Effects of Alkali Swelling and Beating Treatments on Properties of Kraft Pulp Fibers

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Three alkali swelling methods were used to treat two kinds of kraft pulp fibers. The morphological and chemical properties of the treated fibers were elucidated in terms of alkali concentration, with the aim of developing bulky paper and conserving wood resources. The effects of beating before and after alkali swelling were examined. The water retention value of fibers increased when higher concentrations of NaOH were used for swelling. Alkali swelling increased fiber width, while fiber length decreased. With increasing NaOH concentration, fibers became curled or kinked; the crystalline structure changed from cellulose I to cellulose II, and the crystalline index decreased. Beating before and after the alkali swelling affected the swelling behavior of kraft pulps, but there was no distinct influence on the crystalline structure. The beating treatment before alkali swelling improved the alkali swelling of fiber. However, beating after the alkali treatment diminished the alkali swelling effects. In addition, the beating after alkali swelling straightened the curled fibers.

Keywords: Alkali swelling; Beating; Fiber characterization; Morphological property; Water retention value; Sodium hydroxide concentration

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INTRODUCTION

The massive consumption of wood for paper production is a critical limitation in the paper industry because of the price of resources and ethical pressure to adopt environmentally sustainable practices. Many efforts have been made to develop eco-friendly production technologies that use less pulp to make paper. Bidirectional approaches that save resource materials by using recycled pulp (Doshi 1994) or fillers, such as precipitated calcium carbonate (Kim *et al*. 2010; Shen *et al*. 2010), or technologies that manufacture bulkier paper while maintaining a similar basis weight have been considered. Bulk is defined as the inverse of density; bulkier paper indicates thicker paper in a given area. In the latter approach, the addition of mechanical pulp, especially chemi-thermo-mechanical pulp (CTMP) (Nam *et al*. 2007; Gao *et al*. 2008; Ono *et al*. 2008; Chen *et al*. 2013) or the utilization of bulking agents, such as surfactants (Ikeda *et al*. 2003; Sone *et al*. 2005; Tomoda *et al*. 2006; Takashi 2007; Ono *et al*. 2008; Clayton 2010; Campbell and O'Toole 2010; Nam *et al*. 2014a,b), are commonly used to reduce the consumption of pulp fiber during papermaking. In addition, control of the refining, drainage, and pressing processes improves paper bulk (Eber and Janda 1992; Sung and Keller 2009; Sousa *et al*. 2011). However, these approaches may result in adverse effects, such as a decrease in paper strength (Clayton 2010; Chen *et al*. 2013; Nam *et al*. 2014a, b) or durability because of high lignin content (Takashi 2007). Alternative technologies that overcome these limitations are necessary.

The structural, physical, and chemical properties of pulp fibers affect the structural and mechanical characteristics of paper. For example, an increase in the fiber curl could enhance paper bulk but reduce paper strength. It is speculated that these paper properties can be controlled through the modification of various characteristics of pulp fibers. One method that changes the properties of pulp fibers and thus paper structure is alkali swelling (Klemm *et al*. 1998). Alkali treatment changes the structure of the fiber wall and its pore *via* a ballooning phenomenon, which might have a positive effect on paper bulk (Lawson and Hertel 1974; Le Moigne and Navard 2010; Zhang *et al*. 2013). Lawson and Hertel (1974) restored the pH of cotton fiber slurry to neutral by 1% acetic acid solution and rinsing in tap water before evaluation of fiber properties. Le Moigne and Navard (2010) and Zhang *et al.* (2013) observed the changes in fiber wall structure in alkali solution state. In addition, if increased by alkali treatment, the degree of fiber hydration may improve paper strength. The alkali swelling treatment may modify fiber properties, such as dimension and hydration degree, to potentially reduce the loss of paper strength while improving paper bulk. Therefore, it is necessary to know how alkali treatment conditions affect fiber structural and chemical and, consequently, how alkali treatment affects paper properties including bulk and strength. In addition, beating is a mechanical treatment applied to pulp fibers during papermaking to introduce desired properties into paper. Beating improves fiber bonding and hence paper strength by various effects, including fiber swelling, fibrillation, cutting, and bruising (Young 1980). A beating process could be utilized to promote alkali swelling. Hence, it is also necessary to know how a combined alkali swelling/beating process affects fiber characteristics and paper properties.

Low-concentration alkali treatment (< 2%) increases the water retention value (WRV) (Choi *et al*. 2014; Kim *et al*. 2014). The crystalline structure of cellulose fiber is unaffected by alkali treatment, while crystallinity slightly decreases, with the exception of the pulp samples with pre-beating (beating treatment before alkali swelling). Handsheets made from pulp swollen in low-concentration alkali exhibit slightly increased bulk. In this study, the effects of higher concentration alkali treatment (> 4%) on the morphological and chemical properties of kraft pulp fiber were investigated. In addition, the effect of mechanical treatment (beating) and the effect of the order of the treatments were evaluated. Two types of kraft pulps were treated with alkali swelling and beating in different sequences. For each sample, the changes in morphological properties, such as fiber length, fiber width, curl and kink, and chemical properties, including water retention value and liquid (isopropyl alcohol) retention value, were tested. Changes in the crystalline structure were also evaluated.

