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Quantitative Characterization of Internal Fibrillation of Pulp Fiber

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ABSTRACT

Internal fibrillation of pulp fiber is an important factor affecting paper properties. Internal fibrillation of pulp fiber is usually introduced with several kinds of modifications of fiber by the mechanical treatment such as refining, high shear and/or high consistency mixing, etc. Unfortunately there are no standardized methods that can characterize the extent of internal fibrillation and its contribution on the paper properties. The purpose of this study is to try and find the potential methods that can characterize the internal fibrillation of pulp fiber quantitatively. Softwood bleached kraft pulp was treated with Hobart mixer to introduce the internal fibrillation without the significant fiber damage and external fibrillation. The extent of internal fibrillation was increased with the increase of mechanical treatment consistency. Several fiber properties were measured to find the potential means that could characterize and quantify the internal fibrillation.

Laminated area could not be used as a means for quantifying the internal fibrillation because of the effect of swelling and the different internal fibrillation behavior at different mechanical treatment consistency. Micro and macro internal fibrillation models were proposed for describing the different behavior for the mechanical treatment at low and high consistencies of pulp. The Internal fibrillation showed good correlation with swelling of fiber wall. This trend was confirmed through the measurement of wall thickness and/or cross section area of fiber. Therefore the internal fibrillation possibly can be described as the indices indicating the change of wall thickness and/or cross section area.

Keywords : *internal fibrillation, wall thickness, swelling, micro internal fibrillation, macro internal fibrillation*

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1. Introduction

The requirements of paper properties are greatly depend on the end uses. Such paper properties can be controlled and met to the end uses by several means such as the selection and combination of pulp fiber, refining, wet end additives (retention aids, sizes, fillers, strengthening agents, dyestuff, etc.), forming methods, wet pressing, calendering and converting. The first choice is the selection of pulp type. The pulp properties are varied by the species, pulping process, bleaching methods etc. Characteristics of pulp fiber are greatly affected by the species and pulping methods. One of representative example is that sulfite pulp is more flexible and can be easily refined than the kraft pulp. Pulp properties also can be modified and controlled by the refining. The main effects of refining are external fibrillation, foliation, internal fibrillation, fiber shortening by cutting, and fiber deformations (curl, kink, micro-compression, etc.). Many researches on the effects of refining were carried out for a long time. However, the information on the internal fibrillation is not enough to assess the contribution of internal fibrillation on the paper properties because the quantification method of internal fibrillation is still not standardized and the measurement is not easy.

Internal fibrillation as a main effect of refining has never been questioned, and at least some internal fibrillation or splitting must occur in all bar-fiber actions during refining. If the fibrous elements are split far enough apart to remain separated state, the fiber becomes very much less rigid than if the elements were joined, since the ease of bending a bundle of round rods depends inversely on the fourth power of their diameters. The ability of fibers to deform plastically is increased by the process of internal fibrillation. Internal fibrillation means that the disruption of the lateral bonds between adjacent molecules within the amorphous polysaccharide phase. This process commences in the case of dry fibers when they are immersed in water. In the dry state their total surface area accessible to nitrogen molecules is less than 1 m^3/g (1). This value is very close to the external surface area of unbeaten fibers obtained by other methods so that if there are any voids in the air dry fibers these must be largely sealed off (2). Won etc. (3-4) confirmed that the mechanical treatment with Hobart mixer can be used as a means to introduce the internal fibrillation into fiber, and found that such mechanical treatment increased flexibility and WRV of fiber, Scott bond, apparent density and tensile index of paper. Similar treatment on OCC was applied to improve the papermaking properties of secondary fiber (5-7).

Internal fibrillation can be defined as delamination of cell wall, splitting of cell wall, disruption of the lateral bond between adjacent molecules within fiber (8) and breaking of intra-fiber hydrogen bonding. The internal fibrillation can be introduced by the refining and mechanical treatment at medium and/or high consistency, and fluidization at medium consistency etc. We have adopted the mechanical treatment with Hobart mixer to introduce the internal fibrillation without fiber damage and external fibrillation.

