HYDROGEN PEROXIDE BLEACHING OF HARDWOOD KRAFT PULP WITH ADSORBED BIRCH XYLAN AND ITS EFFECT ON PAPER PROPERTIES

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The adsorption of xylan on pulp fibers improves the strength properties of paper. However, the optical properties are decreased significantly. The objective of our research was to bleach hardwood kraft pulp with adsorbed birch xylan by hydrogen peroxide and study the effect of bleaching parameters on paper properties. The bleaching parameters studied included bleaching temperature, time, initial pH as well as $MgSO_4$ dosage. The optical properties (whiteness, brightness, opacity) and physical properties (tensile index, tearing index, bulk) of handsheets made from the pulp bleached with different process variables were measured. The results showed that better optical properties were obtained with higher bleaching temperature, longer bleaching time, and more $MgSO_4$ dosage. Bleaching from an initial pH of 11 provided the highest brightness value. On the other hand, strength properties were improved with decreasing of the bleaching temperature, and increasing the initial pH and $MgSO_4$ dosage. The relationship between strength properties and bleaching time varied depending on bleaching temperature. According to the results, both good mechanical properties and optical properties could be achieved when the operating parameters were controlled properly. Therefore hydrogen peroxide bleaching was proved to be a suitable method for bleaching hardwood kraft pulp with adsorption of birch xylan.

Keywords: Hydrogen peroxide; Bleaching; Hardwood; Kraft pulp; Birch xylan

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INTRODUCTION

Xylans are the most abundant hemicelluloses found in the cell walls of land plants, of which they can constitute more than 30% of the dry weight (Joseleau et al. 1992). Softwoods contain arabino-4-O-methylglucuronoxylan, which is substituted by one 4-O-methyl-D-glucuronic acid group per 5 or 6 D-xylose units on average. Softwood xylan also contains one α-L-arabinose unit per 5 to 12 xylose units. In hardwoods the xylan is O-acetyl-4-O-methylglucuronoxylan. On average, every tenth xylose unit is substituted by a 4-O-methylglucuronic acid residue, and the amount of acetyl groups is 3.5–7 per ten xylose units (Schimizu 1991).

In the current trend for a more effective utilization of biomass, more and more attention has been paid to the exploitation of xylans as strength-enhancing additives for paper (Schönberg et al. 2001; Danielsson and Lindström 2005; Lindström et al. 2005;

Köhnke et al. 2009). Adsorption of pre-isolated xylan modifies the surface morphology and surface chemistry of cellulose fibers (Westbye et al. 2006), which has a positive effect on improving strength properties of paper (Schönberg et al. 2001). Besides, adsorbed xylan is spontaneously beneficial to other paper/pulp properties, such as beatability, wettability, and resistance to hornification (Köhnke and Gatenholm 2007). Efforts have been made for the exploitation of birch, barley husk, and other agricultural waste materials as xylan extraction resources, which facilitates using pre-isolated xylan commercially at a large scale for papermaking industry applications (Westbye et al. 2008; Köhnke et al. 2009). On the other hand, the increasing amount of xylan adsorbed on fiber surface reduces the brightness of paper remarkably due to the lignin residues in extracted xylan (Lee et al. 2010). Generally, there are strong chemical and physical interactions between hemicelluloses and lignin in wood, and the so-called lignin carbohydrate complexes (LCC) are well known (Björkman 1957; Lundquist et al. 1979; Eriksson et al. 1980; Meshitsuka et al. 1982; Lawoko et al. 2005). The remaining lignin in the extracted xylan fractions has a much closer linkage with xylan (Westbye et al. 2008). Consequently, an applicable bleaching process is needed to achieve both enhanced strength and high brightness after the adsorption of xylan on pulp fibers.

Increasing concern about the environmental impact of bleaching processes with chlorine and chlorine-based compounds have provided motivation for use of elemental chlorine free (ECF) and totally chlorine free (TCF) bleaching sequences (Shatalov and Pereira 2005; Abrantes et al. 2007). These bleaching processes are based on oxygen-derived compounds, among which, hydrogen peroxide has proved to be a highly efficient and competitive bleaching chemical in terms of delignification efficiency, low costs, and reducing ecological impact (Walsh 1991; Abrantes et al. 2007). As a result, it has gained increasing importance in industrial applications (Zeronian and Inglesby 1995). Peroxide primarily works under alkaline conditions when the perhydroxyl ion, OOH^-, is formed as in the following reactions (Zeronian and Inglesby 1995; Johnson et al. 2002).

$$H_2O_2 + OH^- \longleftrightarrow OOH^- + H_2O \quad (1)$$

$$OOH^- + \text{chromophore} \longrightarrow \text{bleaching (chromophore destroyed)} \quad (2)$$

Some transition metal ions such as iron, manganese, and copper accelerate the catalytic decomposition of the active perhydroxyl ion. On the other hand, other ions (e.g. Mg^{2+}, Ca^{2+} and SiO_3^{2-}) inhibit this acceleration. Therefore, a chelation step influencing the pulp metal profile prior to bleaching is necessary. This step reduces the concentration of the transition metal within the pulp, without removing the alkaline earth metals that stabilize peroxide. Of the hundred chelating agents known, DTPA (diethylenetriamine pentaacetic acid), with eight bonding sites, is a common chelant used in the pulp and paper industry due to its ability to chelate most metal ions in a 1 to 1 ratio. (Lapierre et al. 1995; Potůček and Milichovský 2000).

