EFFECTS OF PULP PREPARATION AND PAPERMAKING PROCESSES ON THE PROPERTIES OF OCC FIBERS

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Changes of the pore structure of recycled fibers and the strength properties of papers produced by old corrugated container (OCC) recycled fibers were studied, after they were subjected to different stock preparation and papermaking processes. In this paper, the effects of beating, sizing, pressing, and drying on fiber properties were investigated, and the porous structure of fibers was analyzed by nitrogen adsorption technique. The results showed that beating, pressing, and other physical processes significantly influenced the fiber properties, whereas the effects of sizing were minor. Significant changes of water retention value (WRV), crystallinity index, and paper strength were observed after those processes. Further, an effort has been made to show relationships between pore structure and macroscopic properties (WRV, crystallinity index) of recycled fibers.

Keywords: Recycled fibers; Pore structure; Crystallinity index; Tensile index

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INTRODUCTION

With the prosperous development of China's paper industry, recycled fiber has become an extremely important raw material. Full utilization of recycled fibers in the paper industry not only can ease the shortage of plant fiber resources, but also save the energy consumption, as well as effectively reduce the pollution load caused by papermaking processes. According to the statistics of the China Paper Association, the total consumption of recycled fibers in the papermaking industry was 44.39 million tons in 2008 in China, and increased to 49.39 million tons in 2009, accounting for 62% of the total fiber raw materials. It is obvious that the share of recycled fibers in total raw fiber resources will continue a steadily rising trend (China Paper Association 2010). In addition, corrugated container, as an environmental friendly packaging material, is one of the main sources of recycled fibers, with a high degree of recycling for many years, accounting for approximately 40% of the total wastepaper (Guo et al. 2011). OCC is mainly composed of used unbleached kraft pulp, bleached kraft pulp, hardwood semi-chemical pulp, and grass pulp. The major problem with recycling of old corrugated container is the loss of its strength properties (Nazhad and Sodtivarakul 2004). Some researchers have investigated the OCC pulp properties after recycling; generally speaking, the OCC pulp characteristics in terms of fibers surface properties and strength properties

of paper sheets tended to decreased with the increase of recycling times (Li et al. 2010; Wu et al. 2010; Seo 2002; Bajpai, 2010)

These various different changes in fiber morphology and surface characteristics are generally undesirable, since the changes tend to impede growth prospects for the use of recycled fibers in papermaking. As always, the main reason accounting for the inferior properties of recycled fibers is the hornification, i.e. the irreversible loss in swelling capacity of the fiber wall resulting from a drying and rewetting cycle (Jayme 1944). The mechanism responsible for hornification has been the subject of a long debate, and many classic theories have been developed to explain it. For instance, Thode et al. (1955) reported that the hornification is caused by the irreversible closing-up of micropores and cracks during drying. Stone and Scallan (1965 and 1968) carried out comprehensive studies on the effect of drying on the structure of the cell wall. It was observed that large and intermediate size pores were reduced for drying. This concept was also discussed by Jayme and Hunger (1958). Other researchers proposed the auto-crosslinking hypothesis can be as a source of fiber hornification during recycling (Back and Klinga 1963; Back et al. 1967). Reorganization and co-crystallization of cellulose chains during drying, as another source of hornification, was discussed by other researchers (Ehmrooth 1978; Ingram et al. 1974; Kulshreshtha et al. 1973; Morosoff 1974). However, the mechanism for hornification has still not been completely understood. To understand the changes occurring in stock preparation and papermaking procedures are important for overcoming the inferior properties of recycled fibers.

A significant amount of research and development efforts has been ultimately attempted to overcome the loss of paper strength made from recycled fibers (Lvov et al. 2006; Lofton et al. 2005; Laine et al. 2000, 2002, 2003; Ekevåg et al. 2004). Physical, chemical and biological treatment can be applied for fiber modification, such as beating, carboxymethyl treatment using the chloroacetic acid, adding cellulose derivatives, enzyme treatment (Blomstedt et al. 2007; Choi and Jong, 2001; Jiao et al. 1998; Pala et al. 2001; Rácz and Borsa 1997; Waterhouse and Liang 1995), etc.

