



# Effects of ultrasonic vibration-assisted pelleting on chemical composition and sugar yield of corn stover and sorghum stalk

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## ABSTRACT

Cellulosic ethanol made from cellulosic biomass is an alternative to petroleum-based transportation fuel. 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. The effects of UV-A pelleting on sugar yield of cellulosic biomass have not been fully investigated. The objective of this paper is to investigate effects of UV-A pelleting on chemical composition and sugar yield of cellulosic biomass. The effects were investigated with and without dilute acid pretreatment using corn stover and sorghum stalk. It was found that there was no significant difference in chemical composition between pelleted and unpelleted biomass whether they went through dilute acid pretreatment or not. After dilute acid pretreatment, cellulose recovery of pelleted biomass was significantly higher than that of unpelleted biomass. UV-A pelleting could significantly increase the sugar yield in enzymatic hydrolysis for both corn stover and sorghum stalk.

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## 1. Introduction

In 2011, about 140 billion gallons of liquid transportation fuels were consumed in the United States and more than half of these fuels were derived from foreign petroleum [1]. Increasing demands and concerns for reliable supply of liquid transportation fuels make it important to develop domestic sustainable alternatives to petroleum-based liquid transportation fuels. Cellulosic ethanol is one such alternative. Land resources in the United States are sufficient to sustain production of enough cellulosic biomass annually to displace 30% or more of the nation's current petroleum consumption [2]. In addition, using cellulosic biomass as feedstocks for ethanol production can mitigate the accumulation of greenhouse gas in the atmosphere and promote economic growth in rural areas [3].

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Major steps in cellulosic ethanol production are shown in Fig. 1. The cost-effectiveness of cellulosic ethanol manufacturing has been challenged by several technical barriers. One such barrier is related to the low density of raw cellulosic biomass (ranging from 24 to 250 kg/m<sup>3</sup> [5]), causing high costs of biomass transportation, handling, and storage. Another barrier is the difficulty in converting cellulose (a major sugar source in cellulosic biomass) into fermentable sugars, leading to low sugar yield in enzymatic hydrolysis and making enzymatic hydrolysis an expensive and slow step.

Pelleting of cellulosic biomass can significantly increase its density to higher than 600 kg/m<sup>3</sup> [6–8]. In turn, the costs of transporting and storing of pelleted cellulosic biomass could be reduced to less than 50% and 10% of those of raw cellulosic biomass, respectively [9,10]. Furthermore, pellets can be handled with existing grain-handling equipment at biorefinery plants. Unlike traditional pelleting methods (e.g., screw extrusion, press briquetting, or ring-die pelleting), UV-A pelleting does not use high-temperature steam, high pressure, and binder material. Pellet density and durability produced by UV-A pelleting are comparative

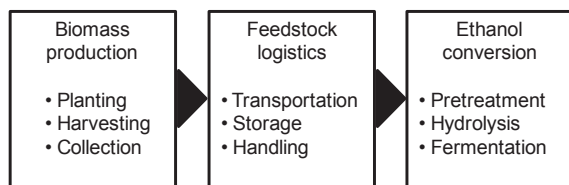


Fig. 1. Major steps in cellulosic ethanol manufacturing (after [4]).

to those produced by traditional pelleting methods in terms of pellet density, durability and sugar yield [11,12].

It has been reported that traditional pelleting methods have positive effects on the sugar yield of cellulosic biomass in enzymatic hydrolysis. Theerarattananoon et al. [13] compared the chemical composition and sugar yield of pelleted (by ring-die pelleting) biomass with those of unpelleted biomass (wheat straw, corn stover, big bluestem, and sorghum stalk). They found that ring-die pelleting changed the chemical composition of tested biomass. In addition, sugar yields of biomass processed by ring-die pelleting were significantly higher than those of biomass not processed by ring-die pelleting. Lamsal et al. [14] compared the sugar yield of wheat bran processed by screw extrusion with that of unprocessed wheat bran. They reported that sugar yield of processed wheat bran was about 30% higher than that of unprocessed wheat bran. The same trend was reported by Yoo et al. [15,16] with soybean hulls. They reported that screw extrusion could increase the sugar yield to 94% for soybean hulls. The sugar yield of soybean hulls not processed by screw extrusion was only 41%.