2. Experimentals

Softwood bleached kraft pulp was used as a raw material to introduce the internal fibrillation and to find the possible characterizing methods of the internal fibrillation. The pulp was treated with Hobart mixer at

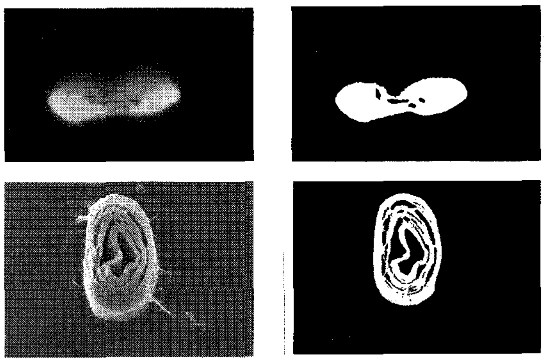


Fig. 1. Images obtained from CLSM (top) and SEM (bottom).

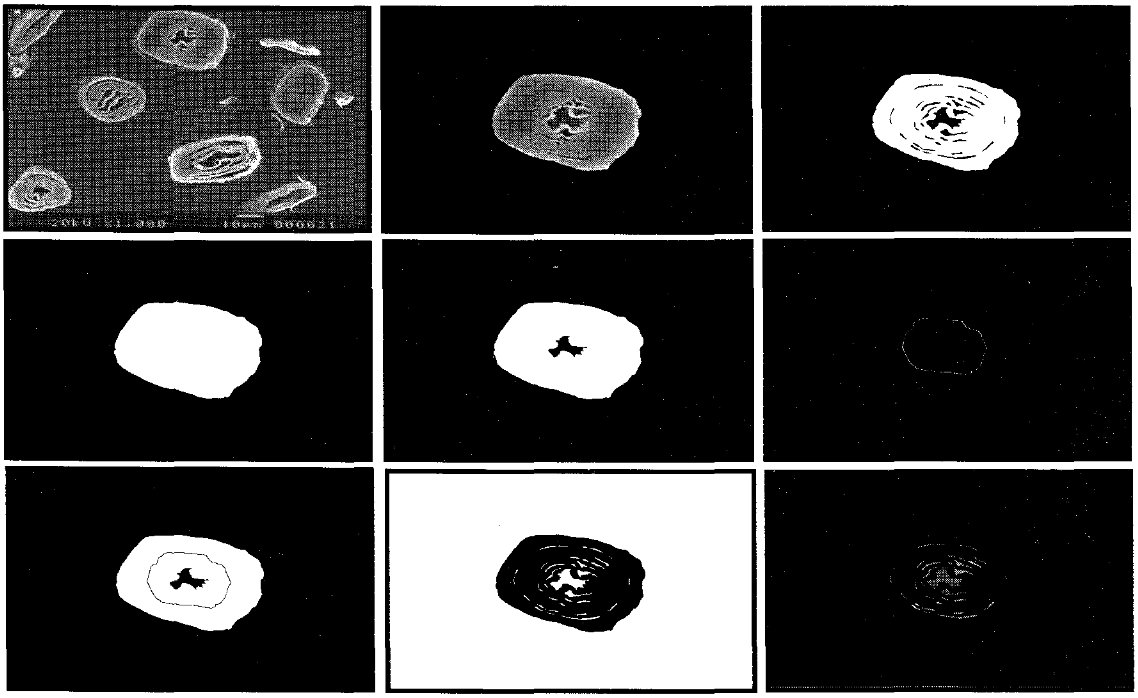


Fig. 2. Various images for image analysis of fiber cross section.

several pulp consistencies (5%, 15%, 30% and 45%) for 30 min. to introduce the internal fibrillation. The obtained fibers are pretreated with ethanol-water mixtures (50%, 70%, 80%, 90%, 95%) and anhydrous absolute ethanol to remove water. The dehydrated fibers are embedded with epoxy resin (Epon 812). The thin section is made from embedded fiber with glass knife. The sectioning was repeated until good cross sections are obtained in order to avoid inclined cross section. The changes in several properties of fiber including circular ratio, circle filling, cross section area, delaminated area, wall thickness, lumen area, collapse index, aspect ratio, sum of perimeter of delaminated portion, etc. were measured to find the possible methods for characterization of internal fibrillation. SEM images were used for this study because the clear images could not be obtained with CLSM as shown in Fig. 1. Although the only one hundreds of fibers for each treatment condition were used for measurement one can find some trends in the internal fibrillation and measured values.