As is well known, papermaking processes significantly affect fiber properties (Hubbe et al. 2003; Hubbe et al. 2007; Lyne and Gallay 1950; Khantayanuwong 2002a; Vainio and Paulapuro 2007). Previous researches on the subject have mainly focused on the changes of fiber properties as a result of single or two-step processes such as beating, pressing, and drying, etc. Indicators such as water retention value (WRV) and handsheet strength have been used to characterize the fiber properties. By contrast, in our research the effects of papermaking processes involved beating, sizing, pressing, and drying on fiber properties and the changes of porous structure within the fiber wall were studied together. The porous structure of fibers plays a critical role in papermaking (Andreasson et al. 2003; Maloney and Paulapuro 1999). Moreover, pore size distribution is an important attribute of porous structure in fiber wall, and changes of pore size and pore volume are also crucial to the ability of molecules to diffuse in and out of the fiber wall (Andreasson et al. 2003). Therefore, analyzing pore size distribution of pores within the fiber wall is justifiable to investigate the effects of different treatments on fiber porous structure. In our study, we aimed to gain a deep understanding of the mechanism of fiber properties degradation from the standpoint of pore structure, and clarify the relationships among paper sheet strength, WRV, crystallinity of cellulose, and fiber pore structure.

EXPERIMENTAL

The experiment consisted of two parts. The former part involved analysis of the porous structure of fiber wall by changing the conditions of beating, pressing, and drying. The latter part was used to compare the effects of papermaking processes on fiber properties, including WRV, crystallinity, and strength properties of handsheets.

Materials and Sample Preparation

OCC was torn into pieces of about 25 × 25 mm and soaked in water for 24 h at room temperature, then slurried with a slusher (N-197VT, Adirondack Machine Corporation, USA) at a consistency of 5% and temperature of 50 ^{o}C for 10 min. The fiber has an average length of 0.53 mm and an average width of 16.91 μm, fines content of 33.13%, measured by Fiber Quality Analyzer (FQA) (KajaaniFS300, Metso Automation, Finland).

The prepared samples were beaten in a PFI refiner according to the TAPPI T248 sp-2000 standard, to obtain pulps with different beating levels determined by Beating Degree Tester according to the GB/T3332-2004 standard. From beaten pulp, a fiber suspension with fiber consistency of 1.2% was prepared, and 0.3% of AKD (based on dry weight of pulp) was added to the pulp suspensions with continuous stirring at 6,000 revolutions. Then the mixture was subjected to the preparation of handsheets with a basis weight of 120 g/m^2 on a handsheet machine (RK3-KWTjul, Vorchdorf, Austria) with the Rapid Köthen method according to the GB/T 24214-2009 standard. Pressing was conducted on a squeezer (400-1, Labtech Company, USA) with the pressure and pressing time controlled independently, firstly, the pressure level varied from 0.1 to 0.5 MPa and pressing time level was fixed at 5 min; then the pressing time varied from 5 to 25 min with pressure at 0.45 MPa. The effects of pressure and pressing time on pulp fibers were studied respectively. Then the sheets were dried on a Formax 12'Drum Dryer (Thwing-Albert Instrument Company, USA). Drying temperature and duration were 80 and 120 ^{o}C for 15 min, 100 ^{o}C for 10, 15, and 20 min.

Determination of Pore Structure

Many different methods can be used to characterize the porous structure of the fiber wall. The most frequently used method is the Solute Exclusion Technique (SET) (Böttger et al. 1983; Stone and Scallan 1968). Other techniques, such as Inverse Size Exclusion Chromatography (ISEC) (Aggerbrandt and Samuelsson 1964; Berthold and Salmén 1997) and the Nuclear Magnetic Resonance (NMR) relaxation method (Li, et al. 1993; Li and Eriksson 1994; Maloney et al. 1997) are also available.

In this study, nitrogen adsorption measurements were used to reveal details of the porous structure of the cell wall of pulp fibers (Mancosky et al. 2004; Sawabe and Kitagawa 1978; Stone and Scallan 1965; Yu et al. 2009). The technique requires samples in dry state, because drying pulps from polar liquid such as water causes collapse of pores and a loss of internal surface (Wang et al. 2006). Thus prior to analysis, the aqueous medium suspending the fiber samples was replaced with acetone, followed by freeze-drying in freeze-drier (Modulyod-230, Thermo Company, USA). The measurement was carried out with an ASAP 2020 volumetric adsorption analyser (Micromeritics Co., USA).

