



An application study of autohydrolysis pretreatment prior to poplar chemi-thermomechanical pulping



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HIGHLIGHTS

- Poplar wood chips were autohydrolyzed prior to chemi-thermomechanical pulping.
- Effects of autohydrolysis on chemical compositions in the AHL were investigated.
- A 44% of the extraction yield for xylose from poplar wood chips can be obtained.
- Autohydrolysis pretreatment prior to CTMP was helpful to refining energy savings.
- Some properties of the resultant pulp made from the hydrolyzed chips were improved.

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ABSTRACT

This study investigated the autohydrolysis pre-treatment prior to chemi-thermomechanical pulping (CTMP) process including the effects of autohydrolysis pretreatment with a lower severity on characteristics of the autohydrolysis liquor and hydrolyzed chips. The intensity of autohydrolysis, characterized by the severity factor, increased from 1.76 to 3.54, the hydrolyzed chips yield decreased from 99.0% to 86.7% and the xylose extraction yield increased from 0.8% to 44.0%. The content of holocellulose, pentosan and acid soluble lignin remained in the hydrolyzed chips decreased dramatically with increasing the treatment severity. After the autohydrolysis pretreatment, the resultant poplar chemi-thermomechanical pulp with a severity factor of 2.37 could obtain a 15.3 N m/g tensile index, 58.6% ISO brightness and a 2.39 cm³/g bulk.

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1. Introduction

Pulp and paper industry is a huge consumer and a main producer of renewable lignocellulosic materials in the world. Chemi-thermomechanical pulping (CTMP) means to make chemi-thermomechanical pulp, and chemi-thermomechanical pulp is mechanically produced by treating wood chips with chemicals and steam, and then grinding the pre-treated chips in refiner for the defibration. It has become one of the key high-yield pulping technologies, possessing advantageous features of raw materials saving and environmental friendliness. However, during the high-yield pulping process, some carbohydrates are still released from the raw material into the process liquor. The pulping yield for this kind of high-yield pulping process is above 85%, which means that about 15% of materials dissolved in the production process. It is reported

that approximately 2–5% of hemicelluloses are dissolved and dispersed into process water in a mechanical pulping process (Boluk et al., 2008). The concept of the integrated forest biorefinery (IFBR) and the concept of value prior to pulping (VPP) both provide pulp and paper industry an excellent opportunity to increase additional revenue by producing high-value bio-based chemicals and materials including fuels, while continuing to produce its traditional pulp and paper products (van Heiningen, 2006; Thorp and Raymond, 2004; Zhu and Pan, 2010). Pre-extraction of lignocellulosic materials prior to pulping has been considered as an important step in the implementation of the IFBR (Thorp and Raymond, 2004; Moshkelani et al., 2013; Cheng et al., 2010).

Autohydrolysis is a process in which lignocellulosic materials are treated with hot water or steam, and it is an alternative process for the fractionation of lignocellulosic materials. During autohydrolysis, the depolymerization of hemicelluloses is catalyzed by hydrogen ions from water and naturally generated compounds such as acetic, uronic and phenolic acids (Liu, 2010). The generation of

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acetic acid was due to the cleavage of acetyl groups from xylan chains in wood and in solubilized xylo-oligosaccharides (Mittal et al., 2009a; Borrega et al., 2011; Li et al., 2010). Autohydrolysis can separate lignocellulosic materials into two fractions: an aqueous extract composed mainly of hemicellulose derivatives along with acid soluble lignin, and a residual solid consisting of cellulose, lignin and remaining hemicelluloses. So far, autohydrolysis treatment has been considered a cost-effective and environmentally-friendly technology of hemicelluloses extraction (Garrote et al., 2003; Yu et al., 2010), because it has many advantages, such as no additional chemicals (catalyst) needed, limited corrosion to equipment, lower capital costs related to the pretreatment and to the recovery of hydrolysates (Romani et al., 2010; Cybulska et al., 2010; Mosier et al., 2005). In recent years, all studies on the autohydrolysis pretreatment have been mainly focused on the kraft pulping process (Yoon and van Heiningen, 2008; Helmerius et al., 2010; Duarte et al., 2011; Vila et al., 2011; Chirat et al., 2012). However, there is only limited work about the autohydrolysis pretreatment prior to the production of mechanical pulps (Houtman and Horn, 2011), and no further publication on the autohydrolysis prior to the wood CTMP. It is expected that the autohydrolysis pretreatment prior to CTMP will be helpful not only to full utilization of wood raw materials but also to the high value-added utilization of the recovered hemicelluloses from the autohydrolysis liquor (AHL), and moreover, it will be helpful to reduction of the refining energy consumption in the hydrolyzed wood CTMP. The ideal autohydrolysis pretreatment should obtain high sugar concentrations at high pulp yields, while causing a minimal interference with the subsequent pulping process. However, it is reported that high amount of xylan with minimal degradation can be realized at the expense of decreases in pulp's strength such as tensile strength, tensile stiffness and burst strength (Helmerius et al., 2010).

