/min); and (IV) CS was pretreated with NaOH in a flowing state and with a rotating magnetic field applied (flow rate, 0.3 L/min; frequency, 6 Hz). Ten grams of CS and 400 ml of NaOH solution (1.5%, w:w) were used in each pretreatment at 50 °C temperature. The high liquid to solid ratio of 40 was to keep the millimeter-scaled channel from being clogged by the mixed sample solution. Homogeneous reactants (30 ml) were sampled at 2, 4, 8, 12, 16, 24, and 36 h and then centrifuged at 5000*g* for 10 min. The solid residue was washed with 50 ml distilled water three times and then oven-dried at 50 °C for 72 h. The supernatant was collected together with the wash water for sugar (pentose and hexose) analysis by high performance liquid chromatography. The remaining residuals were collected for morphological observation and enzymatic hydrolysis. According to a previous study (Zhang et al., 2013), pentose yield was calculated as the sum of the xylose released from per gram of dry CS during pretreatment and enzymatic hydrolysis. Hexose yield was calculated as the sum of the glucose released from per gram of dry CS during pretreatment and enzymatic hydrolysis.