EXPERIMENTAL

Pulp Samples

Two types of kraft pulp, hardwood bleached kraft pulp (HwBKP) produced from acacia and softwood bleached kraft pulp (SwBKP) produced from pine, were used. These pulp samples were obtained from Hankuk Paper, Korea. For HwBKP, the weighted average fiber length was 0.81 mm, and the average fiber width was 16.8 μm. For the

SwBKP, the weighted average fiber length was 2.12 mm, and the fiber width was 30.2 μm. The coarseness of HwBKP and SwBKP were 86.7 μg/m and 183.0 μg/m, respectively.

Alkali Swelling

Alkali swelling was performed at different concentrations of sodium hydroxide (Table 1). First, a 5% pulp slurry was added to the sodium hydroxide solution, and the NaOH concentration was adjusted. The mixture was stirred using a glass rod until the pulp was dispersed. After 1 h at room temperature, the swollen pulp was filtered through a Büchner funnel with a 200-mesh wire (to exclude the influence of fiber fines). The filtered pulp was neutralized with 10% acetic acid and washed with distilled water until the pH of the pulp slurry became 7.

Alkali swelling was performed using three different processes, as follows: (A) alkali treatment of the non-beaten pulp; (BA) beating treatment followed by alkali swelling, *i.e.*, pre-beating; and (AB) alkali swelling followed by beating treatment, *i.e.*, post-beating. After each alkali treatments, the pulp slurry was neutralized and washed with distilled water until the pH of the pulp slurry became 7. Beating was performed based on the ISO method (ISO 5264-1 1979) at neutral pH using distilled water and a laboratory Valley beater (Daeil Machinery *Co*., *Ltd*., Daejeon, Korea). The freeness of each pulp sample after beating was adjusted to 420 mL CSF (Canadian Standard Freeness). The fines of each sample were removed before measuring fiber properties. All fiber properties were evaluated in the wet state after the pH of the pulp slurry was neutralized.

Table 1. Conditions for Alkali Swelling Treatments

Conditions	Contents
NaOH solution concentration (%)	0, 4, 8, 13, and 18
Temperature (°C)	23 (at room temperature)
Reaction time (min)	60
Pulp concentration (%)	5

Morphological Analysis

The morphological properties of each wet pulp fiber sample, such as fiber length, width, curl, and kink index, were measured using an L&W fiber tester (Lorentzen & Wettre, Sweden). Fibers in wood are usually straight, but they become deformed by processing, *e.g.*, beating and pumping. Fiber deformation is generally quantified using the curl and kink index. Fiber in a curl bends at a gentle curve, while fiber in a kink changes direction sharply and forms an angle. Higher values of curl and kink index indicate more deformed fibers. In addition, the dimensional shape of each pulp fiber sample was analyzed using a scanning electron microscope (SEM; Hitachi, Japan). The samples were freeze-dried and imaged at 15.0 kV accelerating voltage.

Solvent Retention Value Analysis

Solvent retention values including water retention value (WRV) and liquid retention value (LRV) were analyzed by the TAPPI (Technical Association of the Pulp and Paper Industry) method (TAPPI UM 256 2011) and the Yiannos method (Yiannos 1965). After alkali swelling or beating, 2 g (oven dried weight) of each wet pulp fiber sample was diluted to 2% concentration (w/w). The sample was drained using a glass filter (1G4) and centrifuged at 1250 × *g* for 40 min. The pulp pad was weighed and then oven-dried at 105 °C; the dried pulp pad was re-weighed. The WRV of each sample was calculated with Eq. 1,

$$WRV(g/g) = \frac{A-B}{B} \tag{1}$$

where *A* is the wet pulp pad weight and *B* is the dried pulp pad weight. LRV was measured in the same way, with isopropyl alcohol as the solvent instead of water.

X-Ray Diffraction (XRD) Analysis

Morphological changes in the crystalline structure of each pulp fiber were analyzed using a high-resolution X-ray diffractometer (PANalytical, Netherlands). The crystallinity of pulp fiber was determined from X-ray diffraction curves based on the Segal method (Segal *et al.* 1959). Crystallinity index (CI) was calculated based on Eq. 2.

$$CI(\%) = \frac{I_{002} - I_{Am}}{I_{002}} \times 100 \tag{2}$$

where I_{002} is the peak height at 22.7° (2θ) and I_{Am} is the peak height of amorphous cellulose to 18° (2θ).

