Fiber Properties of De-inked Old Newspaper Pulp after Bleaching with Hydrogen Peroxide

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Hydrogen peroxide was applied to bleach recycled de-inked pulp from old newspaper (ONP) in this study. Following single-stage bleaching, the fiber properties of the pulp (*viz*. brightness, yield, fiber length, fiber charge, and strength properties) were determined. Finally, the crystal structure of cellulose, fiber surface morphology, and functional groups of the control pulp and the bleached pulp using hydrogen peroxide were analyzed by XRD, SEM, and FT-IR, respectively. The single-stage peroxide bleaching applied to the de-inked ONP pulp could produce a high brightness pulp of 58% ISO at a yield of 92%. Fiber length decreased after bleaching treatment. The crystallinity index of cellulose of de-inked ONP pulp during bleaching or rinsing treatment increased due to the dissolution of cellulose in amorphous regions and/or the dissolution or loss of non-cellulosic constituents (hemicelluloses and lignin). Hydrogen peroxide bleaching resulted in fibrillation and longitudinal tearing of the fiber surface due to delignification, which led to an increase in the paper strength. FT-IR data showed that the content of carboxylic acid groups decreased during peroxide bleaching. The main chromophore (conjugated carbonyl groups) and the guaiacyl units of the pulp were damaged after bleaching resulting in delignification.

Keywords: Hydrogen peroxide bleaching; Brightness; De-inked ONP pulp; Fiber properties; Crystal structure

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INTRODUCTION

In recent years, as resource shortages and environmental problems have become more serious, the use of recycled plant fiber is being given attention globally. Old newspaper (ONP) is a primary material used in papermaking with recycled fibers. However, the improvement of recycled fiber quality (tensile strength, fiber length, and density, *etc.*) is still an issue that needs to be solved and remains the main goal of many research projects (Chen *et al*. 2010). To increase the use of recycled fibers in the papermaking industry, there is a higher requirement for cleaner and brighter de-inked pulps. Therefore, it is necessary to bleach de-inked pulps. In addition to brightening the fibers, the goal of bleaching de-inked pulp is to strip dyes and colors that are present in the wastepaper furnish (Bhardwaj and Nguyen 2005). Increasing concerns about the environmental and toxicity impacts of bleaching processes with chlorine-containing chemicals have provided motivation for the use of O-containing compounds, *e.g.*, O_2, O_3,

hydrogen peroxide, and formamidine sulfinic acid or dithionite (Bhardwaj and Nguyen 2005; Li *et al*. 2011).

Hydrogen peroxide bleaching has become the predominant bleaching method used in the manufacture of paper from recycled pulps in recent years (Pettit 1992; Matjacic and Moze 1998; Philippakopoulou and Economides 1999). For example, Matjacic and Moze (1998) studied one-stage hydrogen peroxide bleaching of old corrugated containers pulp. Philippakopoulou and Economides (1999) reported the effects of pH and H_2O_2 and NaS_2O_4 charges at the oxidative and reductive stages of two-stage bleaching of de-inked ONP/old magazine (OMG) mixtures. Pettit (1992) studied the post-bleaching of news/magazine mix de-inked pulp using H_2O_2, NaS_2O_4, calcium hypochlorite, or formanidine sulfinic acid for newsprint production. Wekesa and Ni (2003) studied the stabilization of peroxide systems by silicate and calcium carbonate and its application of bleaching of de-inked OMG pulp, and found that the residual peroxide and pulp brightness were improved. The hydrogen peroxide bleaching process results in not only increased brightness, but also in the improvement of paper physical properties (Pan 2011).

It is now well established that carboxylic acid groups are beneficial for the bonding of pulp fibers in paper and can increase the strength of paper (Zhang *et al*. 2007). These benefits have been attributed to the improved swelling and/or bonding ability of cellulosic fibers as the acid group content of cellulosic fibers is increased (Chen *et al*. 2010). The number of charged groups depends on the origin of the fiber and on the chemical treatment during pulping, bleaching, and refining, and other papermaking processes (Bhardwaj *et al*. 2004).

