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## Hydrolysis of bamboo biomass by subcritical water treatment



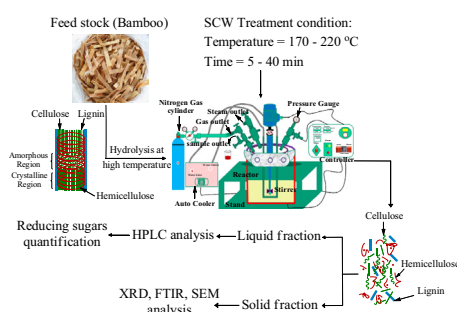
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### HIGHLIGHTS

- Reaction temperature and time shown more impact on total reducing sugars yield.
- Highest yield of total reducing sugars obtained at 180 °C and 25 min of reaction time.
- X-ray diffraction profile shown increase in crystallinity index after treatment.
- Kinetics of bamboo was considered as first-order irreversible series reaction.

### GRAPHICAL ABSTRACT



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### ABSTRACT

The aim of present study was to obtain total reducing sugars (TRS) from bamboo under subcritical water (SCW) treatment in a batch reactor at the temperature ranging from 170 °C to 220 °C and 40 min hydrolysis time. Experiments were performed to investigate the effects of temperature and time on TRS yield. The maximum TRS yield (42.21%) was obtained at lower temperature (180 °C), however longer reaction time (25 min). X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) analysis were used to characterise treated and untreated bamboo samples. The XRD profile revealed that crystallinity of bamboo increased to 71.90% with increase in temperature up to 210 °C and decreased thereafter to 70.92%. The first-order reaction kinetic model was used to fit the experimental data to obtain rate constants. From the Arrhenius plot, activation energy and pre-exponential factor at 25 min time were found to be 17.97 kJ mol<sup>-1</sup> and 0.154 min<sup>-1</sup>, respectively.

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### 1. Introduction

The continuous increase in the population growth pushes towards expanding the utilisation of fossil assets in the production of energy and chemicals (Mohan et al., 2015b). The high utilisation rate of fossil resources has a direct impact on the economic, environmental, political, life quality of population and also increases emission of greenhouse gases, which aggravate global warming.

Hence it requires an alternative energy solution to decrease today's rapid consumption of fossil resources. A potential alternative solution to this problem could be utilisation of lignocellulosic biomass as an alternative energy source for the future (Holm and Lassi, 2011). Currently extensive research is being undertaken to produce environmentally friendly energy power and worth included chemicals from lignocellulosic biomass (Mohan et al., 2015a, 2015b). Use of biomass for energy avoids the increase of CO<sub>2</sub> in the atmosphere (Asghari and Yoshida, 2010). As per the facts, aggregate energy prepared from photosynthesis is ten times higher than that of the fossil-fuel assets utilised within the world consistently (Wang et al., 2010).

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Lignocellulosic biomass is dominated by three naturally occurring polymers, cellulose (30–50%), hemicellulose (25–35%) and lignin (5–30%). The sources of lignocellulosic biomass, which are waste agriculture residue, wood, grass; forestry and municipal solid wastes are recognised as tempting feedstock for the creation of fuel alcohol, due to their accessibility in huge amounts at low price (Roque et al., 2012). Cellulose is the primary constituent in any biomass to produce oligosaccharides (cellotetraose, cellotriose, and cellobiose), and monomeric sugars (glucose and fructose). Hemicellulose is the secondary most abundant polymer in biomass to produce pentose sugars (xylose, arabinose) and hexoses (galactose, glucose and mannose). Hemicellulose sugars have a lower molecular weight compared to cellulose and limb with short horizontal chains that are effortlessly hydrolysed (Agbor et al., 2011). The primary point of lignocellulosic biomass pretreatment is to alter the cellulose structure, and make the cellulose structure more accessible to enzymes and/or chemicals (Holm and Lassi, 2011). Among the various biomass wastes, bamboo is an ancient woody grass and dispersed in tropical, subtropical and mild temperature zones. Generally, bamboo seen as the “poor man’s tree,” in recent years it has climbed to an innovative, modern crude material and substitute for wood. In Asia, India is the second richest country in bamboo genetic assets after China. These two nations together have more than half portion of the aggregate bamboo resources universally (Katwal et al., 2003). For the management and maintenance of natural resources, 170 states have signed on the action program for the 21st century (Agenda 21) in the year 1992 in Brazil. The aim is to search for new solutions to decrease today’s rapid consumption of fossil resources and non-renewable resources such as petroleum, natural gas, coal and minerals (Kamm and Kamm, 2004).