Alkaline hydrogen peroxide bleaching can change the chemical, physical, and optical properties of pulp significantly; therefore adjusting the conditions of bleaching properly appears fairly essential to meet specific requirements of different paper grades (Johnson et al. 2002; Ni et al. 2007). The primary objective of the study presented here is to bleach hardwood kraft pulp adsorbed with birch xylan by hydrogen peroxide and study the influence of bleaching operating variables (bleaching temperature, time, initial pH, and $MgSO_4$ dosage) upon paper properties. The whiteness, brightness, opacity, bulk, tensile strength and tear strength were examined to characterize the optical properties and physical properties of handsheets.

EXPERIMENTAL

Materials

A thoroughly washed (400-mesh), never-dried, non-beaten, industrially produced, bleached eucalyptus kraft pulp was used in our research. The bleaching sequence of D-EOP-D-PO-D was used to produce this bleached pulp. Poly-DADMAC (Poly diallyl dimethyl ammonium chloride), with the molecular weight of 400,000 to 500,000, was purchased from Sigma-Aldrich Company. Commercial birchwood xylan which was also produced by Sigma-Aldrich Company was used. All reagents for the bleaching process were obtained from Samchun Pure Chemical Company.

Adsorption of Xylan on Pulp Fibers

To promote the interaction between xylan and fibers, 0.30% Poly-DADMAC based on oven dried pulp was added initially into the pulp slurry, and this treatment reversed the zeta potential of cellulosic fibers to positive. The stock was then stirred for 20 min and washed twice with 400-mesh to remove the unabsorbed Poly-DADMAC. Xylan was dissolved at 90 ^{o}C for 15 min. After cooling down the solution to room temperature, 6.0% xylan based on oven dried pulp was added into the pulp and mixed for 20 min.

Chelation Pretreatment (Q Stage)

In order to remove transition metal ions, the chelation pretreatment (Q stage) was performed at 70 ^{o}C, 5% pulp consistency for 30 min, followed by washing. For this stage, 0.30% DTPA based on oven dried pulp was added and mixed well with pre-warmed pulp. The pulp was placed in a heat-proof polystyrene bag, sealed, and fully immersed in a pre-heated constant temperature water bath. For the duration of the experiment, the pulp was mixed every 15 min.

Hydrogen Peroxide Bleaching Process (P Stage)

The hydrogen peroxide bleaching process (P stage) was conducted at 10% pulp consistency with 2.0% H_2O_2 based on oven dried pulp in plastic bags placed in a water bath. 3.0% Na_2SiO_3 based on oven dried pulp was used as hydrogen peroxide stabilizer. Before adding bleaching chemicals, the pulp was pre-warmed. During the bleaching experiment, the pulp was mixed every 15 min. The bleaching time, temperature, initial pH, and $MgSO_4$ dosage varied in the ranges 30 min to 60 min, 40 to 80 ^{o}C, 10 to 12, and 0 to 0.30% according to different experiment designs. The required dosage of sodium hydroxide for pH of 10, 11, and 12 were 0.10%, 0.75% and 2.15% respectively. After bleaching, the pulp was washed to neutral pH.

Handsheets Forming and Measurement of Properties

Handsheets with basis weight of 80 g/m^2 were made from the bleached pulp and then were conditioned. The optical properties including brightness, whiteness, opacity, and physical properties including bulk, tensile strength, and tear strength were measured according to TAPPI standard methods.

RESULTS AND DISCUSSION

Effect of Adsorption of Birch Xylan

Table 1 compares the characteristics of hardwood kraft pulp before and after the adsorption of birch xylan. The results demonstrate that the adsorption of xylan improved the strength properties of pulp by 29.5% for tensile index and 22.7% for tear index, but the whiteness and brightness were decreased by 24.0% and 10.0% respectively. Besides, both the opacity and bulk were enhanced. It is commonly accepted that xylan attached on the fiber surface adds flexibility to the cell wall/fiber surface, resulting in stronger fiber-to-fiber joints or greater contact area between the fibers (Bhaduri et al. 1995; Danielsson and Lindstrom 2005). Accordingly, the strength properties of pulp were promoted. Nevertheless, the residual lignin components in xylan can lead to a distinct reduction in the brightness of pulp, which affects the quality of paper to a great extent (Lee et al. 2010).

Table 1. Properties of Original Pulp and Xylan Treated Pulp

Pulp	Whiteness (%)	Brightness (%)	Opacity (%)	Bulk (cm^3/g)	Tensile index (Nm/g)	Tear index (mN·m^2/g)
Original	77.8	87.0	80.9	2.87	4.75	1.32
Xylan treated	59.1	78.2	84.7	2.94	6.15	1.62

Effect of Bleaching Time and Temperature

The hydrogen peroxide bleaching process for researching the effect of bleaching time and temperature was conducted with 0.05% $MgSO_4$ dosage and from an initial pH of 11.