Some researchers have studied the effect of peroxide bleaching on the fiber charge of different pulps. For example, Gierer (1982) demonstrated that bleaching of pulp with alkaline hydrogen peroxide results in conversion of conjugated chromophoric groups associated with lignin structure to carboxylic acids and other degradation products. Sundberg *et al*. (2000) reported that the number of charged groups increased in peroxide bleaching and/or alkaline treatment of mechanical pulps due to alkaline hydrolysis of ester groups in the pulps. Holmbom and Pranovich (1998) found that more acidic groups were formed when spruce TMP fibers were bleached in the presence of peroxide. Toven (2003) showed that pressurized peroxide final bleaching of softwood kraft pulps yielded pulps of higher fiber charge than ClO_2 final bleaching. Bhardwaj and Nguyen (2005) studied the effect of hydrogen peroxide bleaching on de-inked 70% ONP/30% OMG on charge aspects, and found that the loss of the total fiber charge was apparently linearly related to the yield loss, but the change of the fiber surface charge was insignificant. However, the effect of bleaching on the pulp fiber charge is still rather ambiguous due to the different raw materials.

Different researchers have different views about the influences of bleaching on crystal structure of cellulose. Roncero *et al*. (2003) thinks that crystallinity values measured by means of the XRD technique showed that the crystallinity index (CrI) of the pulp increased after totally chlorine-free (TCF) sequences. Roncero *et al*. (2005) observed that CrI increased by almost 4% upon oxygen delignification, which indicated a variation of the ratio of crystalline to amorphous regions. However, Qin *et al*. (2008) state that the degree of crystallinity measured by means of the FT-IR technique was decreased after elemental chlorine free (ECF) and TCF bleaching stages.

The effect of bleaching agents, especially hydrogen peroxide bleaching, on fiber charge and crystal structure has not been satisfactorily explained until now. Therefore,

the objectives of this work were to determine the effect of hydrogen peroxide bleaching on fiber charge and crystal structure of a de-inked ONP pulp and to evaluate the effectiveness of peroxide bleaching in terms of brightness, pulp yield, and strength properties. In addition, changes in fiber surface morphology as measured by SEM and functional groups as evaluated by FT-IR were studied during hydrogen peroxide bleaching of de-inked ONP pulp.

EXPERIMENTAL

Materials

Old newspaper pulp (ONP) supplied by a paper mill in Dongguan city, China was employed in this study. Chemicals were obtained from Aladdin Industrial Corporation (Shanghai). The removal of the ONP ink was carried out in the laboratory. The ONP was firstly cured at 60 °C for 40 min (The other deinking conditions were as follows: NaOH 2%, H_2O_2 2%, Na_2SiO_3 3%, $MgSO_4$ 1.5%, sodium dodecyl benzene sulfonate 0.5%. All chemical charges were expressed as % on o.d. pulp.). Then the cured ONP with 1% consistency was floated at 40 °C for 15 min in a flotation cell. The α-cellulose content, hemicelluloses content, lignin content, and ash content of the de-inked ONP pulp were 60.6%, 18.3%, 20.5%, and 0.7%, respectively. The ISO brightness and water retention value (WRV) of the de-inked ONP pulp were 52.5% and 126.0 %, respectively.

H_2O_2 Bleaching Experiment of De-inked ONP Pulp

Hydrogen peroxide bleaching of de-inked ONP pulp with 10% consistency was conducted in polyethylene bags using various amounts of H_2O_2 and NaOH in a constant water bath at 70 °C for a reaction time of 60 min. Before adding bleaching chemicals, the pulp was pre-warmed. During the bleaching, the pulp was mixed every 20 min. The other conditions were 0.05% magnesium sulfate and 5% sodium silicate. After bleaching, the pulp was washed with de-ionized water to neutral pH. The order of addition of chemicals and pulp was standardized as follows: de-inked ONP pulp, water, magnesium sulfate, sodium silicate, sodium hydroxide, hydrogen peroxide. All chemical charges were expressed as % on o.d. pulp.

