Staged Alkali and Hydrogen Peroxide Treatment of Poplar Chemi-Mechanical Pulp

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In order to improve the physical properties and brightness of poplar chemi-mechanical pulp, a new staged alkali and hydrogen peroxide treatment method was proposed and applied. Wood chips were impregnated and swelled with an alkali solution and then treated with a hydrogen peroxide bleaching liquor. A thorough evaluation and comparison of the physical properties and brightness of the pulps that underwent different treatment methods was conducted. The results showed that when the pulp was treated with an alkali and hydrogen peroxide treatment method with an alkali dosage of 6% and a hydrogen peroxide dosage of 6%, the tear index was 3.64 mN·m^2/g, the tensile strength was 3.61 kN/m, and the pulp brightness was 67.1% (ISO). The obtained physical properties and brightness of the alkali and hydrogen peroxide method treated pulp were greater than the traditional alkaline hydrogen peroxide method values, as well as the values of any other single treatment methods.

Keywords: Mechanical pulp; Alkaline hydrogen peroxide pretreatment; Physical properties; Brightness

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INTRODUCTION

Chemi-mechanical pulp is widely utilized in the production of various papers and paperboards due to its excellent fiber properties, such as high opacity, high bulk, high yield, and outstanding printing properties (Khakifirooz *et al.* 2013; Rainey and Covey 2016; Huang *et al.* 2017). For instance, newsprint could be produced from chemi-mechanical pulp, due to its high opacity and lower brightness requirements (Barimani *et al.* 2014). Good absorbent and high bulk properties makes it a good source for household paper (Chang *et al.* 2018). In addition, high-grade coated paper can also be produced by replacing part of the chemical pulp with chemi-mechanical pulp. Therefore, the treatment process for forming a wide range of characteristics from chemi-mechanical pulp fibers is very important. Moreover, selecting inexpensive chemicals and a suitable treatment method also improves the reaction efficiency at the treatment stage. It remains a challenge to reach an optimum efficiency with a shorter reaction time and lower chemical dosages. As a result, an effective chemical treatment is required to swell the wood chips, enhance the bonding strength between the fibers, and improve the physical strength and optical properties of the fibers (Lei *et al.* 2013).

Currently, various bleaching chemicals, such as hydrogen peroxide, sodium dithionite, sulfite, peracetic acid, sodium borohydride, *etc.*, have primarily been used for industrial pulping purposes (Suess 2009, 2010; Nguyen 2011). Among these bleaching chemicals, sodium borohydride and peracetic acid are expensive and not conducive to reducing manufacturing costs (El Shafie *et al.* 2009; Istek and Gonteki 2009). In addition, sodium dithionite solutions are unstable in the air and easily react with metals to form sulfides, which are corrosive (Narvestad *et al.* 2013). Sodium sulfite was first used in the mechanical pulp bleaching process to prevent the pulp from darkening in the refining process. However, it had greater performance when sulfonating wood chips to improve the strength properties of chemi-mechanical pulp (Jurd 1964). Hydrogen peroxide is widely available due to its conspicuous bleaching efficiency and environmental friendliness (Bajpai 2012). Therefore, in newer bleaching technologies, an alkaline hydrogen peroxide treatment is widely applied, since it not only enhances the physical strength of the chemi-mechanical pulp, it also improves the optical properties of the fibers, which helps meet the requirements of various paper products. During an alkaline hydrogen peroxide treatment, alkali plays a crucial role by swelling the wood chips, softening the fibers, and improving the strength properties of the paper. An alkali treatment primarily removes a portion of the hemicellulose and lignin content. It also cause swelling in the fine cellulose structures, and thus weakens the hydrogen bonds between the fibers, which increases the porosity of the cell wall. Finally, these treatments enhance the reaction area on the fibers accessible to the alkali solution (Cuissinat *et al.* 2008; Le Moigne and Navard 2010; Lund *et al.* 2012). However, in the actual industrial process, in order to reduce the time spent introducing individual chemicals, all the chemicals are delivered at one stage in the chemical treatment. This leads to an excessive mixing of chemicals, and an ineffective decomposition of hydrogen peroxide and alkaline darkening, which reduces the bleaching efficiency of hydrogen peroxide. Hence, there are several major aspects still to be resolved, such as ensuring the bleaching efficiency with an appropriate alkali dosage, avoiding darkening during the alkali treatment, ensuring optimization of both the physical properties and brightness, *etc*.

