



Pilot scale study on steam explosion and mass balance for higher sugar recovery from rice straw



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HIGHLIGHTS

- First study on process parameters for SE of rice straw by water, SA and PA.
- A pilot-scale batch pretreatment system was successfully developed.
- Effect of inhibitors and oligomers on enzymatic hydrolysis was studied.
- Process mass balance showed effective pretreatment using both water and SA.
- Total saccharification yield of 81.8% and 77.1% obtained for water and SA.

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ABSTRACT

Pretreatment of rice straw on pilot scale steam explosion has been attempted to achieve maximum sugar recovery. Three different reaction media viz. water, sulfuric acid and phosphoric acid (0.5%, w/w) were explored for pretreatment by varying operating temperature (160, 180 and 200 °C) and reaction time (5 and 10 min). Using water and 0.5% SA showed almost similar sugar recovery (~87%) at 200 and 180 °C respectively. However, detailed studies showed that the former caused higher production of oligomeric sugars (13.56 g/L) than the later (3.34 g/L). Monomeric sugar, followed the reverse trend (7.83 and 11.62 g/L respectively). Higher oligomers have a pronounced effect in reducing enzymatic sugar yield as observed in case of water. Mass balance studies for water and SA assisted SE gave total saccharification yield as 81.8% and 77.1% respectively. However, techno-economical viability will have a trade-off between these advantages and disadvantages offered by the pretreatment medium.

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1. Introduction

Lignocellulosic biomass (LCB) is the most abundant material which is economically available across the globe with very few other competitive uses. LCB has been considered as a potent source for bioethanol production owing to its richness in fermentable sugars (Ragauskas et al., 2006; Sukumaran et al., 2010; Agbor et al., 2011; Kumar et al., 2014). These sugars upon anaerobic fermentation using yeast e.g. *Saccharomyces cerevisiae* produce bioethanol. Ethanol is an excellent blend component for transportation fuels like gasoline. Additionally, LCB based fuel production would increase the energy security and reduce greenhouse gas emissions. LCB comprises of cellulose, hemicellulose and lignin besides

extractives and ash. Cellulose is a polysaccharide consisting of linear chains of β -(1-4)-D-glucopyranose units. Hemicellulose is a heteropolymer composed of xylan, arabinoxylans, xyloglucan, glucuronoxylan and glucomannan, while lignin is a complex biopolymer of mono-lignols deposited in the cell walls of LCB, which basically provides strength to LCB against biotic and abiotic conditions (Tokay, 2000). LCB contains about 50–70% of fermentable sugars present in the form of holocellulose i.e. cellulose and hemicellulose. However, releasing sugars from biomass poses a great challenge. LCB on chemical and/or biochemical transformation to bioethanol requires a pretreatment to destruct the biomass matrix making it amenable to subsequent enzymatic hydrolysis by exposing holocellulose, which after enzymatic hydrolysis and subsequent fermentation produces ethanol.

Pretreatment is the most energy intense and expensive step for production of ethanol from biomass (Tokay, 2000; Alvira et al.,

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2010). Several pretreatment technologies have been explored, such as mechanical milling, steam explosion, AFEX, acid hydrolysis, organosolv, alkaline hydrolysis, ionic liquids, hot water and ammonia pretreatment (Chaturvedi et al., 2013; Mosier et al., 2005). All these methods are either expensive (ionic liquids, organosolv) or involve the use of higher acid or base concentration which leads to higher effluent treatment costs and need for expensive metallurgy for reactor and vessels. Among various alternatives, steam explosion has been considered as one of the efficient, greener and cost-effective method for industrial application, thereby prompting researchers to explore this technology (Zhang et al., 2012; Oliveira et al., 2013). Steam explosion has been employed for various LCB, including corn stover, wheat straw and sugarcane bagasse for production of ethanol (Ballesteros et al., 2006; Haixia et al., 2011; Harrison et al., 2013; Tucker et al., 2003).

In India, total surplus biomass is available to the tune of 155.2 Million Metric Tons Per Annum (MMTPA) which has a potential to produce 34.1 billion liters of ethanol. About 8.9 MMTPA rice straw is rendered unused and destroyed in fields by burning, thus leading to air pollution (Sukumaran et al., 2010). This rice straw has a potential to produce 2.1 billion liters of ethanol annually. Considering its high sugar content, rice straw may serve as a potential feedstock for ethanol production in tropical countries like India (Sarkar et al., 2012; Kim et al., 2013). In order to improve the efficiency of pretreatment, impregnation of LCB in dilute acids before steam explosion has been found effective for achieving high sugar yield (Sarkar et al., 2012; Chen et al., 2013).

In the present study, a series of steam explosion experiments on pilot plant scale in batch mode were performed to evaluate effectiveness of very dilute sulfuric and phosphoric acid vis-à-vis water on the rice straw at different operating conditions to enhance the sugar recovery during pretreatment and subsequent enzymatic hydrolysis. Further, the effect of inhibitors like acetic acid, furfural and 5-hydroxymethylfurfural (HMF) produced during pretreatment on enzymatic hydrolysis has been evaluated. In order to establish the commercial viability, the mass balance of the process of pretreatment and enzymatic hydrolysis has been established.