Figure 1 indicates that brightness was enhanced with increasing of bleaching time and temperature, as the effect of delignification was enforced. Besides, in the first 30 min, the tendency was much more evident than later.

Figure 2 shows that when the bleaching temperature was 40 ^oC, the opacity kept decreasing with continued bleaching. On the other hand, when the bleaching temperature was 60 ^oC or 80 ^oC, the opacity decreased first and then increased again. It is supposed that some wood components including lignin were dissolved out during the bleaching and resulted in the collapse of fiber lumens. At the same time, alkaline swelling made the fibers more flexible and softer. As a result, the contact area between fibers was increased and the light scattering coefficient of paper sheet was reduced, which caused the decrease of opacity. Besides, bleaching of pulp diminished the light adsorption coefficient by removing light absorbing colored groups, which also reduced the opacity of paper, according to the Kubelka-Munk theory (Levlin and Söderhjelm 1999). When the bleaching temperature was high, it is assumed that xylan, which had been dissolved out and subjected to loss of acetyl groups, began to redeposit on fiber surface during the process of bleaching. In support of this mechanism, earlier research proved that the removal of acetyl groups happened in the earlier stage of bleaching could facilitate the re-adsorption of xylan and other hemicelluloses on fibers, as the solubility of the hemi-celluloses without acetyl groups was much lower than the ones with acetyl groups (Reis et al. 1994; Gröndahl and Gatenholm 2005; Ni et al. 2007).

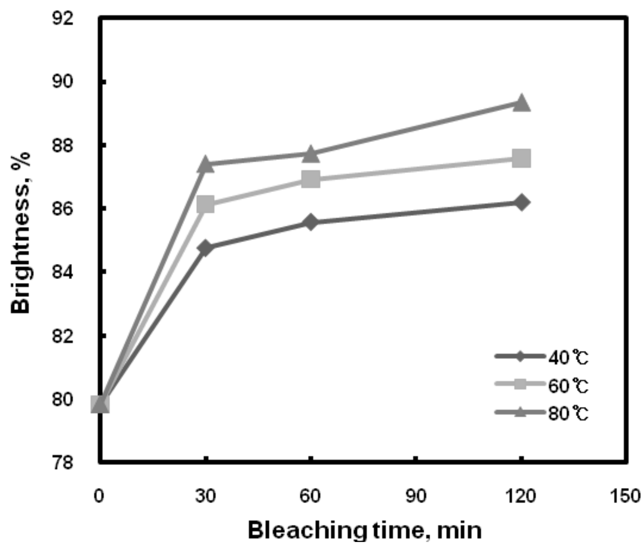

Fig. 1. Effect of bleaching time and temperature on brightness

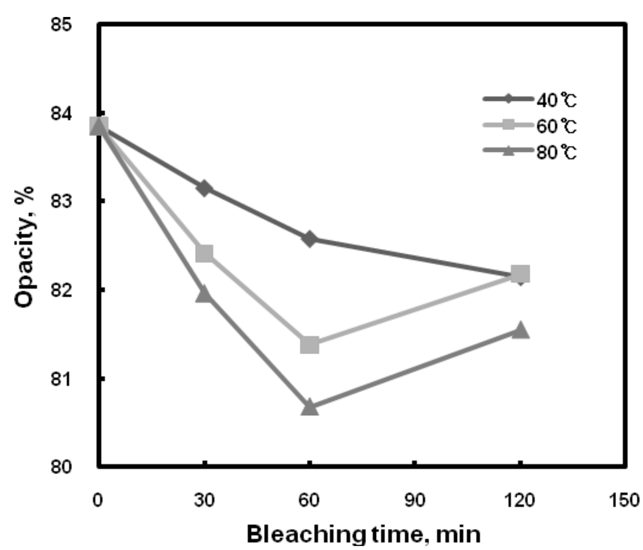

Fig. 2. Effect of bleaching time and temperature on opacity

In Fig. 3, it can be observed that higher bleaching temperature resulted in poorer strength properties (i.e. tensile index and tear index). Besides, the tensile index and tear index were promoted steadily when the bleaching temperature was 40 °C. However, in case of 60 °C and 80 °C, the tensile index and tearing index dropped first, then went up again after bleaching for 60 min. To understand this phenomenon, it is necessary to analyze the chemical and physical changes of pulp in the bleaching process.