N_2 was used as adsorbate, and adsorption-desorption of high-purity N_2 was determined at 77.5 K with a liquid nitrogen trap using astatic volumetric method. The pore size distribution was obtained using the Barrett-Joyner-Halenda algorithm (Barrett et al. 1951; Persson et al. 2004), the isotherm was characterzed as type Ⅳ.

In the second part, the OCC fiber samples were exposed to treatments as shown in the Table 1.

Table 1. Samples Prepared

Sample code	Beating	Sizing	Pressing	Drying
	34 °SR	0.3% AKD	0.3Mpa, 10min	100 °C,15min
a	+	+	+	+
b	-	+	+	+
c	+	-	+	+
d	+	+	-	+
e	+	+	+	air dried

+ stands for sample was subjected to the process, - means not.

The paper sheets were conditioned in a controlled environment (temperature of 23±1 ℃ and relative humidity of 50±1%) before measurement of sizing degree and strength properties. The sizing degree was determined by testing the water absorption with the Cobb method according to the GB/T 1540-2002 standard. The test was performed with 60 seconds of exposure, and the Cobb value was tested to 28 g/m^2. Tensile strength of handsheets was measured by a tensile machine (CE062, Lorentzen Wetter Company, Sweden) according to the GB/T 12914-2008 standard.

Determination of WRV

The water retention value (WRV) is an important property closely associated with fiber cellulose crystallinity and swelling capability (Forsström et al. 2005; Gumuskaya et al. 2003). The measurement of WRV was conducted using the centrifugal method with 1.5 g samples (o.d.) at 3000 g for 15 min according to ISO 23714-2007. After centrifugation, the fiber mat was weighed in a pre-weighed weighting bottle, subsequently dried in a drying oven at 105±2 ℃ for 24 h, and then re-weighed. WRV was calculated with the following equation,

$$WRV = \frac{m_1 - m_2}{m_2} \times 100\% \quad (1)$$

where m_1 is the weight of the wet pulp after centrifugation and m_2 is the weight of the dry pulp (in grams).

Determination of the Infrared Crystallinity Index

Crystallinity index was determined by infrared spectroscopy, which was carried out with a Fourier Transform Infrared (FTIR) instrument (Nexus 670, Thermo Nicolet Company, USA). During sample preparation, the freeze-dried samples were reduced to small pieces prior to mixing with potassium bromide and transformed into pellets for analysis. Crystallinity index was calculated from the relative intensities of the infrared bands, finding the ratios of 1372/2900 cm^{-1} (Nelson and O'Connor 1964),

$$\text{N.O'KI} = \frac{I_{1372}}{I_{2900}} \times 100\% \quad (2)$$

where I_{1372} represents the intensity (1372 cm^{-1}) of the band belonging to the CH bending vibration and I_{2900} is the intensity (2900 cm^{-1}) of the band belonging to the CH and CH_2 bending vibrations.

All experiments were run in triplicate, and the relative standard deviation (RSD) was used to express the errors of analysis. Each pulp sample was tested six times to obtain an average value.

RESULTS AND DISCUSSION

Effects of Different Treatments on Recycled Fiber Porous Structure

Effects of beating on porous structure of fiber wall

Dubinin (Gregg et al. 1982) divided the pores of porous solids into three groups: micropore (radius < 20 Å), mesopore (radius: 20 to 500 Å), and macropore (radius > 500 Å). In our paper we apply these standard terms to classify the pores in fibers observed from the pore size distribution detector.