Brightness and pulp yield Determination

The brightness values were recorded in a Technibrite Micro TB-1C instrument (Technidyne Ltd., U.S.A.). The pulp yield was calculated as follows,

$$Pulp\ yield, \% = \frac{m_2}{m_1} \times 100 \quad (1)$$

where m_1 and m_2 are the total weight of dry pulp before and after bleaching, respectively.

Average Fiber Length and Coarseness Determination

Average fiber length, fines, and coarseness of ONP pulp were measured on a KAJAANI FS-300 fiber analyzer (Valmet Automation Kajaani Ltd., Finland) according to TAPPI Standard T-pm-91.

Carboxylic Acid Content Determination

The carboxylic acid content of each pulp was determined according to TAPPI Standard T237 (Tappi Test Methods, 2013). Pulp (1.5 g o.d.) was stirred in a solution of 0.1 M HCl (300.00 mL) for 1 h. The pulp was then filtered and rinsed in a Buchner funnel with deionized water. The sample was transferred into 250 mL of a 0.001 M NaCl solution that was acidified with 1.50 mL of 0.1 M HCl. This solution was titrated conductometrically with 0.05 M NaOH in 0.2-mL increments, recording the conductivity at each increment using a CyberScan CON 510 conductivity meter (Eutech Ltd., Singapore). The titration data was plotted as conductivity *versus* volume to determine the milli-equivalent of acid groups per kilogram of pulp. The reported results were the average of two measurements.

Scanning Electron Microscope (SEM) Analysis

SEM images of fiber surface morphology were obtained using an S3700 environmental scanning electron microscope (Hitachi Ltd., Japan), operated with a secondary electron detector at an accelerating voltage of 10 kV. The sample was coated with gold prior to analysis. Images of fibers were obtained in magnification of 500.

Determination of Crystallinity of Cellulose by X-ray Diffraction

The X-ray diffraction (XRD) scattering pattern of the pulp was analyzed on a Philips X'Pert MPD D/max-IIIA diffractometer (Rigaku Ltd., Japan) using a Cu-Kα source (λ=0.154 nm) in the 2θ of range 4 to 40° and a scanning step width of 0.02°/scan. Each analysis was repeated in triplicate. The phone scattering was subtracted from the pulp diffraction diagram. The crystalline reflections and amorphous halo were defined according to previously described recommendations (Chen *et al.* 2013). The crystallinity index (CrI) was calculated as follows,

$$CrI,\% = \frac{I_{cr}}{I_{cr} + I_{am}} \times 100 \quad (2)$$

where I_{cr} and I_{am} are the scattering intensities from the crystalline and amorphous regions of cellulose, respectively.

Fourier transform infrared (FT-IR) spectra

Milled dry pulp (3.5 to 4.0 mg) and spectroscopy grade KBr powder (500 mg) were placed in a mortar, mixed well, and pulverized. The mixture was used to press a KBr pellet which was analyzed with a Vetor 33 FT-IR spectrophotometer (Bruker Ltd., Germany) with a frequency range of 400 to 4000 cm^{-1}.

Paper Testing

Treated and control samples were disintegrated for 30,000 revolutions and then were refined for 10,000 in a PFI mill according to TAPPI Standard T248 (Tappi Test Methods 2013). Handsheets were formed according to TAPPI Standard T205 and conditioned for at least 24 h at 23 °C and 50% relative humidity before physical testing.

The tensile strength was measured with a L&W CE062 Tensile Tester using load cell 200 N and crosshead speed 100 mm/min (Lorentzen & Wettre Ltd., Sweden). Five handsheets were made from each pulp sample, and 12 test strips were used for tensile strength testing according to TAPPI Standard T494. Tear strength was measured according to TAPPI Standard T414.

