



Pilot-scale study on the acid-catalyzed steam explosion of rice straw using a continuous pretreatment system

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HIGHLIGHTS

- ▶ A pilot-scale continuous pretreatment system was successfully developed.
- ▶ The pretreatment system shows good operational stability and durability.
- ▶ 73% of the total saccharification yield was obtained.
- ▶ The system can be used for the production of bioethanol and bio-based chemicals.
- ▶ The total sugar yield of rice straw in different scale system was compared.

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ABSTRACT

A continuous acid-catalyzed steam explosion pretreatment process and system to produce cellulosic ethanol was developed at the pilot-scale. The effects of the following parameters on the pretreatment efficiency of rice straw feedstocks were investigated: the acid concentration, the reaction temperature, the residence time, the feedstock size, the explosion pressure and the screw speed. The optimal presteaming horizontal reactor conditions for the pretreatment process are as follows: 1.7 rpm and 100–110 °C with an acid concentration of 1.3% (w/w). An acid-catalyzed steam explosion is then performed in the vertical reactor at 185 °C for 2 min. Approximately 73% of the total saccharification yield was obtained after the rice straw was pretreated under optimal conditions and subsequent enzymatic hydrolysis at a combined severity factor of 0.4–0.7. Moreover, good long-term stability and durability of the pretreatment system under continuous operation was observed.

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1. Introduction

Lignocellulosic materials, such as agricultural, hardwood and softwood residues, are potentially viable sources of sugars for the production of bioethanol, biobutanol, other biofuels or various valuable chemicals. Lignocellulosic materials are particularly attractive because they do not compete with food crops. In biomass processing, the primary goal is to remove the hemicelluloses and destroy the structure of the biomass so that cellulose is more accessible to enzymatic hydrolysis. This process is generally referred to as pretreatment, and it is one of the most difficult processes to optimize. Therefore, pretreatment is a central part of the lignocellulose-to-ethanol process.

Various pretreatment strategies have been examined, including dilute-acid hydrolysis, steam explosion, liquid hot water extraction, alkaline hydrolysis, ammonia treatment and various biological processes. Among these various types of pretreatment, dilute-acid hydrolysis and steam explosion has been widely tested in pilot scale equipment and is considered to more favorable method for industrial applications (Wyman et al., 2005; Alvira et al., 2010; Chandel et al., 2011; Modenbach and Nokes, 2012; Larsen et al., 2012). Moreover, addition of a mineral acid results in more efficient hemicelluloses hydrolysis and the subsequent enzymatic digestion during steam pretreatment. Therefore, the acid-catalyzed steam explosion process is considered to be close to commercialization (Galbe and Zacchi, 2012).

An estimated 600–900 million tons of rice straw are produced globally every year. Moreover, developing countries in Asia produce 90% of the world's rice straw (Sarkar et al., 2012). Therefore, rice straw is an attractive lignocellulosic material for biofuels and other useful biomass chemicals. Nevertheless, various characteristics of rice straw make it intractable. For example, rice straw is soft,

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hydrophobic and non-homogeneous. It also has a low bulk density and tends to wind around the instrument and stick together during processing. Hence, a detailed understanding of rice straw in a pilot-scale continuous pretreatment system will be crucial in enabling industrial applications for rice straw. In general, biomass is traditionally processed via a batch processing method; however, if ethanol production from rice straw is to be successful in the marketplace, a platform for its continuous processing must be developed.

Although many researchers have studied dilute-acid hydrolysis or steam explosion pretreatment in a pilot-scale continuous pretreatment system (Schell et al., 2003; Thomsen et al., 2006; Weiss et al., 2010; Fang et al., 2011a,b; Rocha et al., 2012a,b; Shekiro et al., 2012), less attention has been paid to rice straw in a continuous pretreatment system. INER has developed pretreatment systems for different scales on the basis of previous dilute-acid hydrolysis experiments that were performed at the lab-scale (400 g/batch), the bench-scale (10 kg/batch) and at a pilot plant (1 ton/day) harboring a cellulosic ethanol-testing platform (Chen et al., 2011a,b; Weng et al., 2011). Out of these in-house technologies, the pilot-scale continuous pretreatment system uses the acid-catalyzed steam explosion process to pretreat feedstocks such as rice straw.

The goal of this study was to define the optimal conditions for the acid-catalyzed steam explosion of rice straw in the pilot-scale continuous pretreatment system. The effects of acid concentration, reaction temperature, residence time, feedstock size, screw speed and explosion pressure on the pretreatment efficiency of the rice straw feedstock were evaluated. The pretreatment efficiency was evaluated by estimating the xylose yield after pretreatment and the glucose yield from the subsequent enzymatic hydrolysis. Moreover, the continuous operation stabilities and the corresponding performance will also be reported.