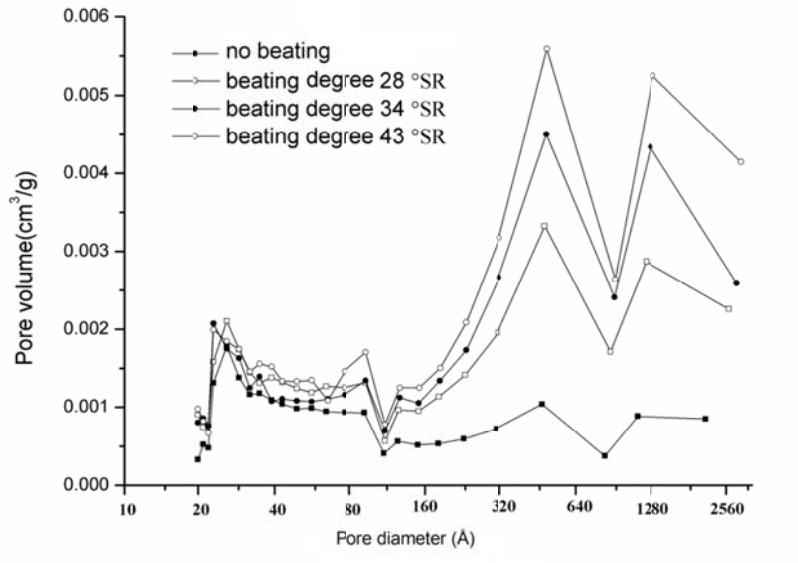

Fig. 1. Pore size distribution of OCC fibers at different beating levels

Figure 1 presents the pore size distribution of OCC fibers treated with various beating levels. Mesopores made up the majority of cell wall pore volume. The pores of cell walls were expanded to different extents by beating, and the pores size and volume increased with the development of beating levels. Two remarkable peaks can be observed at around pore diameter of 472 and 1127 Å for pulp fibers, which was attributed to the significant contributions of pores (at 472 and 1127 Å) to the pore volumes, indicating this kind of pores made up the great part of cell walls of fibers. Meanwhile, the changes of pores with pore diameter at 472 and 1127 Å were more pronounced than other pores.

Changes in pore characteristics of cell wall, such as specific surface area and volume, size, are associated with the extent of fibrillation at fiber surfaces due to the physical contributions of beating. In the early stage of beating, the primary and outer secondary walls of the fiber were disrupted and in part removed, i.e., external fibrillation (Emerton 1957). Subsequently, the refining process tended to open up submicroscopic spaces within the lamellar structure of cell walls, and created further internal delamination of fibers. Thus it was likely to contribute to the increase of pore size and pore volume. The pores with diameter greater than 120 showed more changes in comparison to those with diameter less than 120, indicating that beating significantly influenced the large-scale pores. This is in consistent with the previous report (Wang et al. 2006).

Effects of Pressing on Porous Structure of Fiber Wall

Various authors have observed that the swelling capability of fibers tended to decrease when subjected to wet-pressing conditions (Robertson 1964; Carlsson and Lindström 1984). Maloney et al. (1997) conducted the NMR analysis and found that pressing had a disproportionate effect in closure of the larger pores that were present in fibers. In the study, the effects of pressure and pressing time on pore structure of fibers were studied independently. Figures 2 and 3 present the pore size distributions for OCC fibers subjected to different pressing conditions.