RESULTS AND DISCUSSION

The Effect of Hydrogen Peroxide Bleaching on De-inked ONP Pulp Brightness and Yield

De-inked old newspaper pulp was preliminarily bleached with varying amounts of hydrogen peroxide, which were 1, 2, 3, and 4% relative to the dry pulp in the presence of 1.5% NaOH. The hydrogen peroxide dose response with respect to brightness and yield of pulp is shown in Fig. 1. The yield of de-inked ONP pulp decreased and the brightness increased when the amount of hydrogen peroxide increased. The increase in pulp brightness varied from 2.6% to 5.5% ISO above the control pulp brightness of 52.5% ISO depending on the hydrogen peroxide consumed when the applied hydrogen peroxide was varied from 1 to 4% relative to the dry pulp. The effect was tentatively attributed to delignification. The net brightness gain appeared to increase linearly initially with the increase in the dosage of hydrogen peroxide, but the gain became insignificant when the hydrogen peroxide dosage exceeded 3%. Chromophore elimination or formation reactions may be viewed as occurring co-currently with the dissolution of other components, such as hemicelluloses of the fibers, which determine the outcome of hydrogen peroxide bleaching (Bhardwaj and Nguyen 2005).

As shown in Fig. 1, the pulp yield continuously deceased as peroxide charge increased due to the loss of soluble fines, filler, alkali-soluble wood components, and contaminants during the hydrogen peroxide bleaching of the de-inked ONP pulp. Bhardwaj and Nguyen (2005) suggested that a possible reason is that the recycled de-inked pulp with low freeness has highly fibrillated fibers and fines that offer a large specific surface area for the dissolution materials including anionic substances under alkaline conditions prevalent during hydrogen peroxide bleaching.

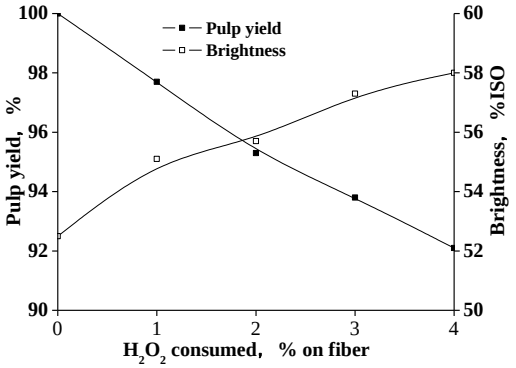


Fig. 1. Effect of hydrogen peroxide dosage (% o.d. pulp) on pulp brightness and yield

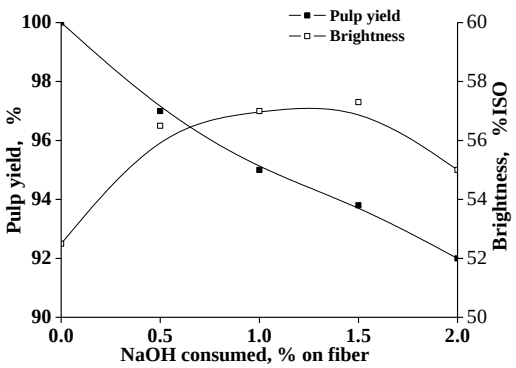


Fig. 2. Effect of sodium hydroxide dosage (% o.d. pulp) on pulp brightness and yield

Sodium peroxide dose is another important factor for the peroxide bleaching. In this study, different amounts of sodium peroxide (0.5 to 2%) in the presence of 2% H_2O_2 were examined to explore its significance to the peroxide bleaching. The brightness initially significantly increased with successively increased alkali charge until a certain critical point of alkali charge was attained. This may be due to the increased sodium hydroxide charge, which evidently began to decompose the hydrogen peroxide (Bhardwaj and Nguyen 2005). A brightness of 57.3% was obtained with a 2% H_2O_2 charge when a moderate alkali charge (1.5%) was present against 55% at higher alkalinity (2%). In addition, the pulp yield continuously decreased with the increase in

the dosage of sodium hydroxide.

