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Investigation on characteristics of corn stover and sorghum stalk processed by ultrasonic vibration-assisted pelleting

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ABSTRACT

Cellulosic ethanol produced from cellulosic biomass is an alternative to petroleum-based liquid transportation fuels. The cost-effectiveness of cellulosic ethanol manufacturing has been hindered by several technical barriers. One such barrier is that low density of biomass causes high costs of biomass transportation, handling, and storage. Another barrier is low sugar yield in enzymatic hydrolysis, making enzymatic hydrolysis an expensive and slow step. Ultrasonic vibration-assisted (UV-A) pelleting of cellulosic biomass can increase its density and reduce the costs of biomass transportation and handling. In addition, sugar yield of biomass pellets processed by UV-A pelleting was higher than that of particles (unpelleted biomass) with diluted acid pretreatment. The reason that UV-A pelleting could increase sugar yield of biomass is still unclear. The objective of this study was to investigate reasons that UV-A pelleting combined with diluted acid pretreatment could increase sugar yield of biomass. High sugar yield is preferred to achieve high ethanol yield. Effects of UV-A pelleting on biomass characteristics (such as chemical composition, crystallinity index, thermal properties, and morphological structure) were investigated. The results showed there was no significant difference in chemical composition between pellets and particles. However, crystallinity of biomass increased after UV-A pelleting. In addition, pellets had higher decomposition temperature than particles, indicating that pellets were more thermally stable than particles. Examinations on morphological structure of biomass showed that softened surface regions of biomass were removed and cellulose microfibrils were revealed after UV-A pelleting.

1. Introduction

Cellulosic ethanol produced from cellulosic biomass (herbaceous, woody, and generally inedible portions of plant matter) is an attractive alternative to petroleum-based liquid transportation fuels. Land resources in the U.S. are sufficient to sustain production of enough biomass annually to displace 30% or more of the nation's current petroleum consumption [1]. Furthermore, cellulosic ethanol has great environmental advantages over grain-based ethanol [2,3].

Cost-effective manufacturing of cellulosic ethanol has been facing several technical barriers. One barrier is related to low density of raw cellulosic biomass (ranging from 24 to 266 kg/m^3 [4]), causing high costs in biomass transportation and storage. Another barrier is low sugar yield in enzymatic hydrolysis, which leads to low biomass-to-ethanol conversion rate and high costs in converting biomass into ethanol.

Pelleting (agglomeration of small particles into larger particles by means of mechanical or thermal processing [5]) can significantly increase density of cellulosic biomass. The density of biomass pellets could reach 1200 kg/m^3 [6]. In turn, costs for transporting and storing of pelleted cellulosic biomass are less than 1/2 and 1/10 of those of raw cellulosic biomass [7,8]. In addition, pellets with uniform size and shape are easier for handling with existing equipment. A variety of traditional pelleting methods have been reported in the literature, including ring-die pelleting [9–11], flat-die pelleting [9,11], screw extrusion [12], and piston press [13]. For these pelleting methods, cellulosic biomass usually needs to be preheated (by high-temperature steam or heated dies) and binders are

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often needed [4]. By contrast, ultrasonic vibration-assisted (UV-A) pelleting does not require preheated biomass and binders. However, UV-A pelleting can produce pellets whose density and durability are comparable to those produced by traditional pelleting methods [6].

Pelleting of biomass has also been considered as an effective pretreatment method to increase biomass sugar yield in enzymatic hydrolysis. Theerarattananoon et al. [14] reported that sugar yield of biomass (big bluestem, corn stover, sorghum stalk, and wheat straw) processed by ring-die pelleting was 3–11% higher than that of unpelleted biomass. Lamsal et al. [15] reported that sugar yield of wheat bran processed by screw extrusion was about 30% higher than that of unpelleted wheat bran. A similar trend was reported by Yoo et al. [16,17] in screw extruding of soybean hulls. Our previous studies showed that UV-A pelleting of biomass was beneficial for increasing sugar yield of biomass in enzymatic hydrolysis. Zhang et al. [18] reported that, with dilute acid pretreatment, switchgrass processed by UV-A pelleting had 20% higher sugar yield than unpelleted switchgrass, while the difference became 75% without pretreatment. Zhang et al. [19] reported that biomass (corn stover and sorghum stalk) processed by UV-A pelleting had higher sugar yield than particles (unpelleted biomass) under dilute acid pretreatment. However, the reason that UV-A pelleting increases sugar yield of biomass is still unclear.

In this study, effects of UV-A pelleting on characteristics (such as crystallinity index, chemical structure, and thermal properties) of corn stover and sorghum stalk were investigated to explain the mechanism which UV-A pelleting increases sugar yield of biomass. A variety of measurement methods were used determine the characteristics, such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA), and solid-state cross polarization/magic angle spinning (CP/MAS) ^{13}C nuclear magnetic resonance (NMR) spectroscopy. Morphological structure of biomass was also observed using scanning electron microscope (SEM).