In this study, an optimized treatment method for poplar chemi-mechanical pulp was developed, in which the wood chips were first fully impregnated with an alkali solution, and then treated with a hydrogen peroxide bleaching liquor. The processing conditions of the staged alkali and hydrogen peroxide treatment (A-P treatment) were optimized with regards to the physical properties and brightness of the chemi-mechanical pulp. Compared to the traditional alkaline hydrogen peroxide treatment (AP treatment), the optimized A-P treatment could more effectively improve the physical strength and brightness of the chemi-mechanical pulp.

EXPERIMENTAL

Materials

The poplar wood chips used in the experiments were obtained from Shan Dong Sun Paper Industry Joint Stock Co., Ltd. (Jining, China). Chip were selected to have a length of 20 mm to 25 mm, a width of 15 mm to 20 mm, and a thickness of 3 mm to 5 mm. The selected chips were air dried and stored at room temperature. The chemical components were measured according to the methodology in the NREL/TP-510-42618 protocol (Sluiter *et al*. 2012), and the results are shown in Table 1.

Table 1. Chemical Components of the Poplar Wood Chips

Arabinose (%)	Galactose (%)	Glucose (%)	Xylose (%)	Mannose (%)	Acid-insoluble Lignin (%)	Acid Soluble Lignin (%)
2.88	3.69	46.6	16.8	3.49	18.71	3.51

Comparison Experiment

Various treatment methods were performed and compared in order to optimize the bleaching process utilizing poplar chemi-mechanical pulp. In summary, the chemical treatment methods in this study contained: (a) a single alkali treatment process (A); (b) a single hydrogen peroxide treatment process (P); (c) the traditional alkaline hydrogen peroxide treatment process (AP); and (d) the staged alkali and hydrogen peroxide treatment processes (A-P), *i.e.*, wood chips were submerged in an alkali liquor first and then washed with tap water until neutral, and subsequently, the washed chips reacted with the hydrogen peroxide (with a liquor ratio of 1:4). In addition, the chemical dosages (based on the weight percentage of the dry poplar wood chips) employed in different treatment methods were as follows: (a) A treatment, with an alkali dosage of 3%, 4%, 5%, 6%, or 7%; (b) P treatment, with a hydrogen peroxide dosage of 4%, 5%, 6%, or 7%; (c) an AP treatment, with a 6% H_2O_2 solution and an alkali dosage of 3%, 4%, 5%, 6%, or 7% and with a 6% NaOH solution and a hydrogen peroxide dosage of 4%, 5%, 6%, or 7%; (d) A-P treatment, with the first stage alkali dosage either 3%, 4%, 5%, 6%, or 7%, the second stage hydrogen peroxide dosage at 6%; or the first stage alkali dosage at 6%, and the second stage hydrogen peroxide dosage either 4%, 5%, 6%, or 7%. The other treatment conditions used in the experiments were as follows: a temperature of 90 °C, a reaction time of 60 min, a 3% Na_2SiO_3 solution, a 0.5% $MgSO_4$ solution, and a 0.5% ethylene diamine tetraacetic acid (EDTA) solution. All these fixed treatment conditions were chosen according to preliminary experiments in this work, the results of previous studies, and the practical application of pulping industry that comprehensively considered the cost and efficiency. Each experiment was carried out at least in triplicates to ensure the accuracy of results.