The significant innovations proposed for hydrolysis of lignocellulosic biomass incorporate acid or base treatments, which are not satisfactory because of the ecological issues, and enzymatic hydrolysis, which is a time taking process (Rogalinski et al., 2008). Whereas, sub or supercritical water treatment have been picking up an expanding consideration as an environmentally friendly technique with an extensive variety of provisions, such as extraction, hydrolysis, and wet oxidation of organic compounds. It is very cheap, non-toxic, non-flammable, and non-explosive and have many advantages compared to other substances, mainly in the field of “green chemistry” (Ehara and Saka, 2005; Zhao et al., 2009). Sub critical water (SCW) is defined as hot water at temperature ranging between 100 and 374 °C under high pressure to maintain water in the liquid state. The physical properties of water in sub critical condition changes with increase in temperature (Aida et al., 2010; Tsigie et al., 2012). SCW treatment has been known as an exceptionally extraordinary medium or solvent for various chemical reactions and extraction of compounds. SCW is also used as an effective catalyst for hydrolysis or biodegradable reactions. A wide variety of organic pollutants from industrial waste is completely removed by SCW within a few minutes. Therefore, SCW is found to be turning into a promising extractant and catalyst (Pourali et al., 2009).

Scanty information is available in the literature on SCW treatment of bamboo biomass. In this present study, SCW treatment has been employed as a method to produce total reducing sugars (TRS) from bamboo at temperature ranging from 170 °C to 220 °C and reaction time 5 to 40 min. Treated and untreated bamboo samples are characterised by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) techniques to analyse the structural changes. Attempts have been made to investigate the reaction kinetics of bamboo to fit the experimental data for obtaining the reaction kinetic parameters

(i.e., rate constant, activation energy and pre-exponential factor). The study also helps in understanding the potential usage of bamboo as a feedstock for TRS production.

## 2. Methods

### 2.1. Materials

Bamboo biomass (*Bambusa cacharensis* R.B. Majumdar) used as a raw material in this study was obtained from nearby IIT Guwahati forest area, Assam, India (26°11′14″N 91°41′30″E). The air-dried biomass samples were chopped using chopping machine, and sieved (mesh size – BSS 30) to get homogeneous powder (particles size 0.5 mm). Before the experiments, bamboo powder was dried at 60 °C for 24 h. The high performance liquid chromatography (HPLC) standards, glucose ( $\geq 99.5\%$ ), xylose ( $\geq 99\%$ ), fructose ( $\geq 99\%$ ), cellobiose ( $\geq 98\%$ ), arabinose ( $\geq 98\%$ ), 5-(hydroxymethyl)furfural (5-HMF,  $\geq 99\%$ ), furfural ( $\geq 98\%$ ) and glycol ( $\geq 99\%$ ) were purchased from Sigma–Aldrich.

### 2.2. Experimental procedure

The hydrolysis of bamboo biomass was carried out in a high pressure reactor (maximum working pressure 350 kg cm<sup>-2</sup> and temperature 500 °C, Model 1734, Amar Equipment Pvt. Ltd., India) equipped with a stainless steel 500 ml reaction vessel (160 mm height, 75 mm internal diameter). The reactor is equipped with a magnetic driven stirrer with 60 ml minimum stirring volume, temperature controller, and U-type internal cooling coil. The reactor was heated with the help of 2.0 kW electric heaters. Six HEX M8 × 70 screws which can afford 12.8 tons of tensile force were used for tightening the reactor with its cap. The hydrolysis process was run under batch mode. For SCW treatment, nitrogen gas (98% purity, purchased in Guwahati, Assam, India) was used to maintain the constant pressure inside the reactor. The bamboo hydrolysis process was performed between 170 °C and 220 °C (interval of 10 °C) for 5–40 min reaction time (time interval 5 min), at a pressure corresponding to the vapour pressure curve at a given temperature. The reaction content was mixed continuously via magnetic driven stirrer at desired temperature (Ozturk et al., 2010).

Experiments were performed in a 500 ml reaction vessel using 5 g of bamboo in 100 ml of deionised water and were kept constant unless otherwise stated. At the beginning to maintain the constant hydrolysate volume, an additional 20 ml of deionised water was added to the system considering the water loss due to evaporation (~10%) and entrainment in the biomass residue (~5%). Temperature inside the reactor was measured using a thermocouple and controlled at operating temperature. The pressure inside the reactor was maintained at 7.9, 10, 12.5, 15.5, 19 and 23.1 bar and the corresponding recorded temperatures were 170, 180, 190, 200, 210, and 220 °C, respectively, or slightly above by using nitrogen as a pressurised gas to ensure that water remained as liquid (Pinkowska et al., 2011; Tsigie et al., 2012). At these conditions, liquid samples were collected through sample outlet at different time intervals, and immediately quench to provide truly representative samples.

In case of biomass residue, samples were cooled up to room temperature within the reactor. Finally, treated biomass was collected and dried at 80 °C for 24 h. The treated dried samples were characterised by XRD, FTIR and SEM analysis. The collected liquid samples were analysed by HPLC. All the experiments were performed in duplicate, and the average values are reported. After

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