2. Materials and methods

2.1. Materials

Corn stover and sorghum stalk used in this study were harvested at the Kansas State Agronomy Farm in November 2010. After harvesting, they were chopped to approximately 180–230 mm using a large tub grinder (Haybuster H-1150 series, DuraTech Industries International Inc., Jamestown, ND, USA). Chopped biomass was then transported to a lab located at Kansas State University in sealed paper bags. Fig. 1 shows the experimental procedure. The chopped biomass was milled into particles using a cutting mill (model SM 2000, Retsch, Inc., Haan, Germany) with a sieve whose mesh size was 1 mm. The milled biomass was referred to as biomass particles which were separated into two portions. One portion of biomass was processed by UV-A pelleting. Both particles and pellets were then pretreated by diluted acid solution. Enzymatic hydrolysis was performed on biomass sample. Moisture content (MC) of biomass particles was measured and adjusted to 10% by following an NREL laboratory analytical procedure before pretreatment [20]. Biomass with 10% MC would produce pellets with high density and durability in previous experiment.

2.2. Experimental setup for UV-A pelleting

UV-A pelleting was conducted on a modified ultrasonic machine (model AP-1000, Sonic-Mill, Albuquerque, NM, USA). The schematic illustration of the experimental setup for UV-A pelleting is shown in Fig. 2. The machine was composed of two systems: an ultrasonic vibration generation system, and a pneumatic loading system.

The ultrasonic-vibration generation system was consisted of three major parts: a power supply, a converter, and a pelleting tool. The power supply converted 60 Hz electrical supply into 20 kHz electrical power. The high-frequency electrical power was applied to the piezoelectric converter and converted into high-frequency mechanical motion. The mechanical motion was amplified by the coupler and transmitted to the titanium pelleting tool which vibrated at the frequency of 20 kHz. The pelleting tool was connected to the converter and used to compress biomass particles into pellets. Ultrasonic power (percentage of power from power supply) can be adjusted from 0 to 100% and controls amplitude of the pelleting tool vibration. The higher the ultrasonic power, the larger the tool vibration amplitude.

The pneumatic loading system was consisted of three major parts: an air compressor, a pressure regulator, and a pneumatic cylinder. The air compressor (1.2 kw, 125 L, Sears, Roebuck and Co., Hoffman Estates, IL, USA) produced compressed air which was fed into the pneumatic cylinder (ARO Equipment Corporation, Bryan, OH, USA). The air pressure in the pneumatic cylinder was controlled by the pressure regulator and referred to as pelleting pressure. The pneumatic cylinder was driven by the compressed air and pushed the pelleting against biomass in a three-piece aluminum mold.

The mold was used to held biomass particles and clamped by a fixture on to the machine. The top two parts of the mold formed a central cylindrical cavity and the bottom part served as a base. The diameter of the mold cavity (18.6 mm) was slightly larger than that of the tip of the pelleting tool (17.4 mm).

There are five steps to make a pellet in this study: (1) assemble the mold and put 1 g of biomass particles in the central cavity of the mold, (2) adjust pelleting pressure to 50 psi and feed the pelleting tool down to compress particles, (3) turn on ultrasonic power at 50% and count 3 min using a stop watch, (4) turn off ultrasonic power and lift up the pellet tool, and (5) dissemble the mold and take out the pellet. Since there is no mass loss in the pelleting process, same amount of the initial particles for both particles and pellets.

2.3. Pretreatment

Pretreatment was carried out on a pressure reactor (Parr Instrument Company, Moline, IL, USA) with a 600 mL reaction vessel. A mixture of 20 g biomass (particles or pellets) and 200 mL 2% (w/v) diluted sulfuric acid (solid content about 10%) were loaded into the reaction vessel at room temperature. The pellets were totally dissolved in diluted sulfuric acid before heating. The biomass slurry was treated at 140 °C for 30 min. Meanwhile, two four-blade impellers were used to stir the slurry to ensure that temperature was evenly distributed in pretreatment. Pretreated biomass was washed with distilled water and centrifuged three times (at 8000 rpm for 20 min) to remove dissolved sugars and sulfuric acid. Washed biomass samples were weighed and split into two portions. The MC of pretreated biomass ranged from 84.5% to 89% after pretreatment for different samples. The rest portion was used for moisture content measurement and chemical compositional analysis.

2.4. Enzymatic hydrolysis

Enzymatic hydrolysis was conducted in a 1.36% (w/v) sodium acetate buffer solution with the addition of 0.02% (w/v) sodium azide to prevent the microbial growth during hydrolysis. The pH value of the solution was adjusted to 4.8. Biomass of 5 g dry weight was mixed with 100 mL buffer solution in 125 mL flasks in a 50 °C