Experimental Procedure

To maximize the effects of the alkali and/or hydrogen peroxide treatments, the whole experimental procedure was divided into three steps, which included: (1) a chemical treatment, (2) a mechanical treatment, and (3) paper sheet preparation. Step 1 was performed in a water bath, in which 100 g of air-dried wood chips were fully submerged into 400 mL of liquor solution, with various amounts of alkali, hydrogen peroxide, and tap water at 90 °C. For step 2, a high consistency continuous disc mill (BR30-300C, Kumagai Riki Kogyo Co., Ltd, Tokyo, Japan) was used for the mechanical treatment. After being chemically treated, the unwashed chips were refined, which was divided into a rough grinding section and a fine grinding section. For the rough grinding section, the chips were fiberized with a refiner plate gap of 0.5 mm. A refiner plate gap of 0.2 mm was used in the fine grinding section to subdivide the long fibers into short fibers and facilitate the fibrillation of the fibers. Afterward, the refined fibers were passed through a 0.2 mm sieve gap to obtain the accepted pulp. A prefiner (PFI) beating process (No. 658, PFI A/S Løten, Norway) was adopted with the conditions as follows: a pulp consistency of 10%, a refining pressure of 3.33 N/mm, and the gap between the beating plate and the inner wall was 0.18 mm. Following the PFI treatment, the paper sheets were prepared according to the TAPPI

T525 om-6 (2006) standard method for the subsequent measurements of the properties of the paper.

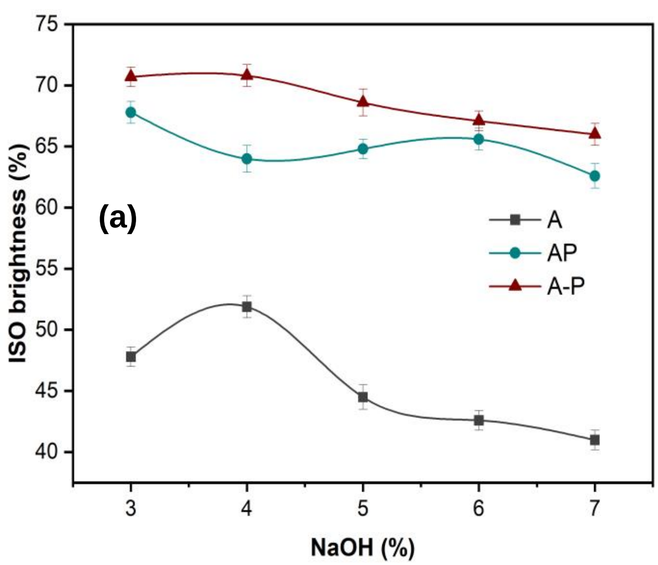
Analysis and Measurement

All the measurements were tested at least five times to ensure that the measurements were accurate. The handsheets, with a basic weight of 70 g/m^2, were prepared for the subsequent measurements of their physical properties and brightness (*et al*. 2011). The brightness of the handsheets was measured according to the TAPPI standard T525 om-06 (2006). The most important properties to evaluate the physical strength were the tensile strength and the tear index. The tensile strength that more likely to be correlated with fiber refining intensity was measured according to the TAPPI standard T494 om-01 (2001). The tear index that more strongly associated with fiber length was measured according to the TAPPI standard T414 om-04 (2004).

RESULTS AND DISCUSSION

Bleaching Efficiency of Various Treatment Methods

The brightness of the pulp obtained under various treatment methods is shown in Fig. 1. As shown in Fig. 1a, the brightness of the pulp treated with the A-P method was remarkably higher than the pulp treated with either the single A process or the traditional AP process. The brightness of the pulp treated using the A method increased initially with an increase in alkali dosage, but it decreased sharply when the alkali dosage was increased, which was most likely due to the darkening during the alkali treatment (He *et al*. 2005). Unlike the A treatment, the brightness of the pulp treated with the AP treatment was clearly higher, with a brightness of 67.8% ISO with an alkali dosage of 3%.