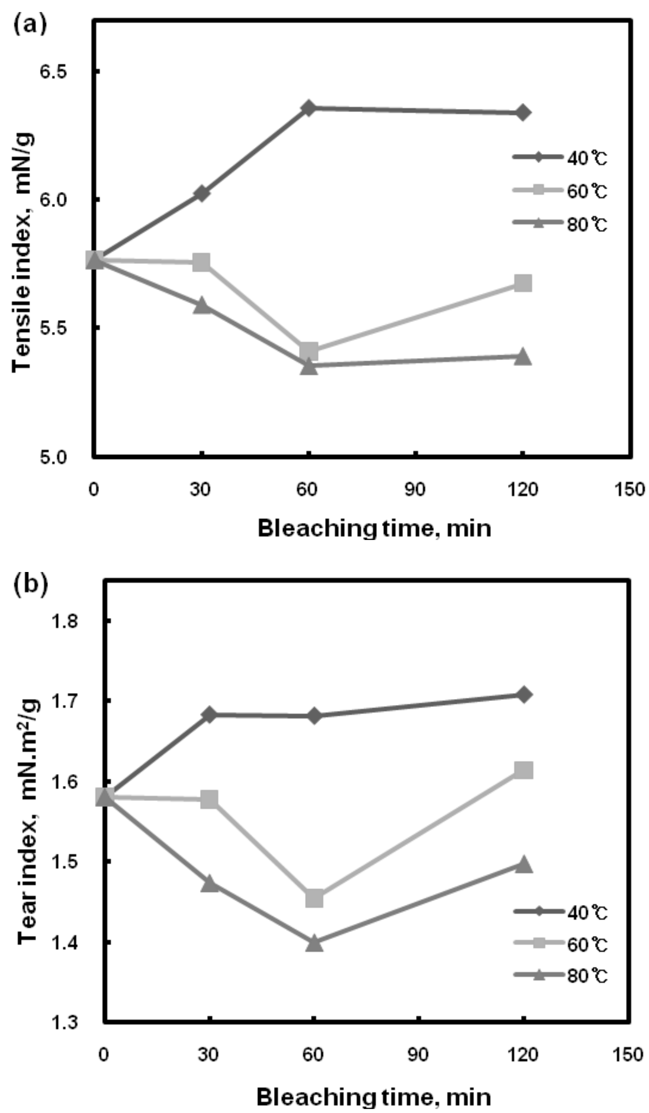

Fig. 3. Effect of bleaching time and temperature on tensile index (a) and tear index (b)

In general, there are five main factors affecting the properties of pulp. First of all, the dissolving of both adsorbed xylan from fiber surface and the original xylan located in the cell wall of fiber has negative effect on the physical properties of pulp (Simonson 1963; Fiserova et al. 1987). Besides, the bleached reaction species of alkaline hydrogen peroxide are hydroperoxy anions HOO^-. The decomposition intermediates, such as hydroxyl HO• and superoxide anion O_2^{-}• radicals, have generally undesirable influence in bleaching processes and also attack the carbohydrates, resulting in strength loss of the fibers (Zeronian and Inglesby 1995; Potůček and Milichovský 2000). Third, the collapse of fiber lumens caused by delignification and dissolving of other wood components increases the contacting area between fibers. Therefore, the fiber-to-fiber bonding is strengthened, which facilitates the development of mechanical properties of paper sheet (Pan et al. 2004). Fourth, 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. Lastly, the removal of acetyl groups from hemicelluloses species promotes the re-disposition of xylan and other hemicelluloses, which also can be expected to affect the strength properties of sheet (Thornton et al. 1994; Dahlman et al. 2003).

When the bleaching temperature was 40 °C, the effect of collapse of fiber lumens and swelling of cellulose fibers played a main role, while the effect of dissolving of xylan, deterioration of carbohydrates caused by hydrogen peroxide free radicals as well as re-disposition of xylan were weak due to the mild reaction condition, which led to the steady increasing of paper strength. On the other hand, when the bleaching temperature was raised to 60 °C or 80 °C, the influence of dissolving of xylan and degradation of carbohydrates caused by hydrogen peroxide free radicals played a primary role in the earlier stage, while the impact of collapse of fiber lumens, swelling of cellulose fibers and re-disposition of xylan overcame the effect of former ones in the later stage.

Effect of Initial pH

The hydrogen peroxide bleaching process for researching the effect of initial pH was performed at 80 °C, with 0.05% $MgSO_4$ dosage for 120 min.

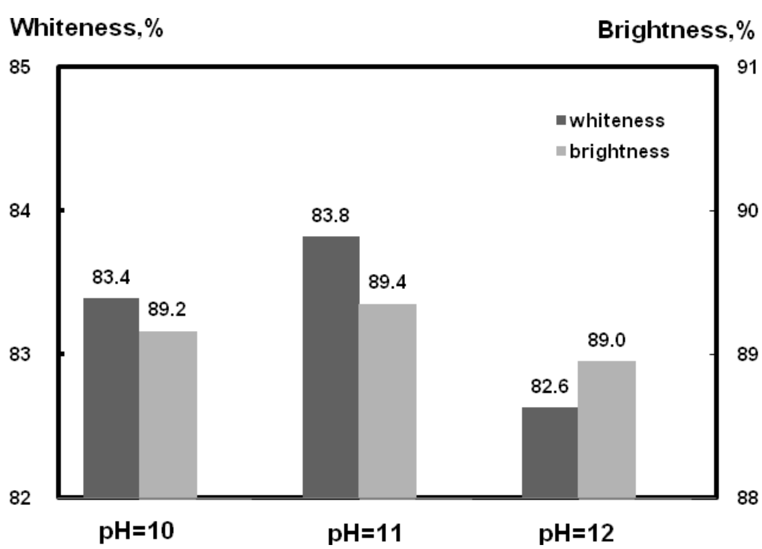

Fig. 4. Effect of initial pH on whiteness and brightness

The data in Fig. 4 demonstrates that the hydrogen peroxide bleaching from initial pH 11 provided both the highest whiteness and the highest brightness value. The explanation is that lower initial pHs represented weak alkaline conditions that could not supply enough OOH^- for the bleaching system. As a result, insufficient bleaching caused lower whiteness and brightness. However, when the initial pH was too high, excess alkali increased hydroxyl ions, which in turn produced perhydroxyl ion too rapidly to be used. That could lead to the following reaction (Johnson et al. 2002).

