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Effects of hemicellulose pre-extraction and cellulose nanofiber on the properties of rice straw pulp

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ABSTRACT

The aim of this study was to evaluate the influence of mild alkaline pre-extraction on the hemicelluloses removal of rice straw. In addition, the performance of cellulose nanofibers (CNFs) addition on some physico-mechanical properties obtained from extracted and un-extracted (control) samples were investigated. An optimum compromise was found as operating conditions (10% soda, 50 °C, and 90 min) that provided moderate hemicelluloses removal (48.1%). The residual extracted rice straw particles were subjected to soda–anthraquinone pulping at 160 °C for 30–60 min with 6 to 18% active alkali charge. Compared with the control samples, the screened yield and Kappa number for extracted pulps decreased slightly. Extracted samples showed better air resistance compared with the un-extracted (control) pulps. However, pre-extraction was found to negatively impact on some mechanical properties such as decrease in burst and tensile indices while addition of CNFs and refining of fibers could improve the strength properties considerably. In general, the addition of CNFs in any concentrations considerably enhanced tensile and burst indices of the sheets in extracted and un-extracted conditions, compared with the unfilled ones. The improvement in mechanical properties was considered one of the key benefits brought by CNFs reinforcement. On the other hand, the stretch properties of the beaten pulps are higher than those of control samples. SEM observations showed that CNFs were deposited on the voids between the rice straw fibers.

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1. Introduction

Traditionally, pulp is the main product of pulp mills, which is used for the production of various paper grades [1]. However, the rising cost of energy and the tight competition for low-cost products, made the pulp and paper mills to seek for new products, new strategies, and new business areas [2]. The application of forest biorefinery concept has been proposed as a promising approach to overcome this difficulty [3]. In biorefinery, the hemicelluloses are either partially or completely extracted out for generation of high value by-products [4]. The extracted hemicellulose can be used directly in polymeric form for novel industrial applications such as biopolymers, hydrogels, or thermoplastic xylan derivatives; or if hydrolyzed, they can serve as a source of sugars for fermentation to fuels, such as ethanol, or chemicals, such as 1,2,4-butanetriol [5]. Moreover, extracting hemicelluloses prior to pulping can benefit energy recovery from pulping black liquors since the heating value of hemicelluloses, about 13.6 MJ/kg, is only about half of that of lignin [4].

There are several pre-treatment technologies suggested for the removal of hemicelluloses from woody material (biomass) such as steam explosion, organic solvents, dilute acid, autohydrolysis, enzyme, and alkaline treatment [6]. In selecting the optimal methods to remove the hemicelluloses from wood prior to pulping, both economical and technological aspects have to be taken into account [7]. For instance, minimal interference with the subsequent pulping process is required since the production of high-quality papermaking fibers at high yield will remain the main concern for pulp and paper mills. Hemicellulose extraction will affect the mill production in different ways; for example, the severe conditions in hot water and dilute acid extraction can have a negative impact on the fibers [8]. The cellulosic fibers can possibly be damaged, resulting in

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a reduction in the degree of polymerization (DP) of cellulose, pulp yield loss, and/or lower strength properties of paper. In contrast, under alkaline conditions, extraction of hemicelluloses has been proven technically viable to a sufficient extent [5]. Hemicelluloses showed high solubility under alkaline conditions and no pH adjustment or water washing is required for subsequent alkaline pulping. Besides, alkali extractions generally require temperatures below 90 °C to minimize carbohydrate modification reactions. Alkaline extraction of hemicelluloses from aspen hardwood chips prior to kraft pulping using 1–2 M NaOH at 50–90 °C provided about 20–25% of hemicelluloses while the pulp yield was maintained, although the reduction in pulp viscosity was observed [9]. Thus, the pre-extraction of hemicelluloses and subsequent pulping process need to be carefully optimized to maintain pulp yield and quality [2]. The application of dry–strength additives has been a successful method to enhance the strength properties of paper. The wood fibers contain their own natural dry–strength additive in the form of hemicelluloses. The hemicelluloses removal from pulps makes it more difficult to develop their bonding characteristics. Various additives have been used throughout the years but a tendency towards the application of cellulose nanofibers (CNF) has only recently been observed. CNFs are abundant biodegradable polymers. Their nanostructures have widths ranging from 5 to 30 nm and are highly crystalline, due to the lateral packing of long cellulose molecules with hydrogen bonding and hydrophobic interaction between the cellulose chains [10]. The resultant stable structure shows high strength, high work of fracture, low moisture adsorption, low thermal expansion, high thermal stability, exceptional barrier properties, and high optical transparency [11]. CNFs can be used in several applications such as reinforcement in biocomposites, strength additive in papermaking, production of hydrogel, anti-microbial films, and high-technology devices [12].

In this study, mild alkaline pre-extraction of rice straw (an agro-residual) was carried out in order to recover the hemicelluloses based on a biorefinery concept. Subsequently, the extracted residues and an un-extracted sample (control) were subjected to soda–anthraquinone (soda–AQ) pulping. In order to compensate for the adverse effect of hemicelluloses extraction on the paper properties, the effects of addition of CNFs on some mechanical properties were also investigated and compared with the control sample.