RESULTS AND DISCUSSION

Fiber Width and Length

Changes in the fiber width of a hardwood bleached kraft pulp (HwBKP) and a softwood bleached kraft pulp (SwBKP) after treatment with various concentrations of sodium hydroxide, with and without beating, were investigated (Fig. 1). The fiber width of alkali-treated samples (A in the figures) increased with increasing NaOH concentration for both HwBKP and SwBKP, indicating that NaOH caused pulps to swell. Fiber width changed the most when HwBKP and SwBKP were beaten before alkali swelling (*i.e.*, pre-beating). For HwBKP, post-beating (AB in the figures) fiber width was less than that of pre-beating fibers but higher than those treated with alkali alone. The width of SwBKP post-beating fibers decreased when sodium hydroxide concentration was greater than 4%, even lower than the case of alkali swelling. However, pre-beating (BA of SwBKP) fiber width increased at higher NaOH concentration. These results suggested that alkali-treated SwBKP fibers were compressed by mechanical forces during beating. Thus, the differences between HwBKP and SwBKP post-beating fibers may have resulted from the higher compressibility of SwBKP.

Changes in fiber length resulting from alkali swelling and beating are illustrated in Fig. 2. As the concentration of sodium hydroxide increased from 0% to 13%, the fiber length of all pulp samples decreased. Above 13%, there were few changes in fiber length. These data suggest that fiber length decreased because of the increases in fiber width. Alkali treatment without beating resulted in the highest fiber length for both HwBKP and

SwBKP. Mechanical treatment (beating) increased the magnitude of the decrease in fiber length. Pre-beating and post-beating reduced fiber length more than alkali swelling alone. For HwBKP, pre-beating and post-beating treatments produced similar fiber lengths in all NaOH concentrations. In SwBKP, the magnitude of the decrease in fiber length of post-beating was higher than that of pre-beating, but lower than alkali swelling. Differences between SwBKP and HwBKP were the result of differences between the pulps rather than the treatments; SwBKP fibers were generally longer than HwBKP fibers.

The order of beating (*i.e.*, pre-beating or post-beating) may affect fiber width and length differently because of differences in the surface area of pulp fiber available for interaction with NaOH (Ingmanson and Thode 1959). The enlargement of specific surface areas by beating increases absorption of water by fibers (Eriksson *et al.* 1996). Thus, beating was expected to increase the swelling capacity. However, in case of post-beating, the magnitude of changes in fiber width and length was lower than those with pre-beating. The data indicate that post-beating mitigated the changes in the fiber width and length effected by alkali swelling treatment. The impact forces during refining squeezes some water from fiber, and post-refining after fiber swelling reduces the swelling rate (Olejnik 2012).

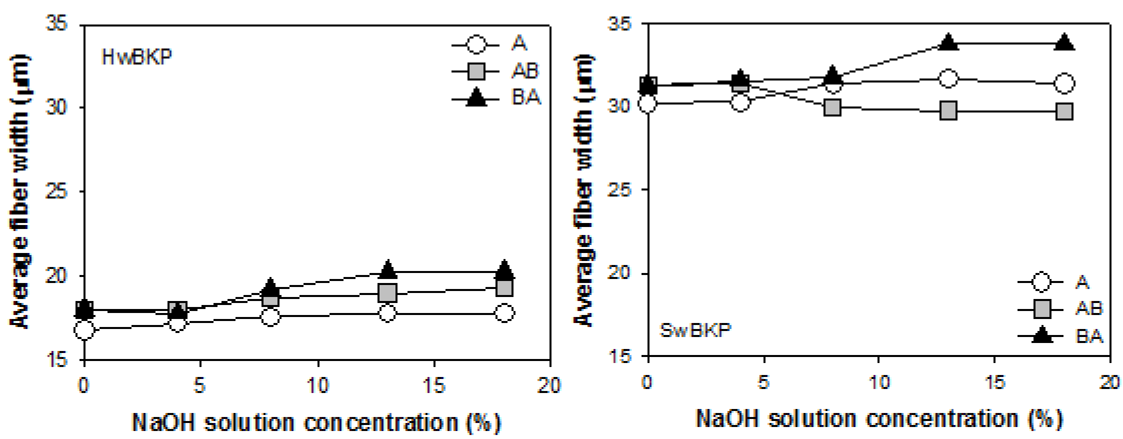


Fig. 1. Changes in HwBKP and SwBKP fiber width with NaOH concentration and alkali swelling processes. A: alkali treatment; BA (pre-beating): beating treatment followed by alkali swelling; AB (post-beating): alkali swelling followed by beating treatment.