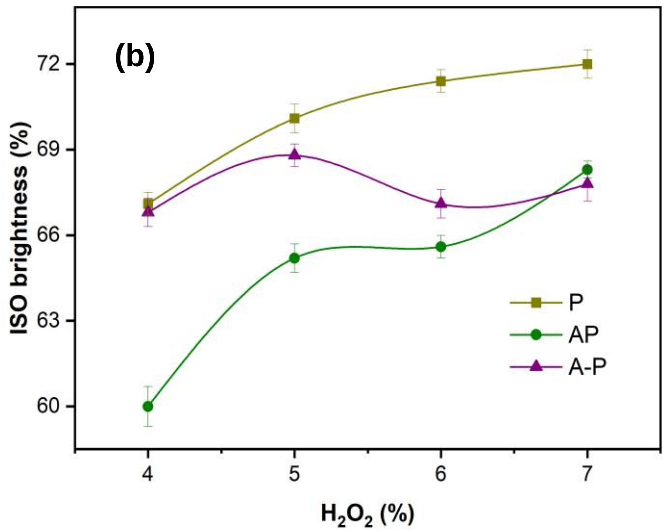


Fig. 1. Bleaching efficiency with different treatment methods under (a) different alkali dosages, the hydrogen peroxide dosage of the A, AP, and A-P treatments were 0%, 6%, and 6%, respectively; and (b) different hydrogen peroxide dosages, the alkali dosage of P, AP, and A-P treatments were 0%, 6%, and 6%, respectively. The other treatment conditions were the same: a temperature of 90 °C, a reaction time of 60 min, a 3% Na_2SiO_3 solution, a 0.5% $MgSO_4$ solution, and a 0.5% ethylene diamine tetraacetic acid (EDTA) solution.

For the traditional AP process, the results demonstrated that an alkali dosage of 3% was more suitable for the bleaching reaction and had the highest hydrogen peroxide bleaching efficiency; however, with a further increase in alkali dosage, the pulp brightness gradually decreased, which indicated that an excessive alkali dosage reduced the bleaching efficiency of hydrogen peroxide. The brightness of the pulp treated with the staged A-P treatment was more preferable than the pulp of other treatment methods. For example, when the alkali dosage was 3%, the brightness of the pulp that underwent the A-P method treatment was 70.7% ISO, which was higher than the pulp treated with the AP method. This may be explained by the fact that after the alkali impregnation of the poplar wood chips, some extracts and lignins were removed. Therefore, the reaction area between the fiber and the bleaching solution was improved, which was conducive to the subsequent hydrogen peroxide bleaching (Song *et al.* 2012).

As shown in Fig. 1b, with an increase in the hydrogen peroxide dosage, the brightness of the pulp treated with the P method was higher than that of the pulp treated with the A-P method, while the brightness of the pulp treated with the A-P method was better than the pulp treated with the AP method. For example, when the hydrogen peroxide dosage was 6%, the brightness of the pulp from the P, AP, and A-P treatments was 71.4% ISO, 65.6% ISO, and 67.1% ISO, respectively. The brightness of the pulp from the P treatment was superior to the other treatment methods, which was most likely due to the alkali treatment leading to darkening during the alkali treatment process (Kutney and Evans 1985). It was clearly observed that the brightness of the pulp from the A-P treatment and AP treatment gradually improved with the addition of hydrogen peroxide. It was also observed that the brightness of the pulp from the A-P treatment, which had little fluctuation and remained at a high level, was higher than the AP treatment under the same chemical dosages. As demonstrated by the results, a temperature of 90 °C and an alkali dosage of 6% was the optimum technological conditions for comprehensive consideration. In summary, the brightness of the pulp from the A-P treatment was remarkably better than the pulp from the AP treatment, not only with various alkali dosages, but also with different hydrogen peroxide dosages.