Fiber properties were measured and calculated from the SEM images as shown in Fig. 2 for characterizing of internal fibrillation as follows.

• Circular ratio (CR) :

$$CR = \frac{R_{\max}}{R_{\min}} \quad [1]$$

R_{max} : Maximum radius
R_{min} : Minimum radius
Circle = 1

• Circle filling :

$$C_{Fil} = \frac{Object_{area}}{\pi \times R^2_{\max}} \quad [2]$$

• Collapse index (CI) :

$$CI = 1 - \frac{LA}{LA_0} \quad [3]$$

LA : Lumen area obtained from the cross-sectional image of fiber after treatments

LA_o: Lumen area obtained from the cross-sectional image of fiber before treatment

- Aspect ratio (*AR*) :

$$AR = \frac{F_t}{F_w} \qquad [4]$$

F_t : Fiber thickness
F_w : Fiber width

- Fiber wall Thickness (*FWT*) :

$$FWT(1) = \frac{P_0\sqrt{(P_0^2 - 4\pi A_{FW})}}{2\pi} \qquad [5]$$

$$FWT(2) = \frac{A_{FW}}{P_c} \qquad [6]$$

P_o : Outer perimeter (㎛)
A_{FW}: Area of fiber wall cross section(㎛^{2})
Pc : Center line perimeter (㎛)

3. Results and Discussion

Cross section areas without/with delaminated area were increased with the increase of mechanical treatment consistency (Fig. 3 and 4). The change of wall thickness in thick walled fiber and mixed fiber was well fitted with the internal fibrillation, but the

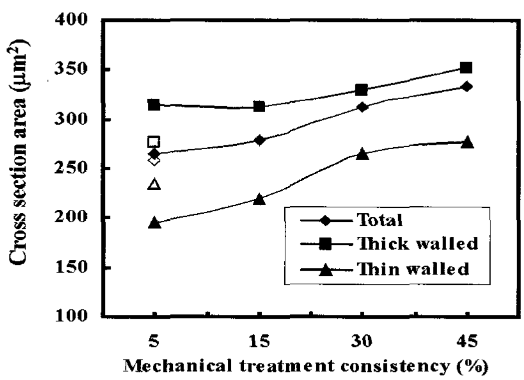


Fig. 3. Effect of mechanical treatment on the cross section area without delaminated area.

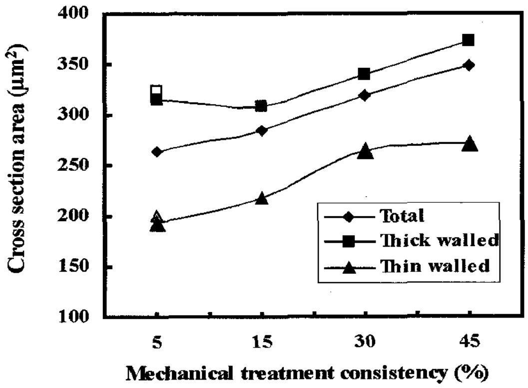


Fig. 4. Effect of mechanical treatment on the cross section area with delaminated area.

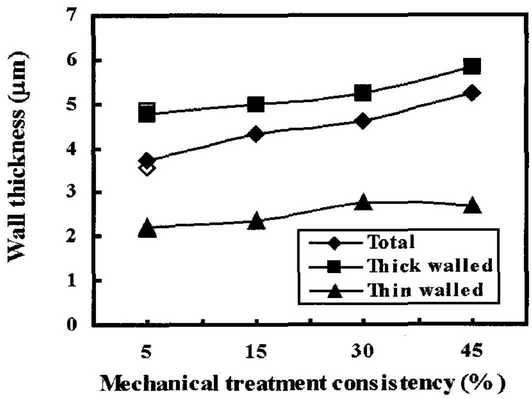


Fig. 5. Effect of mechanical treatment on the wall thickness of fiber.