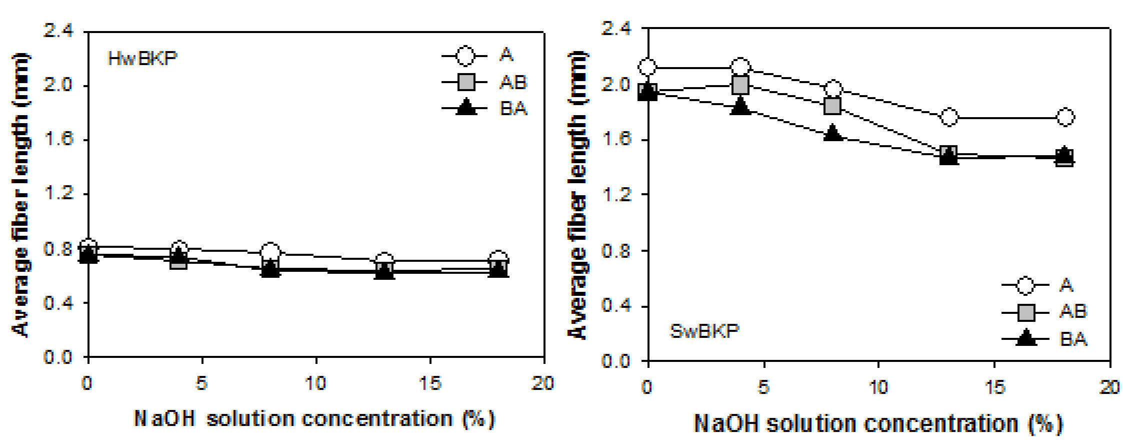


Fig. 2. Changes in HwBKP and SwBKP fiber length with NaOH concentration and alkali swelling processes

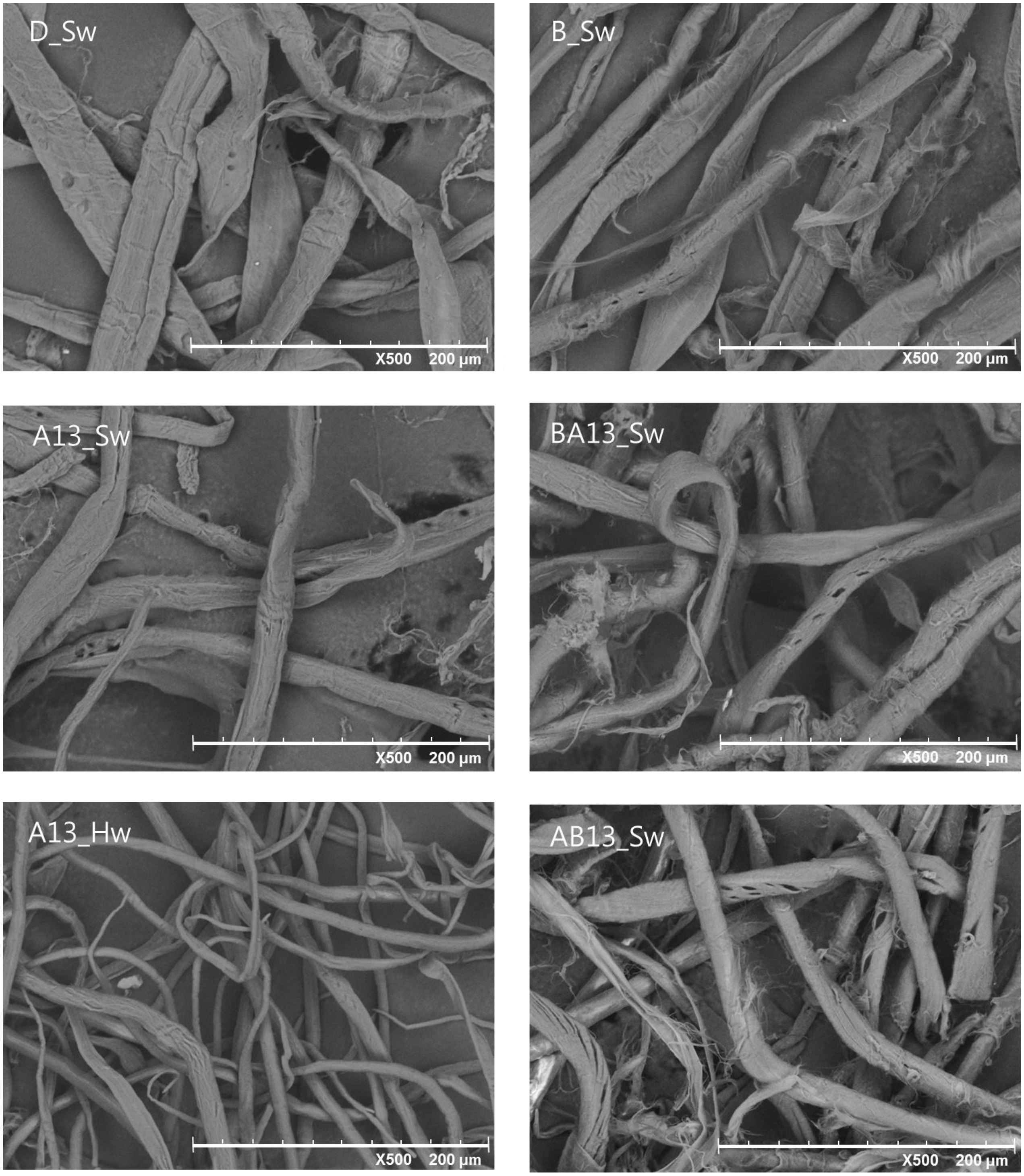


Fig. 3. SEM images of SwBKP and HwBKP fibers after beating and alkali swelling treatments. D_Sw: SwBKP fibers dispersed in water; B_Sw: SwBKP after beating treatment; A13_Sw: alkali swelling of SwBKP in 13% NaOH; BA13_Sw: pre-beating of SwBKP followed by 13% NaOH; A13_Hw: alkali swelling of HwBKP; AB13_Sw: alkali swelling of SwBKP in 13% NaOH followed by post-beating