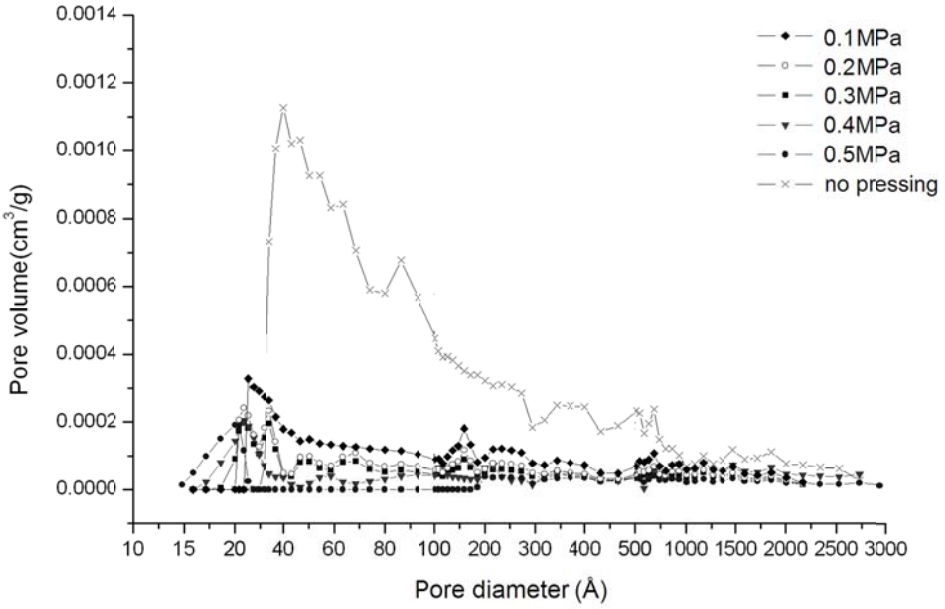

Fig. 2. Pore size distribution of OCC fibers under different pressures (pressed for 10 min)

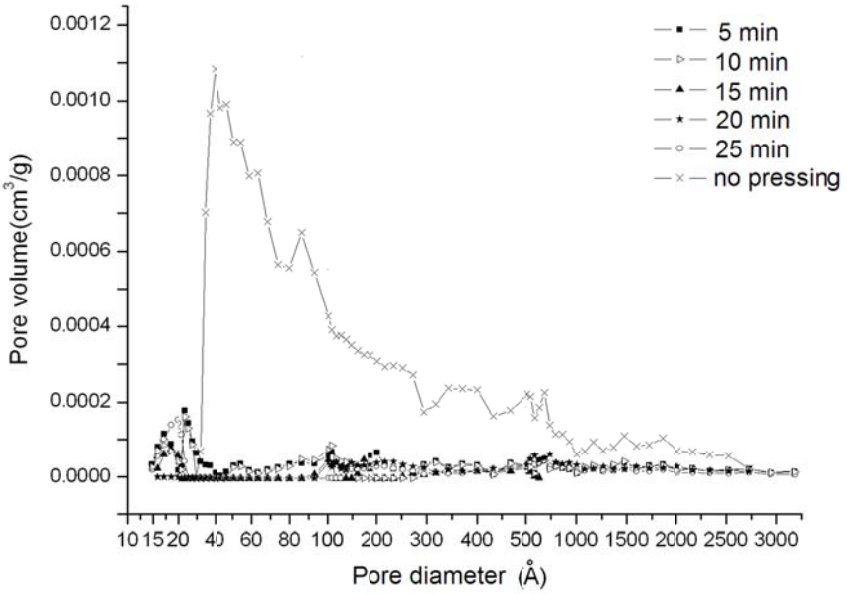

Fig. 3. Pore size distribution of OCC fibers at different pressing time (0.45 MPa)

As shown in Figs. 2 and 3, distinct differences in pore size distributions were observed for pressed and unpressed fibers. The pore volumes in cellulose fibers decreased significantly with the increase of pressing pressure and time. It is a well-documented phenomenon that pressing results in pore closure in cellulose fibers. This is because cellulose fibres shrink due to the removal of pore water by applying a pressure pulse (Bežanović and Van Duijn 2007). The moisture content in fibers is crucial for pore structure of cell wall. Various researchers have conducted similar investigations, concluding that the differences in water contents of fiber substantially influenced the pore characteristics of cellulose fibers (Häggkvist et al. 1998). For instance, Stone and Scallan (1966) observed significant decreases in the surface areas of sulfite pulp in cases where the water content was reduced to 42% or below.

As can be seen from Figs. 2 and 3, no distinct differences in pore size distributions were observed for the pulp fibers pressed 5 min and other time levels (or pressed at different pressures, as in Fig. 2), demonstrating that the pores closed severely and irreversibly once the pulp fibers were exposed to pressing treatments, regardless of the severity of pressing conditions. Moreover, the most significant changes among the pores in fibers were associated with the mesopores, showing the effect of pressing was mainly affecting the mesopores.

Effects of Drying on Porous Structure of Fiber Wall

As shown in Figs. 4 and 5, the changes of OCC fibers exposed to restrictive drying were evaluated. First, we fixed the drying time at 15 min, varying the drying temperature as 80, 100, and 120 ^{o}C. Then the temperature was fixed at 100 ^{o}C, while varying the time as 10, 15, and 20 min.