Figure 3 shows that higher pulp brightness can be achieved along with increased ONP pulp yield losses. The result suggests that a single-stage peroxide bleaching applied to the de-inked ONP pulp can produce a high brightness pulp 58% ISO at pulp yield of 92%. It should be noted that alkaline peroxide bleaching results in chromophore removal through lignin modification and/or solubilization (Bhardwaj and Nguyen 2005). However, the removal of the chromophores, which resulted in the brightness gain, was not entirely responsible for the yield loss. Pan (2003) suggested that the dissolution of other pulp components, particularly hemicelluloses, contributed significantly to the yield loss due to the presence of alkali.

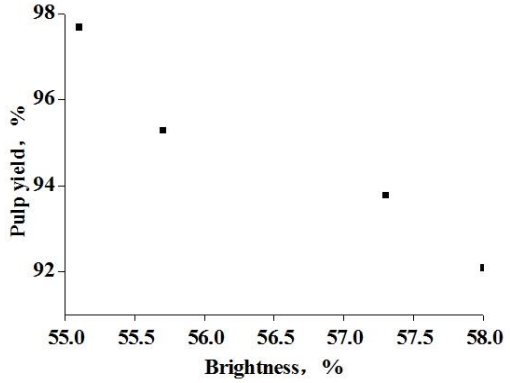


Fig. 3. Relationship between pulp yield and brightness

Fiber Length, Fines, coarseness of De-inked ONP Pulp

The fiber length, fines, and coarseness are important morphological factors because of their influence on the quality of paper produced from recycled pulp. The longest fibers contribute to better resistance to tearing, in comparison to the shortest ones. The percentage of fines is very important for bonding, as they increase the possibility of bonds among the fibers (Requejo *et al*. 2012). Compared with the control pulp, the average fiber length, fines, and coarseness of the bleached pulp became slightly smaller (Table 1) because bleaching treatment made the fibers more flexible which enabled easier passage through screens.

Table 1. Effect of Hydrogen Peroxide Bleaching on Fiber Average Length, Fines, and Coarseness

Sample	L_w (mm)	Fines (*n*) (%)	Coarseness (mg/m)
Control pulp	1.99	30.4	0.155
H_2O_2 (%)	L_w (mm)	Fines (*n*) (%)	Coarseness (mg/m)
1	1.95	29.6	0.144
2	1.97	29.7	0.143
3	1.89	28.4	0.146
4	1.96	30.3	0.148
NaOH (%)	L_w (mm)	Fines (*n*) (%)	Coarseness (mg/m)
0.5	1.93	29.5	0.151
1	1.9	30	0.149
1.5	1.89	28.4	0.146
2	1.97	30.1	0.145

Note: L_W was the fiber weight length; Fines (*n*) was the fines number-average content.

The Effect of Hydrogen Peroxide Bleaching on Pulp Fiber Charge

Due to the loss or dissolution of alkali-soluble wood components (carboxylated hemicelluloses species) during the bleaching and/or rinsing procedure, the fiber charge decreased with increasing doses of hydrogen peroxide (Fig. 4). This result is in accordance with previous work (Bhardwaj and Nguyen 2005). Bhardwaj and Nguyen (2005) considered that the relationship of hydrogen peroxide consumption with fiber charge indicated that the evidence of gain perhaps due to the formation of carboxyl from carbonyl groups after the dissolution reached a maximum. Demethylation of pectin substances frees methyl-esterified carboxylic groups (Sundberg *et al*. 2000), although the oxidized lignin and pectic acids from the fibers and fines may be dissolved into the water phase as anionic trash (Thornton *et al*. 1993; Brauer *et al*. 2001). When the consumption amount of hydrogen peroxide increased from 1% to 3%, compared with 91.6 mmol/kg of the control sample, the fiber charge of bleached pulp decreased by 5% and 43.9%, respectively.

