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Mechanical pretreatment improving hemicelluloses removal from cellulosic fibers during cold caustic extraction

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HIGHLIGHTS

- A concept of combined refining and CCE for hemicelluloses removal was proposed.
- Refining changed fiber morphologies, facilitating hemicelluloses removal in CCE.
- The combined process decreased the alkali dosage/concentration in subsequent CCE.
- The combined process led to other advantages, such as high Fock reactivity.

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ABSTRACT

Hemicelluloses removal is a prerequisite for the production of high-quality cellulose (also known as dissolving pulp), and further recovery and utilization of hemicelluloses, which can be considered as a typical Integrated Forest Biorefinery concept. In this paper, a process of combined mechanical refining and cold caustic extraction (CCE), which was applied to a softwood sulfite sample, was investigated. The results showed that the hemicelluloses removal efficiency and selectivity were higher for the combined treatment than that for the CCE alone. The combined treatment can thus decrease the alkali concentration (from 8% to 4%) to achieve a similar hemicelluloses removal. The improved results were due to the fact that the mechanical refining resulted in increases in pore volume and diameter, water retention value (WRV) and specific surface area (SSA), all of which can make positive contributions to the hemicelluloses removal in the subsequent CCE process.

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1. Introduction

Lignocellulosic feed stocks for chemicals, materials and fuel have been the focus in recent years (Dashtban et al., 2014; Fatehi et al., 2013). The wood-based regenerated cellulose products which can be manufactured from dissolving pulps include viscose rayon, cellulose nitrate, and cellulose acetate, among others (Gübitz et al., 1997; Schild and Sixta, 2011). Also, the dissolving pulps can be superior raw materials for the production of nanofibrillation cellulose (NFC), nanocrystalline celluloses (NCC) and microcrystalline cellulose (MCC) (Wang et al., 2015; Sixta et al., 2013).

The well-commercialized dissolving pulps are derived from softwood, hardwood or mixtures of them based on the acid sulfite or pre-hydrolysis kraft (PHK) pulping process. A low hemicelluloses content is one of the important quality parameters for the dissolving pulps (Schild and Sixta, 2011; Miao et al., 2014). There have been many studies reporting the negative impact of hemicelluloses on the manufacturing processes of various products and the properties of resulting products, from dissolving pulps (Sixta et al., 2013; Wang et al., 2014). Moreover, the removed hemicelluloses can be recovered and further converted into other products, such as bio-ethanol, furfural, and xylitol (Shi et al., 2011; Shen et al., 2013; Panthapulakkal et al., 2015; Liu et al., 2013a).

In the literature, many studies have been devoted to the development of relevant technologies on the hemicelluloses removal. For example, Gehmayr and Sixta (2012) investigated the hemicelluloses removal from a softwood sulfite pulp by using cold caustic extraction (CCE). Liu et al. (2013b) examined the effect of alkaline treatment on the pentosan content of a sugarcane bagasse. Hakala

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et al. (2013) studied the enzyme-aided caustic extraction of xylan from a hardwood kraft pulp.

It is well known that CCE is very effective in removing the hemicelluloses, with a typical alkali concentration of 8–10% (Sixta, 2006). The CCE process in removing the residual hemicelluloses from pulp fibers can be divided into two stages: (1) physical interaction between fiber and aqueous sodium hydroxide resulting in fibers swelling; (2) diffusion of hemicelluloses, from fibers interior to exterior, and finally into bulk phase, through the pores in the fiber wall (Sixta, 2006). However, during a typical CCE stage (with its NaOH concentration of 8% or higher), the gradual transition of cellulose lattice from cellulose I (native cellulose) converting into Na-cellulose, and finally into cellulose II (regenerated cellulose), can take place (Schild and Sixta, 2011; Sixta et al., 2013). Thus, upon drying, the reactivity of cellulose decreases due to the very compact fiber structure of cellulose II (with extensive inter-molecule hydrogen bonding). In addition, it was known that it was a challenge in removing residual alkali from the highly swollen pulp after a CCE process, which consequently leads to high capital investment (effective multiple post-washers would have to be installed), and high chemical consumption (Gehmayr and Sixta, 2012).