The above mentioned results cannot be directly applied to evaluate effects of UV-A pelleting on sugar yield of cellulosic biomass. The pelleting mechanism of UV-A pelleting is different from those of traditional pelleting methods. Zhang et al. [16] reported that increasing of ultrasonic power increased sugar yield in the earlier studies. Similar results were also reported by Zhang et al. [7,17]. However, they only use one type of materials and reason that UV-A pelleting could increase sugar yield of biomass is still unclear. Thus, effects of UV-A pelleting on chemical composition were investigated to explain the mechanism that UV-A pelleting could increase sugar yield of biomass. In this paper, chemical composition and sugar yield of biomass processed by UV-A pelleting were compared with those not processed UV-A pelleting. The comparisons were made with and without dilute acid pretreatment based on two types of cellulosic biomass (corn stover and sorghum stalk).

## 2. Materials and methods

### 2.1. Experimental procedure

The experimental procedure is shown in Fig. 2. The corn stover and sorghum stalk used in this study were harvested on the Kansas State University Agronomy Farm in November, 2008. After harvesting, the biomass (corn stover and sorghum stalk) was chopped to the size of 18–23 cm using a tub grinder (Haybuster H-1150 series, DuraTech Industries International Inc., Jamestown, ND, USA). The chopped biomass was milled into small particles using a cutting mill (SM 2000, Retsch Inc., Newtown, PA, USA). Particle size was controlled by using a sieve with 1-mm screen size. After milling, the moisture content of biomass particles was measured and adjusted to 10% by following an NREL laboratory analytical procedure [18]. Previous studies showed that biomass particles with about 10% moisture content would produce pellets with high density and durability [19,20]. The prepared biomass particles were separated into two groups. Group A was processed by UV-A

pelleting and group B was not. Each group of biomass was further separated into two portions: portions A<sub>1</sub> and B<sub>1</sub> went through dilute acid pretreatment and enzymatic hydrolysis, and portions A<sub>2</sub> and B<sub>2</sub> went through enzymatic hydrolysis without dilute acid pretreatment. Compositional analyses and sugar yield analyses were conducted to determine the chemical composition and sugar yield of different biomass samples.

### 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). Fig. 3 is a schematic illustration of the experimental setup for UV-A pelleting. 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 motion was amplified by the coupler and transmitted to the titanium pelleting tool. In this study, the pelleting tool vibrated at the frequency of 20 kHz.

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 was 0.34 MPa in this study.

A three-piece aluminum mold was used to hold biomass particles in UV-A pelleting. 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). In this study, 1 g of biomass was put in the mold and compressed for 3 min to make a pellet.

### 2.3. Pretreatment

Two different pretreatment conditions were adopted in this study. Portions A<sub>1</sub> and B<sub>1</sub> (Fig. 2) went through dilute acid pretreatment before enzymatic hydrolysis. Portions A<sub>2</sub> and B<sub>2</sub> were directly processed by enzymatic hydrolysis without pretreatment.

Dilute sulfuric acid pretreatment was carried out in a pressure reactor (Parr Instrument Company, Moline, IL) with a 600-mL reaction vessel. A slurry of 20 g of biomass (milled particles or pellets) and 200-mL diluted sulfuric acid solution (2% w/v) was loaded in the reaction vessel. The slurry was heated by a heater and its temperature was maintained at 140 °C for 30 min. Two four-blade impellers were used to stir the slurry. For pretreatment of biomass pellets, the pellets were soaked in the diluted sulfuric acid solution at room temperature until they were dissolved before they were heated. After pretreatment, the slurry was centrifuged and separated into two fractions: a liquid fraction (an acid solution) and a solid one. The solid fraction was then washed with hot distilled water three times to remove sulfuric acid. After each time of washing, the water was separated from biomass and added to the acid solution. The final solution (which consisted of the acid solution and all of the water added into it) was referred to as filtrate of biomass. A part of washed biomass was used for moisture content and chemical composition analyses. The other part was used for subsequent enzymatic hydrolysis.

### 2.4. Enzymatic hydrolysis

Enzymatic hydrolysis was conducted in a 100-mM sodium acetate buffer solution (pH 4.8) with the addition of 0.02% (w/v) sodium azide to prevent the microbial growth during hydrolysis. Biomass (1 g dry weight) was mixed with 50-mL buffer solution in 125-mL flasks in a 50 °C water bath shaker (Model C76, New

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