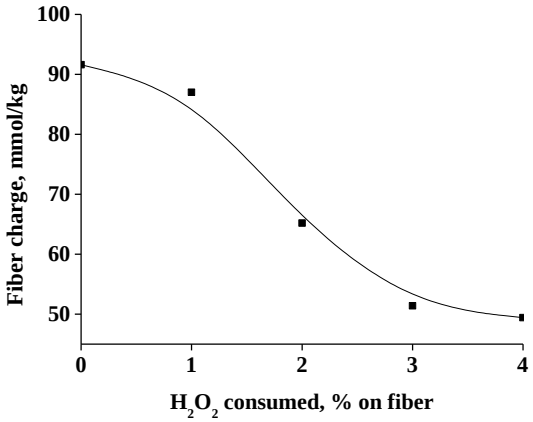


Fig. 4. Relationship between hydrogen peroxide dosage and fiber charge

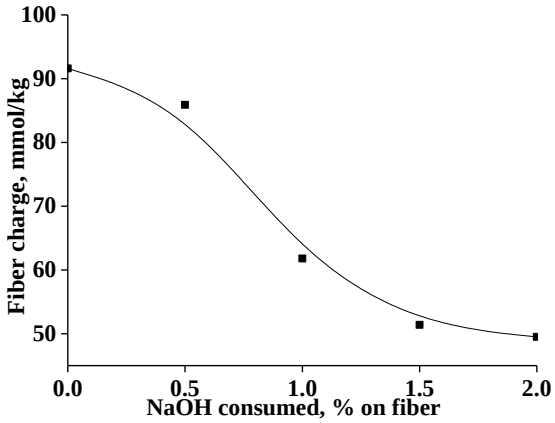


Fig. 5. Relationship between sodium hydroxide dosage and fiber charge

Figure 5 shows that fiber charge had a decreasing trend with an increase in the dose of sodium hydroxide consumption. This suggested that the charged material (carboxylated hemicelluloses species) was substantially released from the fibers and rinsed away during the rinsing procedure, leaving the fibers themselves with a lower cationic demand. When the consumption dose increased from 0.5% to 1.5%, compared with the control sample, the fiber charge of bleached pulp decreased by 6.3% and 43.9%, respectively.

Strength Properties of Peroxide-Bleached De-inked ONP Paper

Table 2 shows the changes of strength properties of paper made from bleached pulp. Tear index continuously decreased and tensile index increased with an increase in the amount of hydrogen peroxide and sodium hydroxide. Two reasons are considered to be responsible for the result that strength properties of ONP pulp had an evident improvement after peroxide bleaching. One of them is that the collapse of fiber lumens caused by delignification and dissolving of other wood components increases the area of contact between fibers. Therefore, the fiber-to-fiber bonding was strengthened, which facilitated the development of mechanical properties of paper (Pan 2004; Li *et al*. 2011). Besides, the alkaline environment of bleaching increases the swelling of the cellulose

fibers and thereby increases the surface area. That is believed to result in improved paper strength (Li *et al*. 2011).

Table 2. Strength Properties of Paper Made from Bleached Pulp with Different Hydrogen Peroxide Dosage and Sodium Hydroxide Dosage

H_2O_2 dosage (%)	Control	1	2	3	4
Tensile strength (Nm/g)	29	34.8	35.2	35.7	36.9
Tear strength (mN·m²/g)	15	14.5	13.8	13.7	13.2
NaOH dosage (%)	Control	0.5	1	1.5	2
Tensile strength (Nm/g)	29	34.3	34.9	35.7	35.7
Tear strength (mN·m²/g)	15	14	13.8	13.7	12.7