Physical Properties of Poplar Pulp after Various Treatment Methods

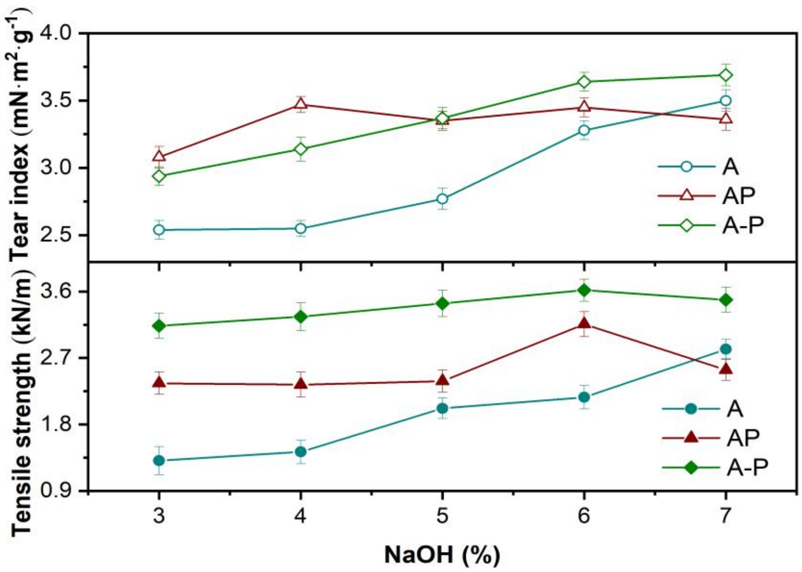


Fig. 2. Physical properties under different alkali dosages and other conditions: a 6% H_2O_2 solution, a temperature of 90 °C, a reaction time of 60 min, a 3% Na_2SiO_3 solution, a 0.5% $MgSO_4$ solution, and a 0.5% EDTA solution

Figure 2 shows the changes in the tear index and tensile strength after various alkali dosages during the A, AP, and A-P treatment methods. As shown in Fig. 2, the tear index of the chemi-mechanical pulp treated with the A treatment and the A-P treatment was improved with an increased alkali dosage. When the alkali dosage was increased from 3% to 7%, the tear index of the AP treated pulp increased from 3.08 mN•m^2/g to 3.36 mN•m^2/g, which showed a lower growth rate. This was attributed to the fact that the alkali dosage for the AP treatment was excessive, and the degree of damage to the fibers was enhanced by the superposition of the alkali and hydrogen peroxide. It was observed that the tear index of the A-P treated chemi-mechanical pulp surpassed the AP treated pulp when the alkali dosage was greater than 5%. Furthermore, when the alkali dosage was higher than 5%, the tear index of the A-P treated pulp increased demonstrably with a further increase in alkali dosage, which was much greater than the pulp of other treatment methods, especially the tear index at a 7% alkali dosage (3.69 mN•m^2/g). Moreover, as the alkali dosage increased, the tensile strength of the A-P treated pulp was notably greater than the AP treated pulp, while the tensile strength of the AP treated pulp was greater than the A treated pulp. In addition, when the alkali dosage was 6%, the tensile strength of the A-P treated pulp was 3.64 kN/m, which was preferable over the pulp from other treatment methods. Consequently, by combining the tear index and the tensile strength results from different alkali dosages, it was observed that the physical strength properties of the A-P treated pulp was obviously better than the pulp from other treatment methods.