2. Methods

2.1. Biomass material

Rice (*Oryza sativa*) straw was collected in August 2012 from the Mathura District (27.49°N, 77.67°E) in Uttar Pradesh (India) having an average elevation of 174 m. Mathura has a tropical climate with hot summers (up to 44 °C) and cold and foggy winters with temperature dipping to 5 °C. The average rainfall is approximately 600 mm, received mostly during the monsoons from July to September. Rice straw was air dried and shredded into the size of 5 mm by knife mill and stored in sealed plastic bags at 30 °C. All standards and chemicals like xylose, glucose, arabinose, galactose, acetic acid, 5-hydroxymethylfurfural (HMF), furfural, sulfuric acid, phosphoric acid, avicel and lignin were analytical grade procured from Merck and Sigma Aldrich (India) and were used as received. Compositional analysis of rice straw on dry basis was carried out by NREL procedure and showed glucan, 39.6%; xylan, 15.0%; lignin, 20.9%; galactan, 0.9%; arabinan, 2.7%; ash, 13.7% and extractives, 6.2% (w/w), totaling to 99% (Sluiter et al., 2005, 2006, 2008).

2.2. Impregnation

Impregnation of rice straw was carried out in batches of 15 kg rice straw by placing it in a stainless steel (SS) wire mesh bin and by dipping this bin into a plastic tank having either water or

0.5% (w/w) dilute sulfuric acid (SA) or 0.5% (w/w) phosphoric acid (PA). The liquid was recirculated using a pneumatic pump for 30 min at 30 °C. The soaked rice straw was dewatered using a hydraulic press at 100 bar. The pressed rice straw was properly mixed to determine moisture content using an IR based moisture analyzer (MA 150 Sartorius, Germany) following NREL protocol and found to contain about 60% moisture (Sluiter et al., 2008).

2.3. Steam explosion and combined severity factor (CSF)

Steam explosion pilot plant, designed in-house, comprised of high pressure reactor of 10 L (SS) equipped with feeding device, cyclone separator, quick opening pneumatic butterfly valve, electric heater and a noise absorber. Before starting the experiment, steam explosion digester was flushed 3–4 times with steam at 10 bar to quickly attain the desired operating temperature. 0.4 kg of pressed rice straw on dry basis was introduced in the digester and temperature was increased by injecting high pressure steam about (10–15) bar. The operating temperatures were either 160, 180 or 200 °C and reaction time at each temperature was 5 or 10 min. After pretreatment, the treated rice straw was recovered from cyclone separator, separated into two fractions by filtration to xylose rich hydrolyzate and cellulose rich residue.

Each experimental condition imparted different severity to the medium which can be represented as a combined severity factor (CSF). CSF serves as a predictive tool to translate the harshness of process conditions employed in the pretreatment process and relates to the production of holocellulose and inhibitors. Multiple responses can be easily visualized together as a function of this parameter. CSF ($\text{Log } R_o$) was calculated using the following equation (Pedersen and Meyer, 2010):

$$\text{Log } R_o = \text{Log}_{10} \left(t * \exp \left(\frac{T - T_R}{14.75} \right) \right) - \text{pH} \quad (1)$$

where, 't' is the reaction time (min), 'T' is the operating temperature (°C), 'T_R' is the reference temperature (100 °C), and 'pH' is that of the pretreatment hydrolyzate.

2.4. Analysis methods

Total solids, glucan, xylan, lignin, ash content and extractives in native and pretreated rice straw were analyzed by NREL protocol. The glucan, xylan and lignin content in cellulose rich residues of pretreated rice straw were measured by a LAP NREL procedure, the extractives in glucan rich residue were not determined as these were lost during impregnation (Sluiter et al., 2005, 2006, 2008).

Sugar and inhibitors concentration in the xylose rich hydrolyzate received after pretreatment were measured by HPLC (Waters, USA) with refractive index detector following NREL protocol (Sluiter et al., 2008). The column used for sugar analysis was Aminex HPX-87P maintained at 75 °C and Milli-Q water was used as mobile phase at 0.6 ml/min. Inhibitors were analyzed using Biorad Aminex HPX-87H column at 50 °C, 0.008 N H₂SO₄ at a flow rate of 0.6 ml/min as mobile phase and using UV detector. Both the columns were equipped with suitable guard columns. The pH of hydrolyzate was measured after each experiment using pH meter (HACH SensION + PH31).

Oligomeric sugars in hydrolyzate were analyzed following NREL LAP protocol using 4% sulfuric acid (Sluiter et al., 2006, 2008). The oligomeric sugar concentration was determined by subtracting the monomeric sugar concentration in the hydrolyzate from the total sugar concentration of hydrolyzate received after 4% sulfuric acid treatment for complete hydrolysis.

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