Cellulose Crystallinity of Peroxide-Bleached De-inked ONP Pulp

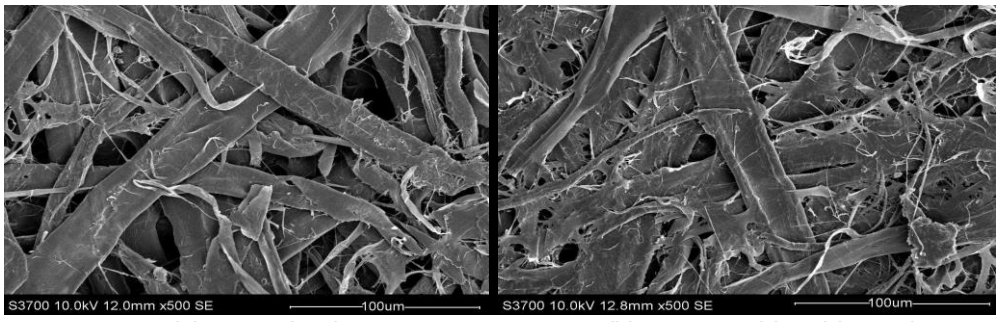
Crystal structure of peroxide bleaching de-inked ONP was investigated by XRD in this study. Crystallinity of cellulose of de-inked ONP pulp increased with an increase in hydrogen peroxide dosage or sodium hydroxide dosage in varying degrees (Table 3), which is perhaps due to the dissolution of cellulose of the amorphous region and/or the dissolution and loss of noncellulosic constituents (such as hemicelluloses and lignin) during peroxide bleaching or rinsing procedure, resulting in the remaining cellulosic material having a higher crystalline content (Roncero *et al*. 2003). When the amounts of hydrogen peroxide and sodium hydroxide were 4% and 2%, crystallinity of cellulose increased by 3.4% and 3.8%, respectively.

Table 3. Effects of Hydrogen Peroxide Dosage and Sodium Hydroxide Dosage on Crystallinity of Cellulose

H_2O_2 dosage (%)	Control	1	2	3	4
CrI (%)	79.5	80.1	80.9	81.7	82.2
NaOH dosage (%)	Control	0.5	1.5	2	-
CrI (%)	79.5	81.4	81.7	82.5	-

SEM Analysis

Fiber surface morphologies of control pulp and peroxide bleaching pulp were observed by SEM (Fig. 6). Figure 6a shows that the control pulp fibers were mostly intact, there were more long fibers, and the fiber surface of control pulp was smoother.



(a) Control pulp (b) 1% H_2O_2 bleaching pulp

Fig. 6. SEM images of fiber surface morphology of control pulp and peroxide bleaching pulp

Compared with the control pulp, the peroxide bleaching pulp surface became rougher and no more intact, and more fibrils and longitudinal tearing could be observed on the fiber surface, which demonstrated that the delignification of the peroxide bleaching pulp occurred on the fiber surface, releasing fibrils (Fig. 6b). Therefore, these changes of fiber surface morphology led to better bonding between fibers in handsheets, resulting in an increase of paper strength of hydrogen peroxide bleaching ONP pulp.

FT-IR Analysis

The FT-IR spectra of the control pulp and the hydrogen peroxide bleaching pulp are shown in Fig. 7, and the assignments of the observed bands and their relative intensities are listed in Table 4. The absorption bands and the corresponding structure assignments from the infrared spectra are based on literature values (Chen *et al*. 2013). The normalized FT-IR absorption spectra at a band position of 1370 cm^{-1} was primary due to aliphatic C-H stretch in CH_3 in pulp.