In fact, CCE is a very selective process in removing hemicelluloses from pulp fibers (Gehmayr and Sixta, 2012; Sixta, 2006), due to the excellent swelling of fibers and solubility of hemicelluloses under the conditions. The hemicelluloses removal in the CCE process is mainly influenced by the fiber morphology/structure, which can be modified effectively by mechanical treatments, such as pulp refining, as reported in the literature. For example, it was shown that the accessibility of lignocellulosic materials to macromolecules, such as enzymes, can be improved by using pulp refining (Suurnäkki et al., 1996). In this study, the performance of combined mechanical refining and CCE in removing the hemicelluloses from a softwood sulfite pulp was investigated. Firstly, the mechanical refining was applied to pulp fibers by using a PFI refiner. The changes in fiber morphologies, including pore volume, pore size, specific surface area (SSA) and water retention value (WRV), were determined. Subsequently, the efficiency and selectivity of hemicelluloses removal from the mechanically refined pulp during the CCE process were investigated.

2. Methods

2.1. Materials

A softwood acid sulfite pulp was provided from a mill in Canada. A Bauer-McNett fiber classifier (MC Tec Co., Ltd., Giessen, Netherlands) was used to obtain those that were retained on the 30-mesh screen, and they were the raw material used in this study.

2.2. Mechanical treatment and cold caustic extraction

The mechanical treatment was performed by using a PFI refiner according to Tappi standard T 248 sp-08. A 30 g (equivalent to oven dried) pulp was refined at a 10% pulp consistency for 10,000 revolutions. Subsequently, a cold caustic extraction (CCE) was carried out. A 20 g (equivalent to oven dried) pulp was treated at a 4% or 8% NaOH concentration with other conditions being 10% pulp consistency, 25 °C and 30 min in polyethylene bags.

2.3. Analyses

2.3.1. Pore volume and diameter

The pore volume and median pore diameter of samples were measured by using the solute exclusion method (Stone and Scallan, 1968a,b). The solute molecules used were a glucose (alpha-D-glucose) and a series of dextran fractions from Sigma–Aldrich Chemical Co., USA. The relationships between molecular diameter and molecular weight for glucose and dextran were listed in Table 1 (Hui et al., 2009). The experimental procedures and data analyses method followed Stone and Scallan (1968a). The total pore volume was the inaccessible pore volume for the largest probe molecule (dextran with the diameter of 56 nm) used in this study, which was as $V_{inac,56}$. The median pore diameter was the pore size at which one half of the pore volume was contained in larger pores and one half in smaller pores (Stone and Scallan, 1968b). The accessible pore volume for a given probe molecule, $V_{ac,m}$, was simply that the largest inaccessible pore volume, $V_{inac,56}$ (in this study), subtracted the inaccessible pore volume, $V_{inac,m}$ (Wang et al., 2012). Therefore,

$$V_{ac,m} = V_{inac,56} - V_{inac,m} \quad (1)$$

2.3.2. Water retention value

The water retention value (WRV) of samples was determined based on a method in the literature (Hui et al., 2009), by using a laboratory centrifuge at 900*g* for 30 min.

2.3.3. Specific surface area

The specific surface area (SSA) of samples was determined based on the nitrogen adsorption method by using a Belsorp-Max volumetric gas adsorption instrument (Bel Japan, Inc., Osaka, Japan) (Tian et al., 2014). The samples were diluted to 0.5% pulp consistency to ensure in an adequate dispersion of pulp fibers, subsequently freeze–dried prior to the measurement.

2.3.4. Hemicelluloses content

The hemicelluloses content of samples, namely, the dissolved carbohydrates content in the 18% NaOH solution (known as the S_{18}), was determined by following Tappi T 235 cm-09.

2.3.5. Pulp yield and hemicelluloses removal selectivity

The samples were collected and weighted before and after a caustic treatment. The mass ratio of samples before and after a caustic treatment was calculated as the pulp yield. After a caustic treatment, the hemicelluloses removal selectivity was calculated as followed:

$$\text{Hemicelluloses removal selectivity } (\%) = \frac{m_i * C_i - m_f * C_f}{m_i - m_f} \times 100 \quad (2)$$

where m_i and C_i are the initial sample weight and hemicelluloses content, respectively, and m_f and C_f are the sample weight and hemicelluloses content after a caustic treatment, respectively (Li et al., 2015).

2.3.6. Fock reactivity

The Fock reactivity of samples was determined based on an improved method (Tian et al., 2013). The samples were air-dried prior to the measurement.

Table 1
Molecules and their molecular weights and diameters (Hui et al., 2009).

Probe molecules	Molecular weight	Diameter (nm)
Dextran T2000	2,000,000	56
Dextran T500	500,000	27
Dextran T1390	73,000	12
Dextran T40	40,000	9.0
Dextran T9260	9300	4.6
Glucose	180	0.8

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