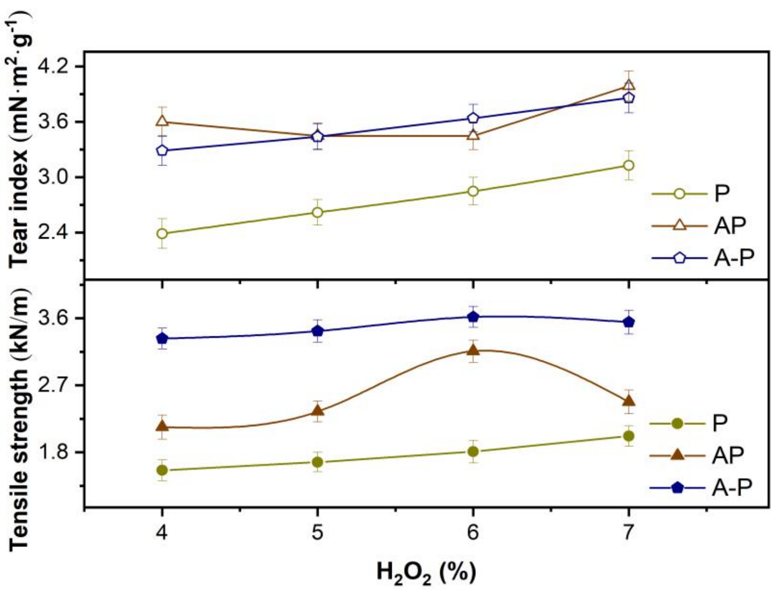


Fig. 3. Physical properties under different hydrogen peroxide dosages and other conditions: a 6% H_2O_2 solution, a temperature of 90 °C, a reaction time of 60 min, a 3% Na_2SiO_3 solution, a 0.5% $MgSO_4$ solution, and a 0.5% EDTA solution.

Figure 3 shows the physical properties after various treatments with different hydrogen peroxide dosages. As shown in Fig. 3, the tear index of pulp after an A-P treatment and a P treatment linearly increased with an increase in hydrogen peroxide dosage, while the tear index of pulp after an AP treatment showed a large fluctuation. Moreover, the tear index of A-P treated pulp was greater than the other treatment methods, and when the hydrogen peroxide dosage was 6%, the tear index of A-P and AP treated pulps were 3.64 mN•m^2/g and 3.45 mN•m^2/g, respectively. In addition, when the hydrogen peroxide dosage was less than 6%, the tensile strength of the A-P, AP, and P treated chemi-mechanical pulp improved as the hydrogen peroxide dosage increased. Among them, the tensile strength of the A-P treated pulp was more preferable over the other treatment methods. For example, when the hydrogen peroxide dosage was 6%, the tensile strength of the A-P and AP treated pulps were 3.62 mN•m^2/g and 3.16 mN•m^2/g, respectively. Therefore, the tensile strength of the A-P treated chemi-mechanical pulp was higher than the AP treated pulp. This was thought to occur because the wood chips swelled after the alkali treatment, which likely increased porosity, thus improving the bleaching efficiency of the wood chips with hydrogen peroxide and the bonding strength of the handsheets (Kong *et al*. 2005). Compared to the traditional treatment methods, the physical strength of A-P treated pulp was remarkably better than the AP treated pulp.

Comparison of the A-P Treatment and the Traditional Two-stage Treatment Methods

The physical properties and brightness of the mechanical pulps treated *via* the staged A-P method and the traditional two-stage method were investigated, and the results are shown in Table 2. A traditional two-stage treatment method meant that in at least one of the stages the chemicals (NaOH and H_2O_2) was delivered simultaneously at each stage. Runs 1 through 3 maintained the same amount of hydrogen peroxide, however they employed different alkali dosages. As shown in Table 2, when the treatment conditions were A 3/3 and P 0/6 (Run 2, first stage NaOH 3% / H_2O_2 0%; second stage NaOH 3% /

H_2O_2 6%), the tear index was 3.54 mN•m^2/g, the tensile strength was 3.61 kN/m, and the pulp brightness was 67.5% ISO. Runs 4 through 6 maintained the same alkali dosage while using various hydrogen peroxide dosages during the two stages. When the treatment conditions were A 3/3 and P 2/4 (Run 4), the tear index was 3.35 mN•m^2/g, the tensile strength was 3.75 kN/m, and the pulp brightness was 69.3% ISO. In contrast, when the pulp was treated with the new staged A-P method with a first stage alkali dosage of 6% (without H_2O_2), and then a second stage 6% hydrogen peroxide dosage (without NaOH), the tear index was 3.64 mN•m^2/g, the tensile strength was 3.62 kN/m, and the pulp brightness was 67.1% ISO (Run 7). Therefore, the physical properties and brightness of the staged A-P treatment pulp were superior to those of the other treatment methods.