Scanning electron microscope images of each pulp fiber sample confirmed the changes in fiber width and fiber length by alkali swelling and beating treatments (Fig. 3). Alkali-treated fiber samples were expanded in the transverse directions for both HwBKP and SwBKP (compare D_Sw, A13_Sw, and A13_Hw). After the dispersion of SwBKP in water, fibers collapsed into a thin, flat shapes (D_Sw). Beating swelled the fibers slightly (B_Sw). At concentrations above 13% NaOH, the fibers became thick and rounded in the transverse directions (A13_Sw and A13_Hw). Fibrils were found in fiber samples after beating (BA13_Sw and AB13_Sw). Pre-beating promoted swelling, as demonstrated by the more rounded shapes in the transverse directions (BA13_Sw). In the post-beaten sample, some collapsed fibers were observed (AB13_Sw).

Curl and Kink Index

The fiber curl of both HwBKP and SwBKP fibers increased as the sodium hydroxide concentration increased (Fig. 4). Fiber curl was similar in the pre-beating and alkali swelling alone samples. Notably, no distinct changes in the curl of post-beating fiber were observed with increasing alkali concentration in either HwBKP or SwBKP fibers. The results imply that post-beating treatment straightened the curled fibers, which were induced during alkali treatment. The changes in the kink index were similar to those of fiber curl (Fig. 5). For alkali treatment and pre-beating, the kink index of HwBKP and SwBKP fibers increased with increasing alkali concentration. Post-beating treatment seemed to straighten the kinked fibers.

Overall, curl and kink fiber deformations were introduced to HwBKP and SwBKP fibers by alkali swelling or pre-beating treatment. Fiber deformations affect paper properties in various ways. High curl and kink values result in the higher bulk, tear strength, porosity, and absorbency of paper (Sood *et al.* 2005). Thus, high bulk paper could potentially be manufactured by controlling fiber deformations by alkali swelling.

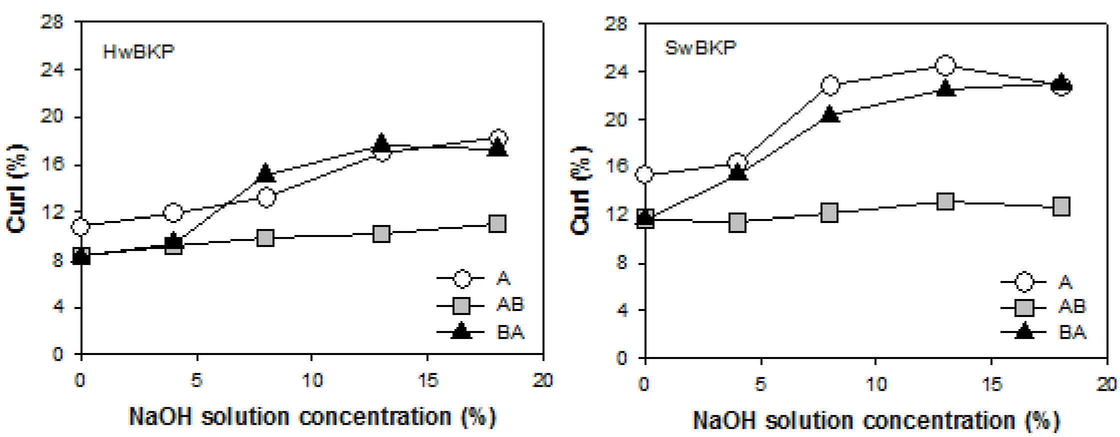


Fig. 4. Changes in HwBKP and SwBKP fiber curl with NaOH concentration and alkali swelling processes

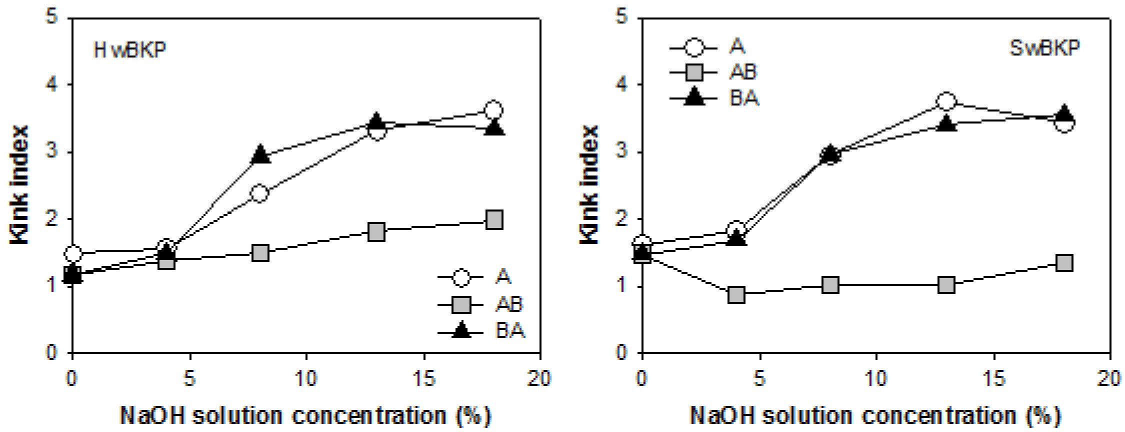


Fig. 5. Changes in HwBKP and SwBKP fiber kink index with NaOH concentration and alkali swelling processes