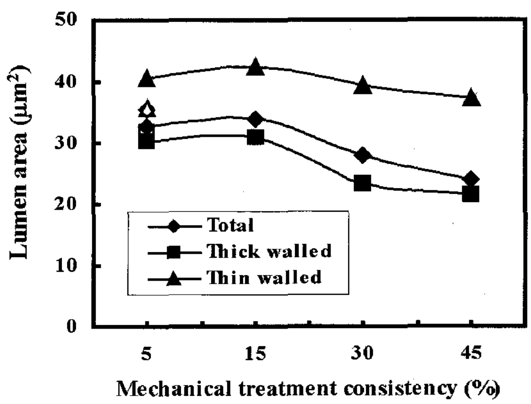


Fig. 6. Effect of mechanical treatment on the lumen area of fiber.

trend in thin walled fiber was negligible (Fig. 5). The obvious trend in the lumen area was not observed (Fig. 6).

The delaminated area was increased with the increase of mechanical treatment consistency. However the delaminated area below the 30% consistency and the delaminated area of thin walled fiber were smaller than those of untreated fiber (Fig. 7). Sum of perimeter of delaminated portion showed also similar to those of delaminated area (Fig. 8). These results may come from both the swelling during mechanical treatment and the different behavior of

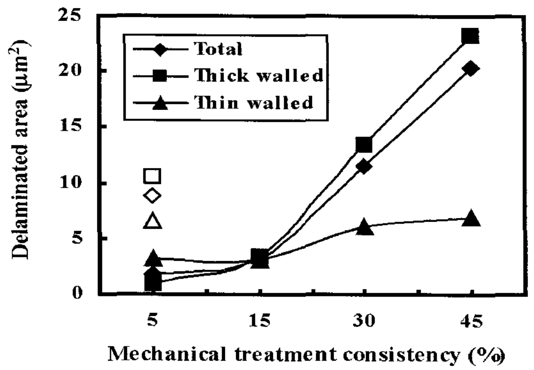


Fig. 7. Effect of mechanical treatment on the delaminated area of fiber.

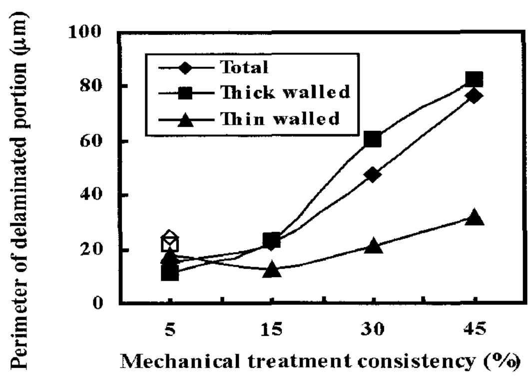
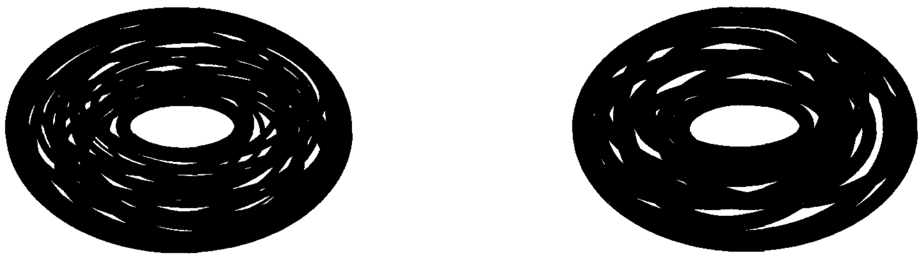


Fig. 8. Effect of mechanical treatment on the perimeter of delaminated portion.



Micro internal fibrillation at low consistency　　Macro internal fibrillation at high consistency

Fig. 9. Micro internal fibrillation and macro fibrillation model.