Table 2. Physical Properties of the A-P Treated Pulp and Traditional Two-stage Alkaline Hydrogen Peroxide Treated Pulp

Run Number	NaOH (%)	H_2O_2 (%)	Tear Index (mN·m^2·g^{-1})	Tensile Strength (kN·m^{-1})	Brightness (% ISO)
1	2/4	0/6	3.03 ± 0.13	3.12 ± 0.12	66.1 ± 0.4
2	3/3		3.54 ± 0.12	3.61 ± 0.1	67.5 ± 0.6
3	4/2		3.34 ± 0.1	3.17 ± 0.14	68.4 ± 0.7
4	3/3	2/4	3.35 ± 0.13	3.75 ± 0.12	69.3 ± 0.5
5		3/3	3.24 ± 0.15	3.66 ± 0.15	65 ± 0.6
6		4/2	3.04 ± 0.16	3.48 ± 0.13	64.6 ± 0.5
7	6/0	0/6	3.64 ± 0.13	3.62 ± 0.12	67.1 ± 0.7

* The other treatment conditions were as follows: a 6% H_2O_2 solution, a temperature of 90 °C, a reaction time of 60 min, a 3% Na_2SiO_3 solution, a 0.5% $MgSO_4$ solution, and a 0.5% EDTA solution

Fiber Morphology

The fiber morphologies of the poplar chemi-mechanical pulp from different treatments are shown in Figs. 4 and 5. The different magnifications of the pulp fiber after a 6% NaOH treatment (A6) are clearly shown in Figs. 4a and 5a. During the A treatment, the pulp fibers were severely fibrillated, which may be due to the fact that the alkali treatment was highly effective in causing swelling of the fibers, thus reducing the damage to the fibers during the mechanical pulping process (Choi *et al*. 2016). During the refining process, the primary and secondary walls were deformed and stripped due to strong external forces, which resulted in the appearance of more fine fiber components (Banavath *et al.* 2011).

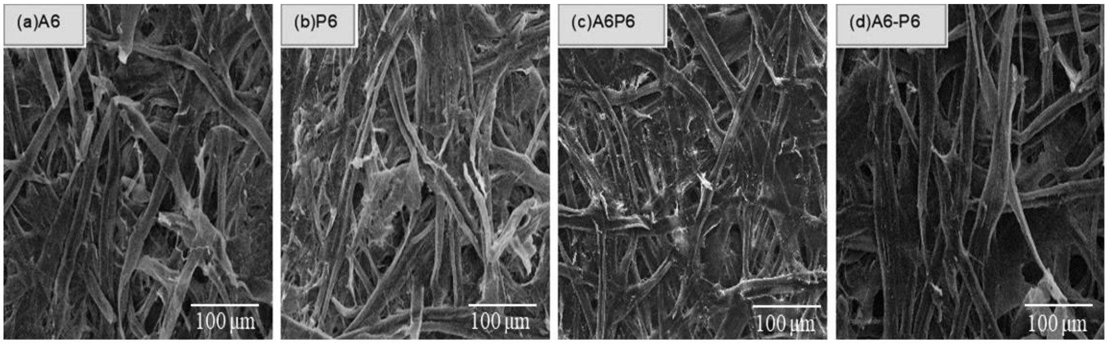


Fig. 4. Scanning electron micrographs of: (a) A6 (6% NaOH treatment); (b) P6 (6% H_2O_2 treatment); (c) A6P6 (6% NaOH and 6% H_2O_2 treatment); and (d) A6-P6 (first stage was 6% NaOH and second stage was 6% H_2O_2 treatment). All pictures were taken under 500 X magnification. The other treatment conditions were as follows: a 6% H_2O_2 solution, a temperature of 90 °C, a reaction time of 60 min, a 3% Na_2SiO_3 solution, a 0.5% $MgSO_4$ solution, and a 0.5% EDTA solution.