Solvent Retention Analysis

The WRV of wet pulp samples increased with sodium hydroxide concentration, indicating that pulp fibers swell more readily in higher NaOH concentration (Fig. 6). Beating treatment promoted fiber swelling. In the case of pre-beating, WRV increased with increasing alkali concentration, especially in HwBKP. For SwBKP, WRV slowly increased with alkali concentration in pre-beating samples but still showed the highest values among the three swelling processes. However, in the case of post-beating treatment, the WRV of HwBKP and SwBKP fibers initially increased with sodium hydroxide concentration and peaked at 4% NaOH. At 13% and 18% NaOH, the WRV values for post-beating were lower than with alkali treatment alone. A possible reason for this decrease in WRV might be the squeezing effect during beating; during post-beating, the stator and rotor bars force water out of fiber walls. Changes in the WRV of HwBKP and SwBKP samples were similar. The changes in LRV of the HwBKP and SwBKP samples were similar to those of WRV, although LRV was generally lower than WRV (Fig. 7). Higher fiber WRV often leads to more flexibility and thus higher fiber bonding during sheet forming (Lund *et al.* 2012). The increased SRV of treated pulp fibers suggested that paper strength could be improved by alkali swelling.

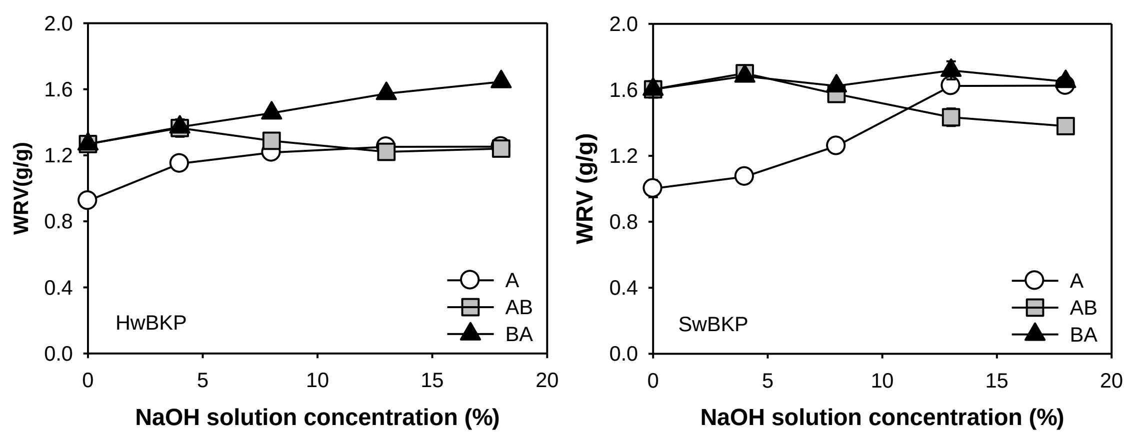


Fig. 6. Changes in HwBKP and SwBKP fiber water retention value with NaOH concentration and alkali swelling processes