2. Methods

2.1. Biomass materials

The rice straw was primarily collected from private farms in Taiwan. The composition of the raw rice straw was as follows: glucan $32.9 \pm 0.1\%$, xylan $18.7 \pm 0.1\%$, arabinan $3.2 \pm 0.1\%$, extractive $10.1 \pm 0.2\%$, ash $11.3 \pm 0.1\%$ and lignin $19.1 \pm 1.1\%$. The composition was calculated on a dry weight basis.

2.2. Pretreatment system

The pilot-scale continuous pretreatment system consisted of a feedstock receiving hopper with a weighmetric screw feeder, a horizontal reactor (presteamer) with a long residence time for the presteaming, and for the acid impregnation of the biomass, a vertical high pressure steam explosion reactor, a receiving flash tank and a solid liquid separator. The unique design of the pretreatment system includes a piston pressure feeding device, special mechanical equipment and a modified outlet device for steam explosion. The horizontal and vertical high-pressure reactors are heated to the desired temperature by the direct injection of 6 and 16 kg/cm² saturated steam, respectively. The flow meters, equipped with automatic temperature or pressure compensation, maintained the desired temperature and pressure. The horizontal presteamer is connected to a diaphragm pump that adds dilute acid solution. The acid mixing and preheating times of rice straw are controlled by the screw speed of the horizontal reactor. Additionally, the feeding rate of the screw feeder and diaphragm pump can be used to regulate the solid–liquid ratio of rice straw and dilute sulfuric acid solution. An advanced Distributed Control System

(DCS) has been used in the pilot-scale continuous pretreatment system. It provides continuous and automatic operation during the pretreatment process. The throughput can vary from 100–200 kg of dry material per hour, depending on the bulk density of the raw material and processing conditions.

2.3. Pretreatment process

The acid-catalyzed steam explosion processes used in this pretreatment system are shown in Fig. 1. Air-dried rice straw with a moisture content of 15% was chopped into smaller pieces (≤ 2 cm) with a shredder and used as the raw material. These rice straw pieces were immediately transferred to the hopper of the pretreatment system by a pneumatic conveyor system designed for continuously transporting lignocellulosic materials in the pilot plant. Subsequently, the rice straw pieces were continuously fed into the horizontal pressurized reactor with a piston pressure-feeding device. The rice straw was presoaked with dilute sulfuric acid and preheated under a selected temperature in the horizontal reactor. Then, the presoaked rice straw was continuously transferred to the vertical high-pressure reactor that proceeded the acid-catalyzed steam explosion. The resultant slurry is automatically continuously transferred to the solid–liquid separator. The xylose-rich hydrolysate and the cellulose-rich residues were obtained.

In this study, the throughput was 100 ± 20 kg of rice straw per hour, and the total feed amount was 1000 ± 50 kg for each test condition. The rice straws were mixed with dilute sulfuric acid solution to an initial solid loading of about 40–50% (w/w) and preheated as they passed through the horizontal pressurized reactor at a screw speed of 1.7 rpm and temperature of 100–110 °C. The test conditions of the acid-catalyzed steam explosion process varied in temperature from 160 to 190 °C, residence times varied from 2 to 10 min and acid concentrations in the acid supply tank varied from 1.0 to 8.4 wt.%. The effects of the feedstock size (≤ 1 , ≤ 2 cm), the screw speed of the horizontal reactor (1.7 rpm, 2.5 rpm, 3.4 rpm, 5.1 rpm) and the explosion pressure (10 kg/cm², 13 kg/cm², 16 kg/cm²) on the pretreatment efficiency were also tested.

In general, the hydrolysate and the pretreated residues were sampled at every 2 h. Samples were analyzed for the sugar concentrations of their hydrolysates, the composition of the pretreated rice straw and the subsequent enzymatic digestibility. Four samples were taken for each test condition. The pH, concentration, percentage of oligomers in the hydrolysate, carbohydrate content of the pretreated residues and the total enzymatic hydrolysis for each test run were the average value of the four samples.

2.4. Enzymatic hydrolysis

The unwashed and washed pretreated residues (about 1 g dry weight) were transferred to a 250 ml shake flask, and the pH was adjusted to 4.8 by adding 50 mM acetate buffer at a 2% (w/v) solid loading. The digestion of the cellulose was conducted at a cellulase (Novozyme celtic CTec2) activity level of 15 FPU/g cellulose. The enzymatic digestion was carried out at 50 °C and 100 rpm for 24 h.

In this study, the pretreatment efficiency is estimated by combining total xylose and glucose conversion yields after both the pretreatment and enzymatic hydrolysis processes. The definition of the total saccharification yield (overall monosugar yield) is the sum of the monomeric xylose and glucose saccharification yields. The xylose/glucose saccharification yields are calculated by the following equations

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