2. Materials and methods

2.1. Materials

Rice straw was collected from Mazandaran province (Iran) and cut to 5–7 cm in length. It was washed, air-dried and screened to remove dirt and soil. For the chemical analysis, the rice straw was then ground using a Wiley mill and the fraction passing through 2 mm holes was stored in plastic bags for experiments.

CNF gel (3 wt%) was supplied by Nano Novin Polymer Co. (Iran). All other reagents and solvents were of analytical grade and were used without further purification. For film formation, mixed cellulose ester (CEM) membrane filter with diameter of 9 cm and pore size of 0.2 μm was used (Toyo Roshi Kaisha Co., Japan). All stock solutions used in this work were freshly prepared to avoid any possible degradation.

2.2. Chemical and morphological characterizations

The chemical characterizations of rice straw fibers were carried out following the appropriate Tappi Test Methods and the other published procedures as indicated. The samples were subjected to the following determinations: holocellulose (T 249 om-88), α-cellulose (T 203 cm-99), Klason lignin (T 222 om-88), hot-water solubility (T 207 cm-88), ash (T 211 om-88) and sugars (T 249 cm-00). Table 1 shows the mean and standard deviations of three replicate determinations.

Table 1
Comparison of chemical compositions of the used rice straw and some agro-residues materials.

Components (%)	Rice straw†	Bagasse‡	Cotton‡	Tobacco‡
Holocellulose	73.4 ± 3.3	73.7 ± 4.3	64.9 ± 3.1	69.1 ± 3.8
α-Cellulose	46.6 ± 3.7	40.5 ± 3.2	36.0 ± 2.2	37.9 ± 4.3
Hemicellulose	26.7 ± 4.2	33.2 ± 5.3	28.8 ± 4.4	31.2 ± 3.1
Pentosans	21.7 ± 0.2	13.7 ± 2.1	16.9 ± 2.4	15.7 ± 2.2
Klason lignin	20.9 ± 2.8	27.7 ± 3.5	26.3 ± 5.2	24.5 ± 3.1
Hot-water solubility	8.4 ± 1.3	6.5 ± 0.6	15.2 ± 1.6	18.9 ± 1.4
Ash	10 ± 1.2	4.8 ± 1.9	4.5 ± 2.0	12.5 ± 4.3
Arabinose	2.9 ± 0.1	2.2 ± 0.5	0.5 ± 0.05	–
Mannose	0.21 ± 0.01	5.7 ± 1.0	2.9 ± 0.4	1.7 ± 0.7
Galactose	1.05 ± 0.02	2.6 ± 0.4	0.8 ± 0.2	3.2 ± 0.8
Glucose	46.6 ± 0.6	76.9 ± 5.2	64.7 ± 3.9	78.1 ± 8.2
Xylose	21.7 ± 0.2	14.5 ± 2.1	12.6 ± 2.7	14.8 ± 3.9
Rhamnose	0.13 ± 0.1	0.4 ± 0.4	–	3.8 ± 0.9

† Present study.
‡ Ashori et al. [14].

The samples for fiber maceration were prepared according to the modified Franklin method, as explained elsewhere [13]. Fiber length (L), fiber diameter (d), cell wall thickness (w), and lumen width (l) were measured directly from the magnified image, with 100 measurements made on each property.

2.3. Alkaline pre-extraction

The pre-extraction was carried out in an autoclave. The extraction conditions were as follows: alkaline charge 10, 12 and 18%, temperature 50, 60 and 70 °C and hold time 60, 70 and 90 min after the set temperature was reached. The solid to liquor (S:L) ratio was fixed at 1:4 (w:w) and held constant in all experiments. The rice straw used for each experiment was 50 g based on oven dry (OD) weight. At the end of treatment, the solid residue was recovered by filtration and washed with distilled water. The solid fraction was used for soda–AQ pulping process. Soluble hemicelluloses liquor was precipitated by adding ethanol and collected after centrifugation.

2.4. Soda–AQ pulping

Pulping experiments were carried out with 100 g of OD extracted and un-extracted (control) rice straw using a 10-L batch cylindrical reactor. The pulping conditions were as follows: initial S:L 1:4, AQ 0.1%, time to maximum temperature 90 min, maximum temperature 160 °C and pulping time at maximum temperature 30, 45, and 60 min. The amount of active alkali (NaOH) charge was varied from 8 to 18% based on OD. After cooking, the pulps were thoroughly washed with fresh water on a fine filter, and then disintegrated in a hydropulper at 30,000 revolutions and 0.5% consistency for 5 min (T 205 om-88). For each raw material (extracted and un-extracted samples), the screened yield and Kappa number (T 236 cm-85) were determined.

Beating was included as a variable in the investigations of the extracted and un-extracted pulps. After determination of the initial properties of pulps, two pulps, which had better quality in terms of yield and Kappa number, were chosen for the beating studies. The beating studies were conducted in a PFI mill according to T 248 sp-00. The amount of pulp sample taken for one run was 20 g (OD basis) at 10% consistency, and no load power was applied. The freeness of the unbeaten and beaten pulps measured in this study was 600 and 300 mL CSF, respectively.

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