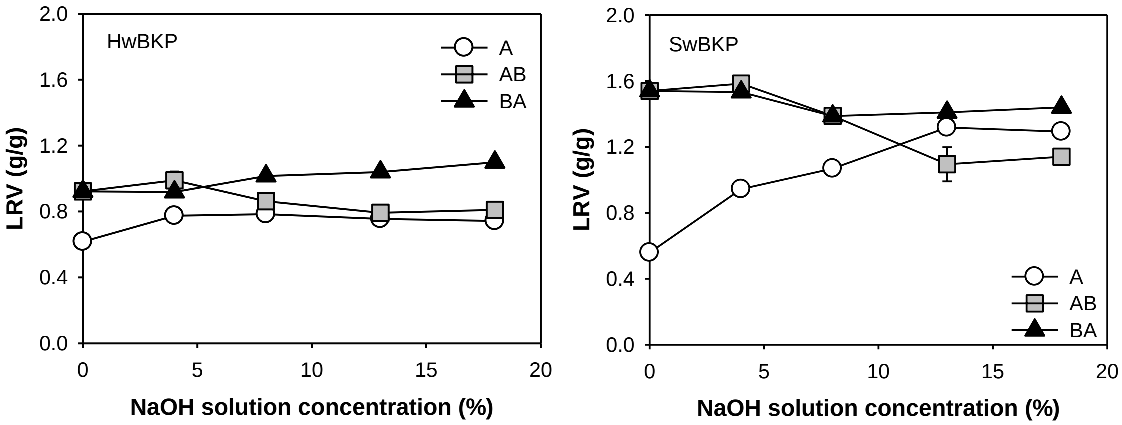
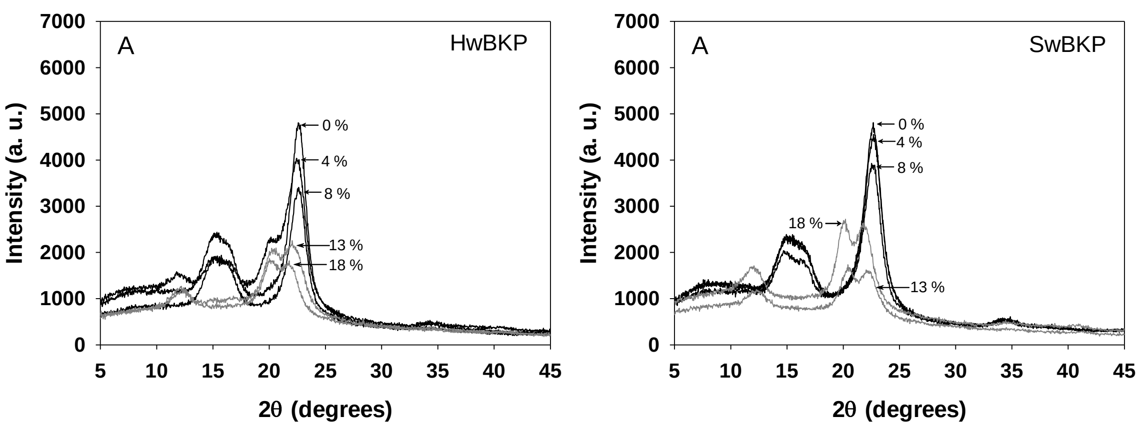


Fig. 7. Changes in HwBKP and SwBKP fiber liquid retention value (isopropyl alcohol) with NaOH concentration and alkali swelling processes

Crystalline Structure and Crystallinity Index (CI)

Crystalline structure analysis showed that all pulp samples were converted from cellulose I to cellulose II at high sodium hydroxide concentration (Fig. 8). The crystalline structure of all HwBKP fibers began to be converted at 8% sodium hydroxide. In the case of SwBKP with alkali swelling and post-beating, changes in the crystalline structure started at 13% sodium hydroxide. Comparatively, for the pre-beating treatment of SwBKP fibers, changes in the crystalline structure started at 8% sodium hydroxide, which was similar to the HwBKP samples. Pre-beating promoted changes in the crystalline structure during alkali swelling. Beating increases the surface area of fibers by fibrillating, but it also reduces the size of the crystalline zone (Nada and Youssef 1997). Thus, it was expected that beating would facilitate fiber swelling, leading to changes in the crystalline structure of cellulose fibers, even at a low concentrations of sodium hydroxide.