The objective of the present study was to investigate the potential of wood hemicelluloses extraction through the autohydrolysis pretreatment followed by CTMP using the resultant autohydrolyzed wood chips. In this study, the effects of autohydrolysis pretreatment with lower severity prior to poplar CTMP on chemical compositions of the AHL and hydrolyzed chips were determined. The testing of properties of the yielded chemi-thermomechanical pulp was also performed to explore the practicability of the CTMP using the autohydrolyzed poplar wood chips.

2. Experimental

2.1. Materials

The poplar chips were obtained from a pulp mill in Shandong province, China. The chips were washed to remove barks and sand, air-dried, and then knots were removed by hand. The obtained chips were finally stored in a cool room at 4 °C. The chemical compositions of the poplar chips determined according to the methods of the US National Renewable Energy Laboratory (NREL) (Sluiter et al., 2008, 2012) are as follows: glucose 51.41%, xylose 19.48%, arabinose 0.45%, acetyl groups 5.64%, Klason lignin 22.54%, acid soluble lignin 2.42%, and ethanol extractives 1.86%. The chemical compositions including holocellulose, pentosan, lignin, and benzene-alcohol extractives of the poplar chips with and without autohydrolysis pretreatment were determined according to the literature (Shi and He, 2010), and the results are shown in Table 2.

All chemicals used in this study were analytical grade.

2.2. Handling of wood chips prior to autohydrolysis

Wood chips were firstly immersed in hot water at 90 °C for 30 min to imitate pre-steaming process in practical production

and then extruded once with a screw extruder (JS30, Shandong Wenrui Papermaking Machinery Co. Ltd., China) at a compression ratio of 4:1. The extruded chips were air-dried and collected in a polyethylene bag to equilibrate the moisture content for later use.

2.3. Autohydrolysis pretreatment

The extruded chips were treated in a 6 L M/K digester (M/K Systems Inc., USA) equipped with a centrifugal pump for liquor circulation and a proportional-integral-differential (PID) temperature controller. To reduce heat-up time, the deionized water to be used for autohydrolyzing was firstly poured into the digester and pre-heated in advance. After the temperature reached to 80 °C, an equivalent of 320 g (oven-dried) extruded chips was then added into the digester and mixed with the pre-heated deionized water at a liquid/solid ratio of 4:1 kg/kg (including the moisture content in the extruded chips). The autohydrolysis temperature was raised to the desired value using the heat exchanger equipped outside the digester at a rate of 2 °C/min. The autohydrolysis reaction was terminated by discharging the AHL through the heat exchanger to prevent the evaporation of volatile components (VC). After finishing the autohydrolysis pretreatment, the hydrolyzed chips were taken out of the digester and collected in a cloth bag and then washed thoroughly with deionized water until the filtrate was colorless and its pH was neutral. The hydrolyzed chips yield (i.e., the oven-dried weight of residues after autohydrolysis pretreatment divided by the oven-dried weight of the original wood chips), the pH of the collected AHL and chemical compositions of the hydrolyzed chips were determined. The AHL was made up of the following three parts: insoluble compounds, volatile components (VC) and non-volatile components (NVC). After 30 g of the AHL was centrifuged at 4500 rpm for 15 min to separate the insoluble compounds in order to obtain the insoluble precipitated lignin (Leschinsky et al., 2009), 15 g of yielded supernatant was taken and oven-dried at 105 °C to determine the total content of NVC, and the remaining supernatant was finally used for the analysis on VC and NVC using high-performance liquid chromatography (HPLC). Acetic acid and furfural, main components in the VC, were determined. The NVC in the AHL is composed mainly of monosaccharides, oligosaccharides, acetyl groups and a fraction denoted "others", calculated by difference respect to the total content of the NVC, including uronic substituents of oligosaccharides, phenolic compounds derived from the acid-soluble lignin fraction, and other extractives (Garrote et al., 2007).

In this study, we used the severity factor (expressed as $\log R_0$), which is proposed by Overend et al. (1987), to quantify the intensity of the hydrothermal treatment of biomass by the following expression:

$$R_0 = t \times \exp((T - 100)/14.75) \quad (1)$$

where t is the duration time in minutes, and T is the reaction temperature in °C.

It is clear that the severity factor depends on two process variables, namely the treatment temperature and duration time, and can be used as a critical parameter to control the changes in chemical compositions of the AHL and relevant hydrolyzed chips. The autohydrolysis conditions and the corresponding severity factor in the present study are listed in Table 1.

2.4. Chemi-thermomechanical pulping (CTMP) process

The poplar wood chips were extruded once in the screw extruder with a compression ratio of 4:1, and then air-dried and packed in a polyethylene bag for equilibrium moisture content. Following the extrusion treatment, impregnation treatment was performed in a polyethylene bag according to the following conditions: 220 g

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