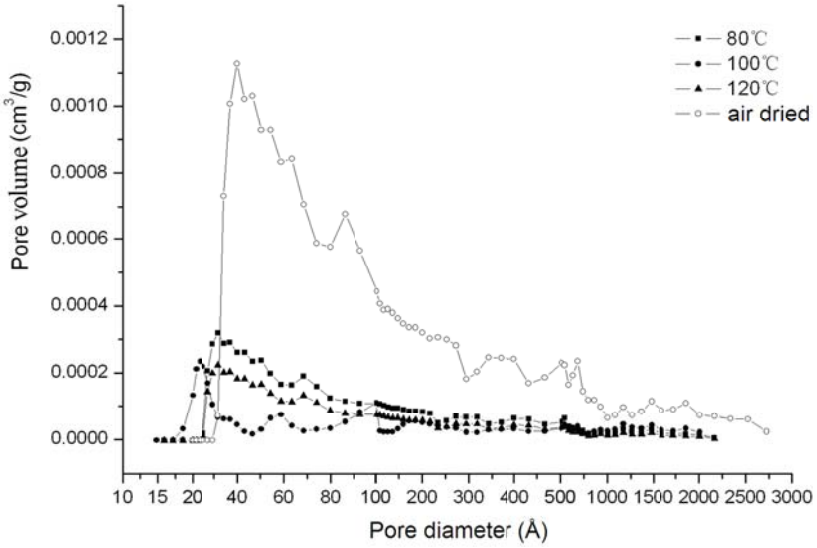

Fig. 4. Pore size distribution of OCC fibers at different drying temperatures (15min)

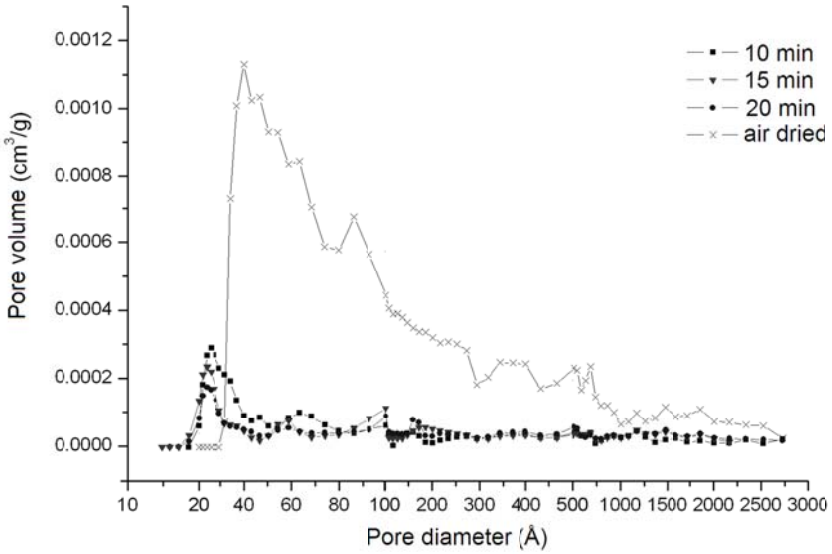

Fig. 5. Pore size distribution of OCC fibers at different drying time (100 ^{o}C)

As indicated in Figs. 4 and 5, the drying procedure significantly influenced the pore characteristic of fibers. Compared with air dried fibers, considerable losses in pore volume were observed for dried fibers, and when pulp fibers were subjected to unrestrained drying, the pore in cell wall of fibers shifted to small-scale pores, demonstrating the phenomenon of pore closure in fibers during drying process. The subject on mechanism for pore closure in fibers during drying has been investigated by many researchers (Sheikhi et al. 2010; Häggkvist et al. 1998); however, it has not been completely understood. One possible mechanism views that when water evaporates from the pore, high surface tension of water pulls the cell wall together and the pore size becomes smaller or pores suffer from closure (Park et al. 2006).

Effects of Different Treatments of Recycled Fibers on Crystallinity Index

The following figures show that the recycled fiber properties (WRV, crystallinity index of fiber, and tensile index of paper sheet) changed in response to beating, sizing, pressing, and drying, illustrating the effects of different treatments on these properties of recycled fibers. Samples subjected to treatments are listed in Table 1.