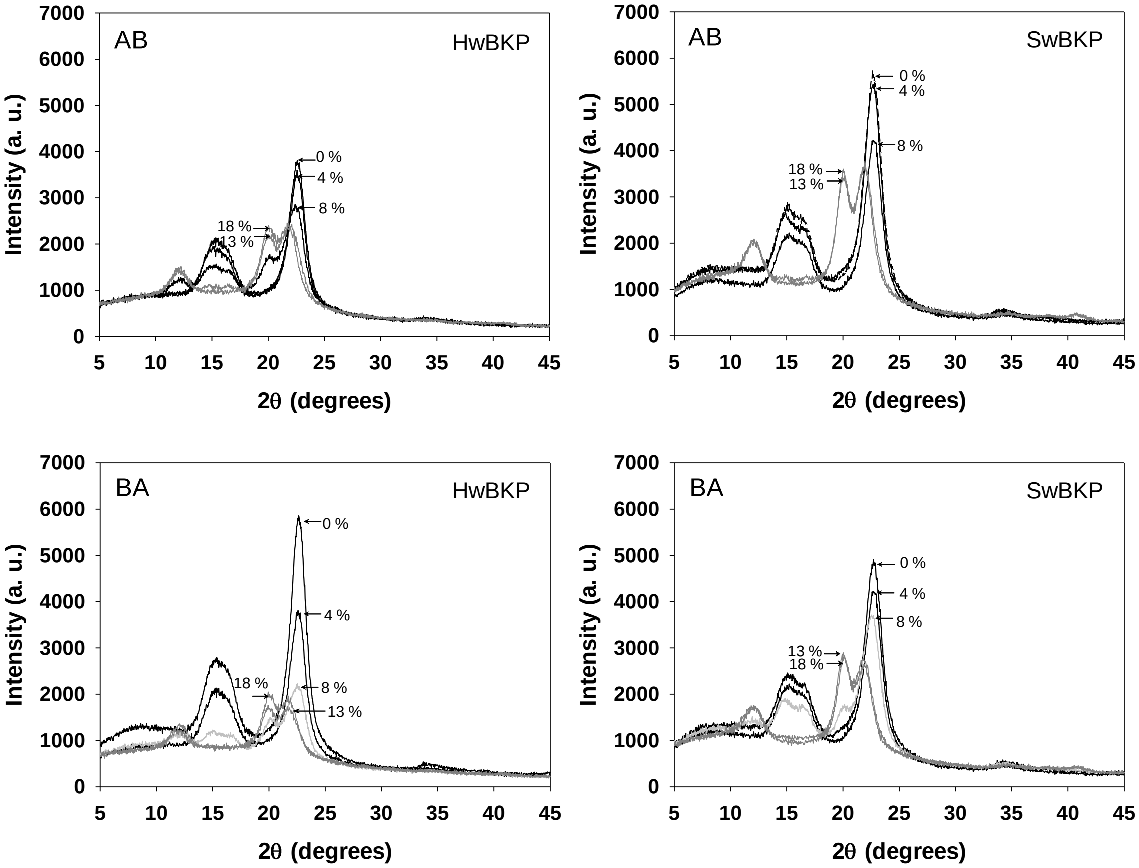


Fig. 8. Changes in HwBKP and SwBKP fiber XRD spectra with NaOH concentration and alkali swelling processes

Changes in the crystalline structure of HwBKP occurred at a lower alkali concentration than in SwBKP. These results might be associated with the difference in specific surface area between HwBKP and SwBKP. As mentioned above, a higher specific surface area is associated with a higher absorption rate in the fiber material (Eriksson *et al.* 1996). The specific surface area of HwBKP is higher than that of SwBKP, so HwBKP could be more easily swollen by alkali than SwBKP.

According to X-ray diffraction curves calculated by the Segal method (Segal *et al.* 1959), the crystallinity index of HwBKP and SwBKP fibers decreased as the concentration of sodium hydroxide increased (Fig. 9). This trend was distinct in the case of pre-beating because the crystallinity index of pre-beating samples was much lower than that of pre-beating samples and alkali swelling alone samples treated with 13% NaOH. These results imply that the increased specific surface area of fibers resulting from beating accelerated the changes in crystalline structure by alkali swelling. In comparison, post-beating showed the highest crystallinity index at high alkali concentration. Similar to crystalline structure, the decrease in crystallinity index of HwBKP occurred at a lower sodium hydroxide concentration (8% NaOH) than in SwBKP (13% NaOH).

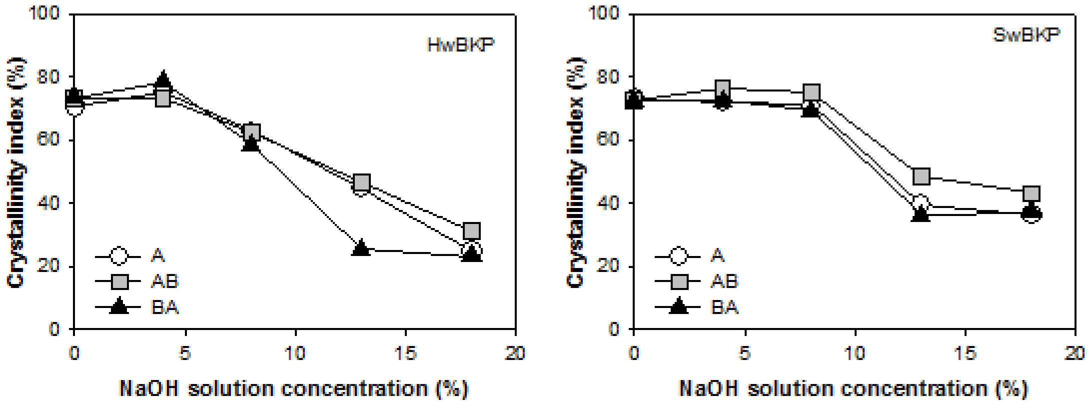


Fig. 9. Changes in HwBKP and SwBKP fiber crystallinity index with NaOH concentration and alkali swelling processes

A question left unanswered by the present work is whether the observed swelling effects, pertaining to the wet fibers, will still be reflected in properties of the resulting paper. In other words, one may want to know what will happen with respect to dimensions and bulk of the fibers and the paper as a consequence of drying during the manufacturing process. Such issues need to be addressed in future work.

CONCLUSIONS

1. The efficiency of alkali swelling was enhanced by pre-beating treatment; the mechanical force of beating promoted fiber swelling. Thus, pulp fibers modified by beating and alkali swelling could improve paper bulk through the dimensional expansion of fiber and could also improve paper strength *via* increased SRV. All evaluation was performed in the wet state after the pH of the pulp slurry was neutralized.
2. Conversely, fiber swelling with sodium hydroxide was diminished by post-beating.
3. Morphological characteristics, including fiber width and curl and kink index, and chemical properties, including SRV, increased with increasing sodium hydroxide concentration, while fiber length and crystallinity index decreased in the same conditions.
4. After various alkali swelling processes, changes in the morphological and chemical properties of SwBKP fibers were more dramatic than in HwBKP fibers.

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