$$H_2O_2 + OOH^- \longleftrightarrow OH^- + O_2\,(g) + H_2O \quad (3)$$

Consequently, hydrogen peroxide would be decomposed before it was used for bleaching. At the same time, pulp could also become darkened due to alkaline darkening reactions, which can be simply described as (Johnson et al. 2002),

$$\text{lignin} + OH^- \longrightarrow \text{chromophoric groups} \quad (4)$$

where phenolic lignin units could be oxidized by alkali to light absorbing structures, resulting in lower optical properties.

The experimental results for the effect of initial pH on bulk, opacity, tensile index and tear index are shown in Figs. 5 and 6, respectively. It is obvious that both bulk and opacity declined with the increasing of initial pH, whereas tensile index and tear index increased greatly. The reason is that higher initial pH created a stronger alkaline environment, which facilitated the collapse of fiber lumens caused by delignification and dissolving of other wood components. As a result, bulk was decreased. But the collapse of fiber lumens increased the contacting area between fibers. In addition to the better swelling of fibers, the strength properties of paper were promoted evidently. At the same time, owing to the reduction of non-contacting areas between fibers, the light scattering coefficient went down and thereby the opacity declined.

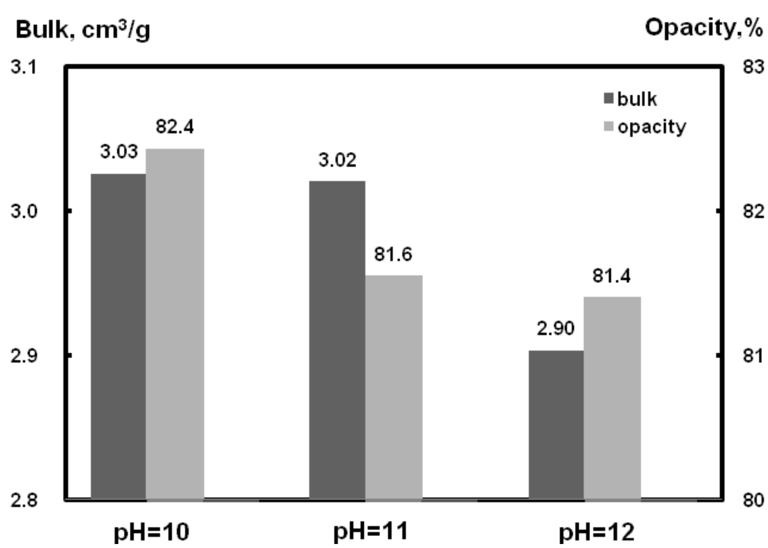

Fig. 5. Effect of initial pH on bulk and opacity

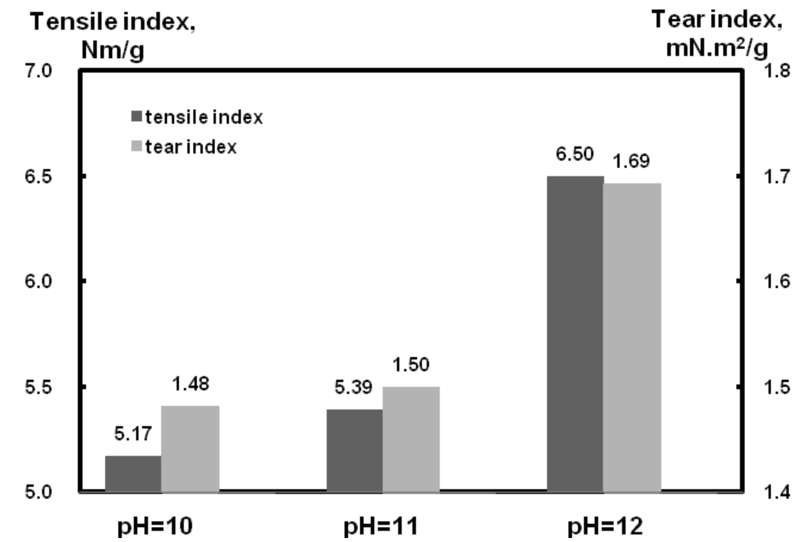

Fig. 6. Effect of initial pH on tensile index and tear index

Effect of $MgSO_4$ Dosage

The hydrogen peroxide bleaching process for researching the effect of $MgSO_4$ dosage was conducted at 80 ^{o}C, from an initial pH of 11 and for 120 min.

According to Figs. 7 through 9, it appears that both optical properties and mechanical properties were improved with the increasing addition of $MgSO_4$.