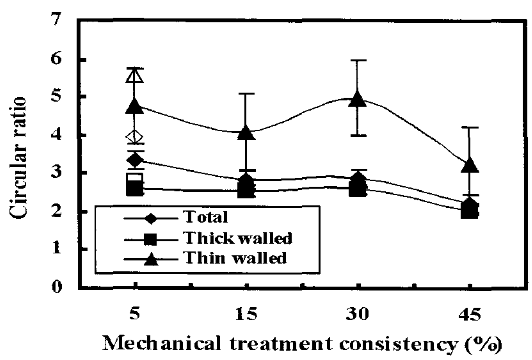


Fig. 10. Effect of mechanical treatment on the circular ratio of fiber.

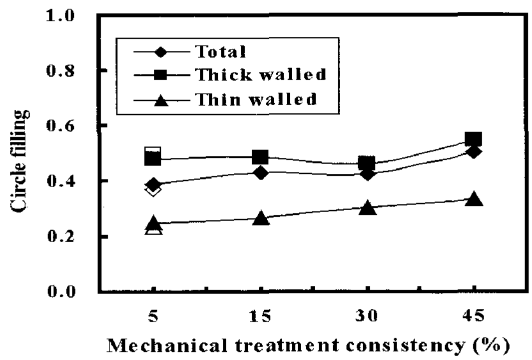


Fig. 11. Effect of mechanical treatment on the circle filling of fiber.

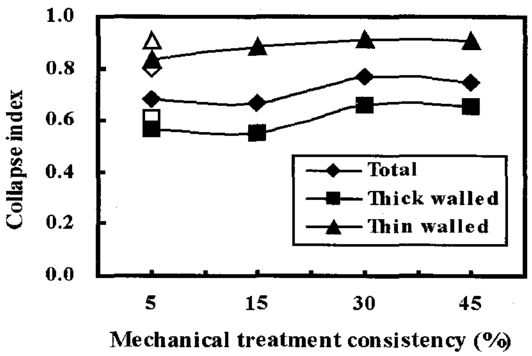


Fig. 12. Effect of mechanical treatment on the collapse index of fiber.

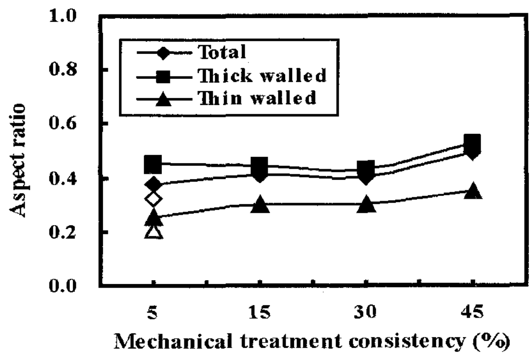


Fig. 13. Effect of mechanical treatment on the aspect ratio of fiber.

internal fibrillation at different mechanical treatment consistency (micro internal fibrillation at low consistency and macro internal fibrillation at high consistency) as shown in Fig. 9.

The circular ratios of thick walled fiber and mixed fiber were decreased slightly with the increase of mechanical treatment consistency, but the standard error in thin walled fiber was too large to describe any trends (Fig. 10). The circle filling (Fig. 11), collapse index (Fig. 12) and aspect ratio (Fig. 13) did not showed significant increase with the increase of mechanical treatment consistency

4. Conclusion

Softwood bleached kraft pulp was treated with Hobart mixer to introduce the internal fibrillation without significant fiber damages and external fibrillation. The extent of internal fibrillation was increased with the increase of mechanical treatment consistency. Several fiber properties were measured to find the better means which can characterize and quantify the internal fibrillation. Laminated area could not be used as a means for quantifying the internal fibrillation because of the effect of swelling and the different internal fibrillation behavior at different mechanical treatment consistency. Internal fibrillation showed good correlation with swelling of fiber wall. This trend was confirmed through the measurement of wall thickness and/or cross section area. Therefore the internal fibrillation possibly can be described as the indices indicating the change of wall thickness and/or cross section area.

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