The crystallinity index obtained from FTIR spectrum of fibers subjected to different treatments and the effects of papermaking processes on crystallinity index are showed in Fig. 6. As shown, the increases in crystallinity index were observed during pressing and drying processes, and the increase of crystallinity index during drying was higher than pressing (around 15%). The explanations are related to the increase of hydrogen bonding between cellulose molecules during pressing, and the re-organization of cellulose chains and the transformation of amorphous regions to crystalline regions in cellulose during drying (Karnis 1994). Nevertheless, the crystallinity index was decreased by 6.76% after beating, which is slighting different from the results obtained by Ioelovich (2010). He noted that the crystallinity of bleached sulfate softwood cellulose fibers was increased by approximately 3.1%, which is probably because the fiber materials and measurement method were different between the two studies. However, no significant effect on crystallinity due to the sizing process was observed.

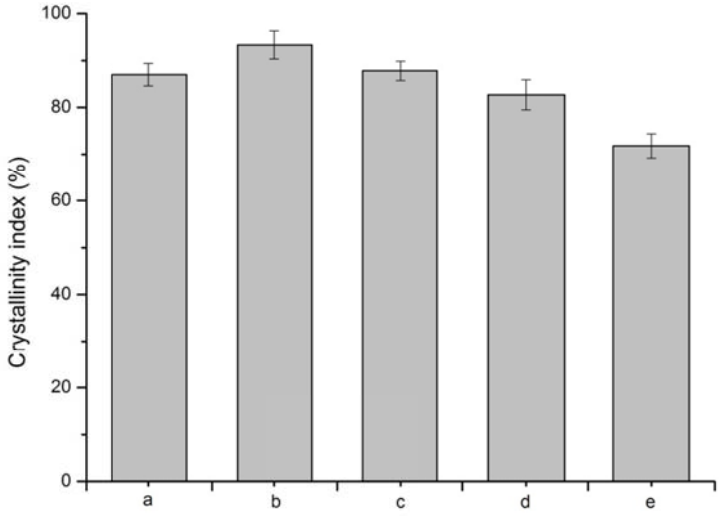

Fig. 6. Changes of crystallinity index with different treatments
(A pair of bars denotes a rage of 95 % confidence level,
a: all treatments,b:no beating;c:no sizing,d:no pressing,e:no drying)

Effects of Different Treatments of Recycled Fibers on WRV

Figure 7 illustrates the effects of different treatments (beating, sizing, pressing and drying) on WRV. The beating operation had the most significant effects on swelling capability, compared to other processes, and the WRV was considerately enhanced by 24.7% after beating. This is attributed to the delamination behavior, an important feature induced by beating (Chevalier-Billosta et al. 2007). The delamination of cell wall created large pores, which improved the water-carrying capacity of the fiber. At the same time, fiber swelling was accompanied by an increase in fiber flexibility and fibrillation in the

beating process (Sheikhi et al. 2010), and as a result the WRV increased. No significant changes in WRV were found for the sizing process. However, the swelling capability of fibers deteriorated to some extent during the pressing and drying processes, for which the losses of WRV were 4.75% and 8.57%, respectively. The explanations may be related to a collapse of the pores upon pressing and drying, which reduced the water-carrying capacity of the fiber (Brancato et al. 2007).

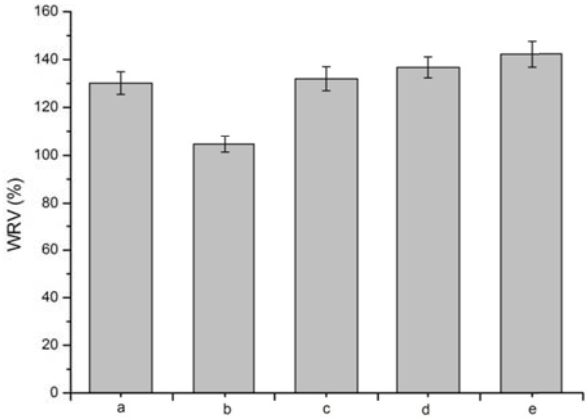

Fig. 7. Changes of WRV with different treatments
(Limit bars denotes a range of 95 % confidence level,
a: all treatments, b: no beating; c: no sizing, d: no pressing, e: no drying)