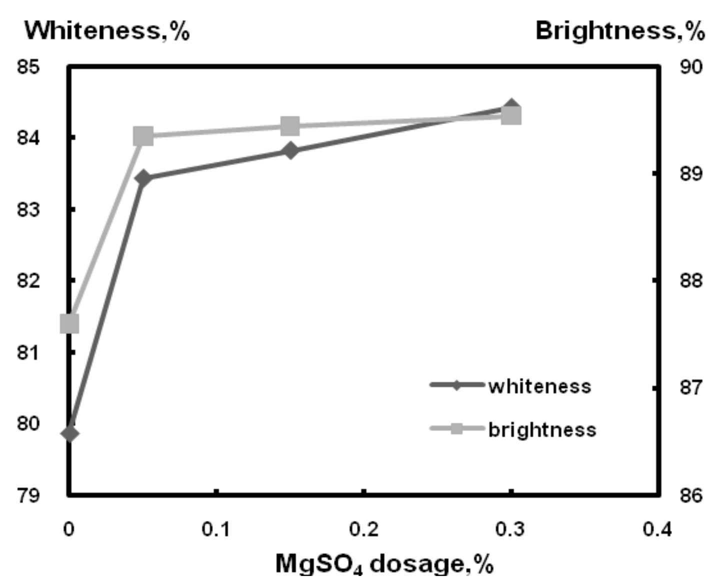

Fig. 7. Effect of $MgSO_4$ dosage on whiteness and brightness

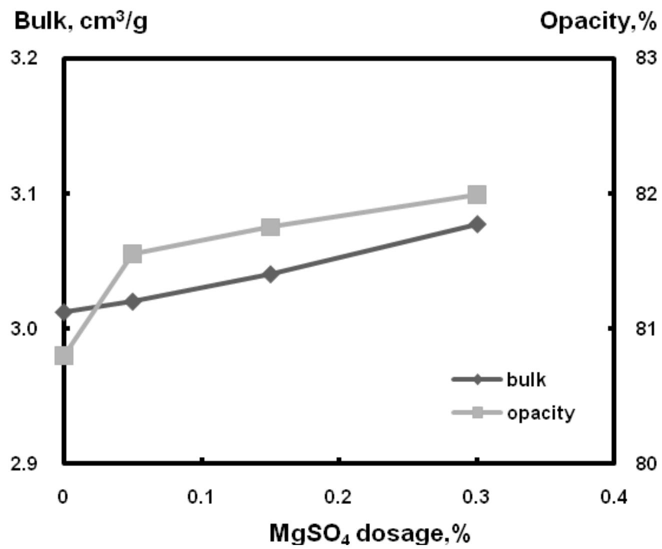

Fig. 8. Effect of $MgSO_4$ dosage on bulk and opacity

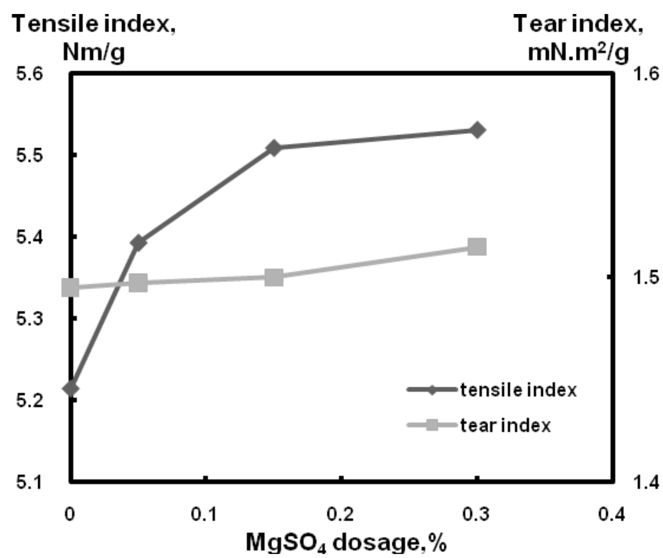

Fig. 9. Effect of $MgSO_4$ dosage on tensile index and tear index

Previous studies proved that peroxide can be catalytically decomposed by some transition metal ions such as iron, manganese, and copper as follows (Persley and Hill 1996):

$$H_2O_2 + M^{2+} \longleftrightarrow M^{3+} + OH^- + OH\bullet \quad (5)$$

$$H_2O_2 + M^{3+} \longleftrightarrow M^{2+} + H\bullet + OOH\bullet \quad (6)$$

$$OOH\bullet \longleftrightarrow O_2\,(g) + H\bullet \quad (7)$$

$$H\bullet + OH\bullet \longleftrightarrow H_2O \quad (8)$$

Therefore, a part of hydrogen peroxide was consumed without taking part in the bleaching reaction. In addition, the newly created free radicals attacked the carbohydrates, leading to a loss of fiber strength. However, magnesium sulfate, as an alkaline earth metal, can inhibit this catalytic decomposition, stabilize peroxide, and protect the carbohydrates (Zeronian and Inglesby 1995). Moreover, earlier studies concerning the adsorption of carboxymethyl cellulose (CMC) on the pulp has shown that a higher electrical conductivity of the pulp slurry causes increased an amount of CMC to be adsorbed onto the pulp (Watanabe et al. 2004; Liimatainen et al. 2009). Similarly, the addition of $MgSO_4$ could increase the electrical conductivity of the pulp, which was also expected to favor greater retention of the xylan on the fibers. As a result, both better optical properties and physical properties of paper could be obtained with the usage of $MgSO_4$.