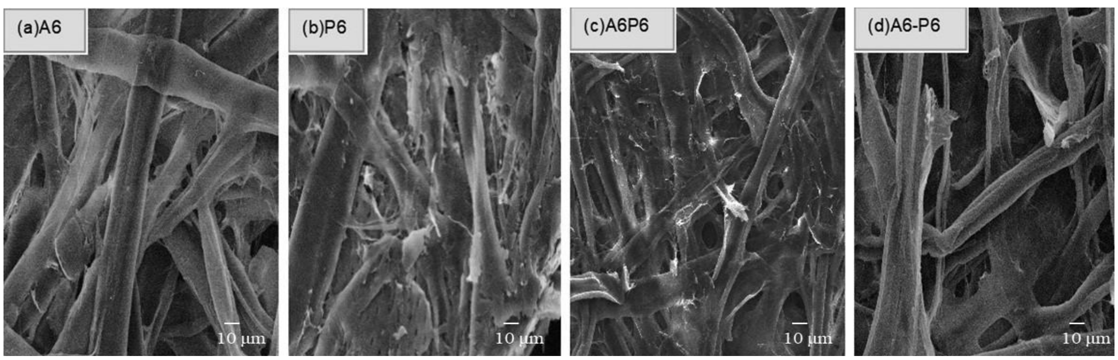


Fig. 5. Scanning electron micrographs of: (a) A6, (b) P6, (c) A6P6, and (d) A6-P6. All pictures were taken under 1000 X magnification.

Figures 4b and 5b show the fiber morphologies after a 6% H_2O_2 treatment (P6). Compared with the pulp fibers in Fig. 5a, the pulp fibers in Fig. 5b were distributed in a disorderly manner. There were obvious signs of fiber breakage due to an excessive extrusion and grinding intensity, and the fragments and faults of the fibers were visible. As shown in Fig. 4c and Fig. 5c, the fibers after a 6% NaOH and 6% H_2O_2 treatment (A6P6), the surface of the handsheets were smoother than the handsheets treated by the A and P methods; moreover, the fibers were evenly distributed and closely interwoven. Figure 4d and Fig. 5d displays the fiber morphologies of pulp treated with a first stage of 6% NaOH and a second stage of 6% H_2O_2 (A6-P6), which did not obviously display any fibrillation on the fiber surface. This may be due to the fact the fine fiber content increased after the alkali swelling, and the hydrogen peroxide bleaching reacted with the fine fibers, which resulted in the decrease of the composition of the fine fibers. Overall, the fiber fragments and fine fiber content were less, the interweaving distribution of the fibers was more uniform, and the distribution of the long fibers was obviously smooth in Fig. 5d.

CONCLUSIONS

1. A new staged alkali and hydrogen peroxide (A-P) treatment method was adopted to

improve the physical properties and brightness of poplar chemi-mechanical pulp. The results showed that the brightness of the A-P treated pulp was much higher than the traditional alkaline hydrogen peroxide (AP) method, not only with various alkali dosages but also with different hydrogen peroxide dosages.

2. The tear index and tensile strength of the A-P treated pulp was remarkably higher than the other treatment methods. The optimized conditions for the A-P treatment method were an alkali dosage of 6% in the first stage and a hydrogen peroxide dosage of 6% in second stage; which yielded a pulp with a tensile strength and tear index of 3.64 kN/m and 3.62 mN•m^2/g, respectively.

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