Effects of Different Treatments of Recycled Fibers on Paper Sheet Strength

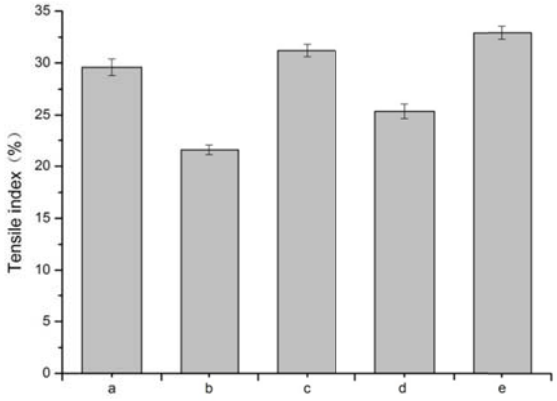

Fig. 8. Changes of tensile index with different treatments
(Limit of bars denotes a range of 95 % confidence level,
a: all treatments, b: no beating; c: no sizing, d: no pressing, e: no drying)

As shown in Fig. 8, sizing had less influence on tensile index of paper sheets compared with other processes. The tensile index of handsheets was decreased by 5.13% after sizing in comparison with sample c and a. Nevertheless, the strength property of paper was greatly increased by beating and pressing, compared with unbeaten and unpressed samples, the increase of tensile index were 37.2% and 16.7%, respectively. The explanation for this increase may be related to internal fibrillation and reinforced

hydrogen bonding between fibers during beating process (Wang et al. 2003). In relationship to the pressing process, it densified the sheet structure, resulting in the increase of bonded area and numbers of bonds, which was favourable for improving inter-fiber bonding (Vainio and Paulapuro 2007); consequently the tensile strength of paper sheet was developed. However, tensile index of handsheet was decreased by 10% after drying, which may be due to the collapse of pores and loss in fiber swelling capability, the characteristics of hornification upon drying (Nazhad 1994).

The Relationships among the WRV, Crystallinity, and Pore Structure of Fiber Wall

It is well known that pore structure within cell wall, swelling capability and cellulose crystallinity, are important for the strength of paper made from the fibers (Andreasson et al. 2003; Sheikhi et al. 2010; Hubbe et al. 2007; Chevalier-Billosta et al. 2007; Nanzhad 1994). The opened pore structure is favorable for the absorption of water molecules to fibers (Stana-Kleinschek et al. 2001), which will result in the increase of fiber swelling and flexibility. Meanwhile, fiber flexibility and swellability are the main contributors to the strength of interfiber bonding (Sheikhi et al. 2010). On the other hand, in the structure of cellulose, the crystalline regions are interrupted every 60 nm with non-crystalline amorphous regions for all raw materials (Sheikhi et al. 2010). Therefore, the crystalline structure of cellulose substantially affects the physical and mechanical properties of cellulose fibers. In addition, the fiber swellability is closely associated with crystalline structure of cellulose (Kongdee et al. 2004; Khantayanuwong et al. 2002b). The water does not penetrate into crystalline domains of cellulose (Salmen 1988); thus the increase in crystallinity of fibers will reduce the water up-take by the cell wall, as a result the swelling ability of fibers will be restricted (Nazhad 1994). In other words, the flexibility of fiber is inversely related to the cellulose crystallinity.

CONCLUSIONS

Changes of pore structure in fibers under different conditions and effects of recycling processes (beating, sizing, pressing, and drying) on WRV and crystallinity of OCC pulp fibers, as well as the tensile strength of paper sheet made from the recycled fibers were studied. Relationships of pore characteristics of cell wall within fibers, WRV, and crystallinity of cellulose were also evaluated.

The above-mentioned papermaking processes affected the porous structure of cell wall within fiber. At the beating level at 34 °SR, the fiber swelling capability was substantially developed, resulting from the expanded pores produced in beating, while the crystallinity of cellulose was slightly decreased. Compared with other processes, sizing had less impact on fiber properties. Both pressing and drying processes considerately influenced the fiber properties, with greater changes occurring especially in the drying process. The fiber lumens were collapsed and pores closed irreversibly, inducing the loss of swelling capability. Meanwhile, the transformation of amorphous region to crystalline form promoted the increase of cellulose crystallinity.

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