Comparison of Pulp Properties in Different Phases

Figures 10 and 11 compare the properties of the original pulp, xylan treated pulp, chelating agent-treated pulp, and pulp bleached at different temperatures, with 0.05% $MgSO_4$ dosage, from an initial pH of 11, for 120 min. The results show that the adsorption of xylan improved the strength properties of pulp, but the whiteness and brightness were decreased. The chelation pretreatment with DTPA provided a slight increase in optical properties and a reduction in strength properties.

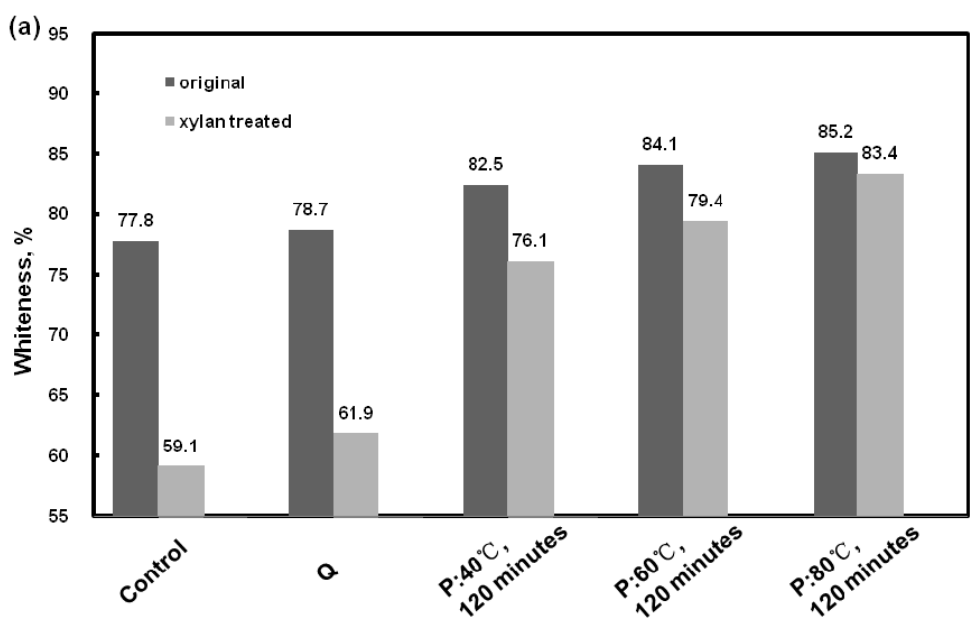

Fig. 10a. Comparison of whiteness (a) and brightness (b)

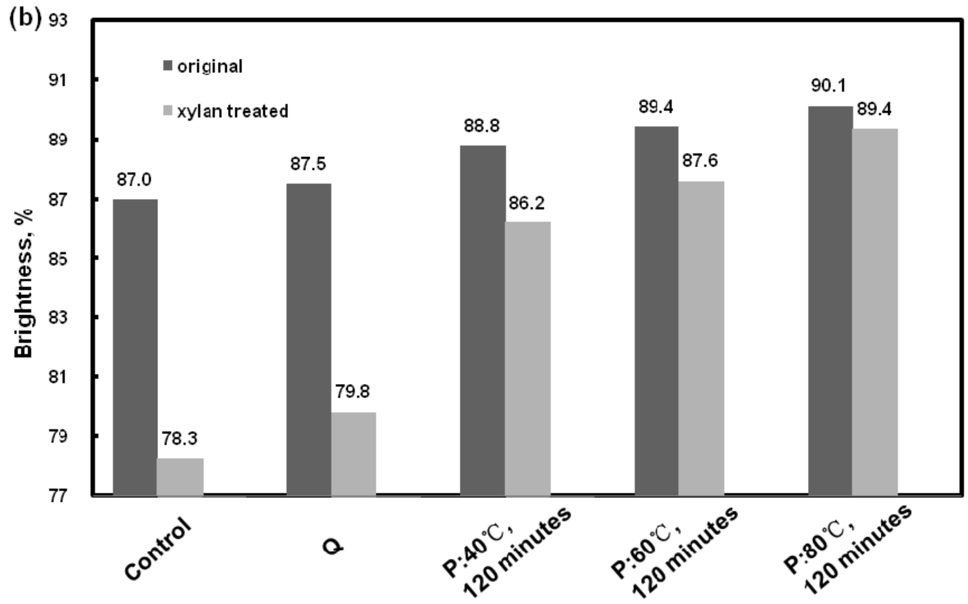

Fig. 10b. Comparison of whiteness (a) and brightness (b)

The hydrogen peroxide bleaching reduced the impact of adsorbed xylan on whiteness and brightness, while the effect on improving strength properties was still remained. Most importantly, when the bleaching process parameters were set properly, both optical qualities and physical properties were enhanced compared to the original pulp.