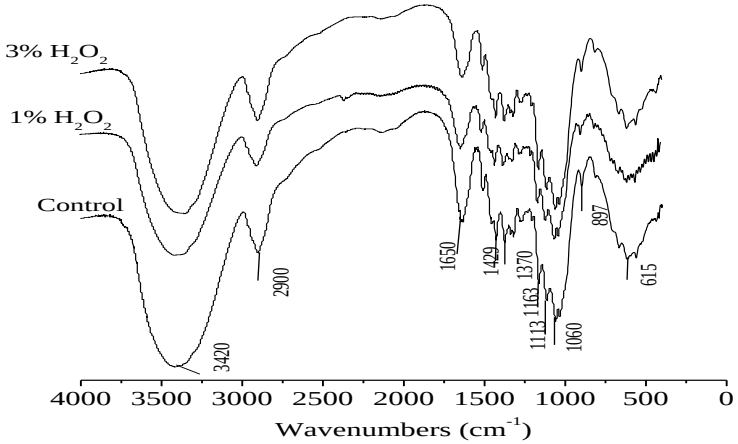


Fig. 7. FT-IR spectra of control pulp and hydrogen peroxide bleaching pulp

Table 4. Band Assignments and their Relative Intensities of FT-IR from Control Pulp, 1% H_2O_2 Bleaching Pulp, and 3% H_2O_2 Bleaching Pulp

Band position	Assignment	Samples		
		Control pulp	1% H_2O_2 pulp	3% H_2O_2 pulp
3390	O-H stretching, related to rupture of cellulose hydrogen bonds	1.82	1.72	1.69
2900	C-H stretching, related to rupture of cellulose methyl/methylene group	1.14	1.01	0.92
1633	Carboxylic acids/ester groups	0.87	0.84	0.79
1424	Aromatic skeletal vibrations, combined with -OCH_3 in-plane deformations	1.05	0.99	0.95
1370	Aliphatic C-H stretch in CH_3	1.00	1.00	1.00
1163	C-O in lignin and xylan	1.40	1.36	1.35
1059	C-O stretch in secondary alcohol	1.81	1.83	1.78
1030	C-O stretch in primary alcohol	1.76	1.73	1.78
1100 /900	Crystalline to amorphous cellulose ration	1.87	1.89	2.11
900	Amorphous cellulose	0.83	0.79	0.75

The band at 3390 cm^{-1} and 2900 cm^{-1} were assigned to O-H stretching related to rupture of cellulose hydrogen bonds and C-H stretching related to rupture of cellulose methyl/methylene group, respectively, and their relative intensities decreased after hydrogen peroxide bleaching. The decrease of relative intensity of the band at 1633 cm^{-1} showed that the main chromophore (conjugated carbonyl groups) of the pulp was eliminated and the content of carboxylic acid groups decreased during hydrogen peroxide bleaching. The results are in accordance with the brightness and carboxyl group content measurement data. The decrease of relative intensity of the band at 1424 cm^{-1} assigned to aromatic skeletal vibrations combined with -OCH_3 in-plane deformations demonstrated that the guaiacyl unit was damaged during bleaching resulting in delignification. The decrease of relative intensity of the band at 1163 cm^{-1} assigned to C-O in lignin and xylan demonstrated that the lignin or hemicelluloses was reduced during the bleaching or rinsing procedure. In addition, the ratio of the relative intensity of the band at 1100 cm^{-1} and 900 cm^{-1} assigned to crystalline to amorphous cellulose ratio significantly increased after hydrogen peroxide bleaching, which is in accordance with XRD measurement data.

CONCLUSIONS

1. The yield of de-inked ONP pulp decreased and the brightness increased when the amount of hydrogen peroxide increased. Because of the loss or dissolution of alkali-soluble components (carboxylated hemicelluloses species) during the bleaching and/or rinsing procedure, the fiber charge decreased with increasing doses of hydrogen peroxide. The strength properties of ONP pulp had an evident improvement after peroxide bleaching.
2. The crystallinity index of the pulp increased during peroxide bleaching and/or rinsing procedure due to the dissolution of cellulose of amorphous region and/or the dissolution and loss of noncellulosic constituents (such as hemicelluloses and lignin).
3. FT-IR results demonstrated that the lignin was reduced during the bleaching resulting in delignification.

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