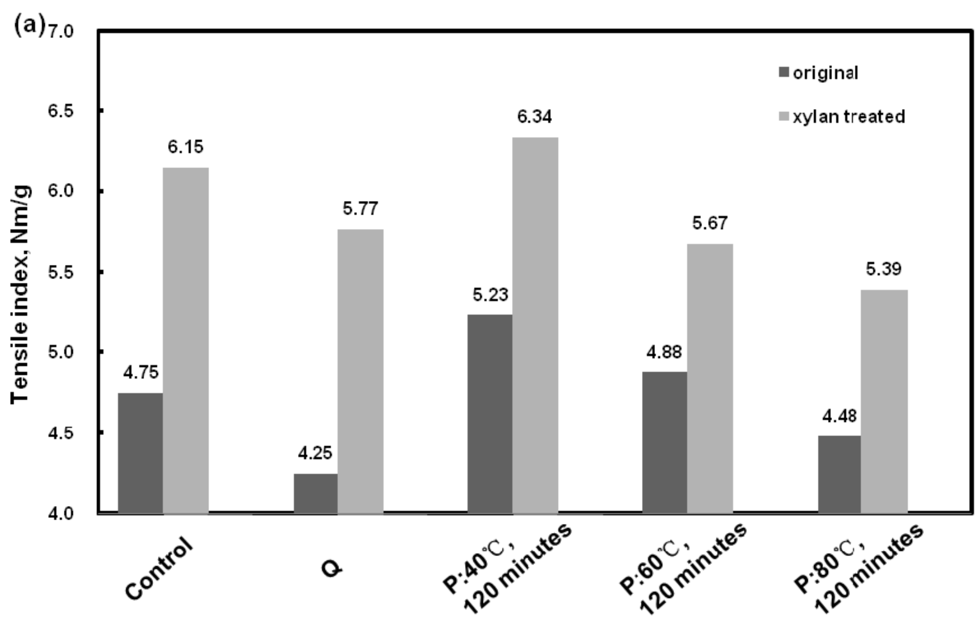

Fig. 11a. Comparison of tensile index (a) and tear index (b)

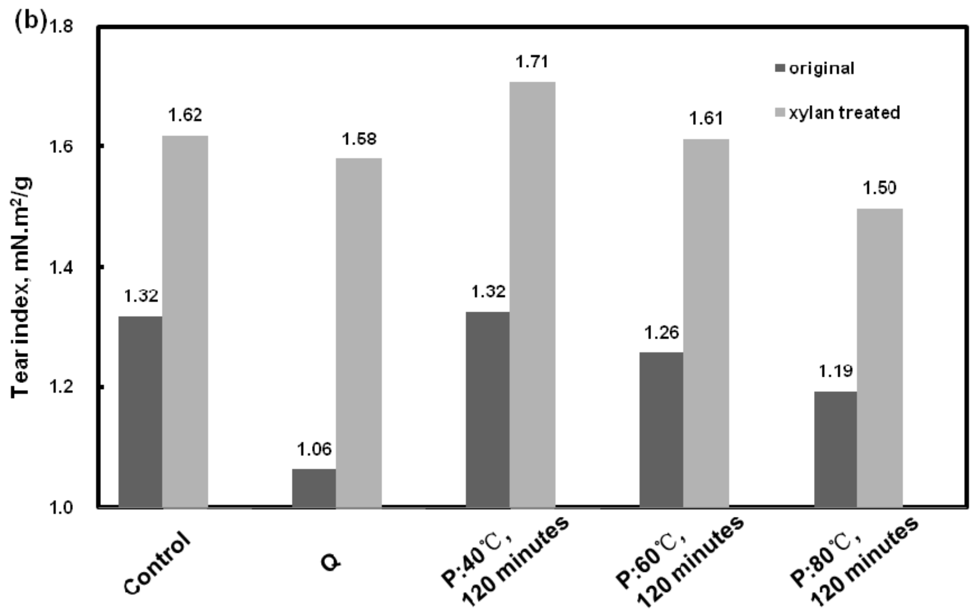

Fig. 11b. Comparison of tensile index (a) and tear index (b)

For example, when bleaching temperature was 60 °C and bleaching time was 120 min, the whiteness and brightness of bleached pulp were as high as 79.4% and 87.6%. Moreover, the tensile index and tear index were promoted by 19.4% and 22.0% respectively. Consequently, it is proved that hydrogen peroxide bleaching is a suitable bleaching process for the hardwood kraft pulp with adsorbed birch xylan.

CONCLUSIONS

1. The adsorption of birch xylan on hardwood kraft pulp fibers improves the strength properties of paper, but reduces the whiteness and brightness.
2. At low bleaching temperature, for example 40 °C, the brightness is enhanced, the opacity keeps decreasing, while the mechanical properties (i.e. tensile index and tearing index) is promoted steadily with the extension of bleaching time. On the other hand, when the bleaching temperature is high, for example 60 °C and 80 °C, the brightness is increased; the opacity and physical properties are first decreased and then improved again with additional bleaching time.
3. Higher temperature of bleaching provides better whiteness and brightness, but lower opacity, tensile index, and tear index.
4. The highest whiteness and brightness value can be obtained by bleaching from initial pH 11. In addition, with the increase of initial pH, the bulk and opacity declines, whereas the tensile index and tear index are raised significantly.
5. The whiteness, brightness, opacity, bulk, and strength properties are promoted by addition of $MgSO_4$ dosage.

6. Hydrogen peroxide bleaching is an applicable method for bleaching hardwood kraft pulp with adsorption of birch xylan. With that, both good mechanical properties and optical properties of pulp can be achieved.

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