Paper V

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研　究　報　文

外部フィブリルが填料リテンションに及ぼす影響

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Effect of External Fibrillation on the Retention of Filler

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The purpose of this study was to examine the effect of external fibrillation of chemical pulp fibers on the retention of filler during sheet forming in a high–vacuum dewatering device. An ultra–fine friction grinder was used to prepare fibers with different degrees of external fibrillation, while keeping their internal fibrillation constant.

The entrapment of filler particles was found to increase with increased internal fibrillation of fibers in the presence of chemical aid, and it can be further increased by promoting mostly external fibrillation of fibers. Increasing the degree of external fibrillation alone, without changing the internal fibrillation, can be used as means to control the retention of filler.

Keywords：External fibrillation, Internal fibrillation, Ultra–fine friction grinder, Filler, Retention
分類：Y_1 基礎科学一般，Y_4 セルロース化学

1.　Introduction

The retention of filler on the forming wire is critical to the optical properties of paper, and to the efficiency and cost of the papermaking operation. The retention of filler particles depends on the chemical and mechanical interactions with other papermaking components in the web forming process[1]. To gain a better understanding of this interaction, a lot of attention[1-6] has been paid to examining the use of a variety of chemical aids such as retention and drainage aids, and the operating conditions in a laboratory–scale drainage tester. Some studies[7-9] have focused on the role of refining of pulp for the retention of filler. According to these studies, the retention of filler particles increases with the beating of pulp, and the increased surface area caused by refining apparently results in increased filler retention. The surface area can be increased by both internal and external fibrillation caused by refining. However, the relative contribution of fibrillation to the retention is not known, because compressive and shear forces coexist in conventional refiners and tend to produce a variety of simultaneous refining effects. If these effects were separated and their specific role for the retention of filler were known, it would be possible to control retention more effectively.

In a previous study[10], the fibrillation of fibers was controlled by using an ultra–fine friction grinder, which primarily produced increased external fibrillation, while keeping internal fibrillation constant, which made it possible to examine the role of external fibrils for paper strength. This technique was found to be useful for the objective of the present study, which was to evaluate the role of external fibrillation of chemical pulp fibers for the retention of filler during sheet forming in a high–vacuum dewatering device, a moving belt former (MBF)[11].

2. Experimental

2.1 Pulp used

A once–dried bleached kraft softwood pulp, consisting of a mixture of Scots pine (*Pinus silvestris*, 56%) and Norway spruce (*Picea abies*, 44%), was obtained from a Finnish pulp mill. The pulp was elemental chlorine free (ECF) bleached. The pulp was disintegrated for 10 min in a Valley beater (ISO 5264–1 : 1979).

2.2 Control and evaluation of fibrillation

The disintegrated pulp was refined by recirculating it up to 22 times through a gap of 240 and 250 μm in an ultra–fine friction grinder (Masuko Sangyo Co. Ltd., Japan) under the conditions shown in **Table 1**. The details of the operation of the grinder are described in an earlier publication[12].

After collecting treated pulp, the fines passing through a 100–mesh screen were removed using a Bauer–McNett classifier. The fiber fraction (R 100) was used for all experiments except for SR measurement, for which the whole pulp was used. Fiber length and curl were measured using a FiberLab 3.0 (Metso Automation). The average length–weighted fiber length is reported in this paper.

The fiber saturation point (FSP) was measured with the solute exclusion technique using a 2×10^6 Dalton dextran polymer (Amersham Biosciences AB, Uppsala, Sweden) to evaluate the change in internal fibrillation of fibers[13], while a scanning electron microscope (SEM, DSM 962, Zeiss) and a light microscope operated in phase contrast mode (DM LAM, Leica) were used for observing the external fibrillation of fibers. At least 20 images taken with the light microscope, each containing several fibers, were then further analyzed using an image processing tool in the Matlab to calculate the proportion of the pixel size of external fibrils to fibers and expressed in percent, which allowed making a quantitative evaluation of the degree of external fibrillation[14].

2.3 Retention of filler

A high–vacuum dewatering device, the moving belt former (MBF)[11], was used for mixing filler with fibers and for forming sheets. A high vacuum pulsation rate is produced in the MBF, which makes it possible to simulate the dynamic dewatering phenomena in the wire section of a paper machine.

To evaluate the interaction of filler with fibers (R 100), precipitated calcium carbonate (PCC, Scalenohedral, FS–260, J.M.Huber, mean particle size 2.6 μm) was used as a filler. PCC was added to the fiber suspension having a pH of 7.7–7.8, both in the presence and absence of cationic polyacrylamide (C–PAM, Fennopol K 3400 R, Kemira, MW : 6.7—10^6g/mol, charge density : 1.1 meq/g). In the absence of C–PAM, PCC, 30% by mass on o.d. fibers, was added to the stirred fiber suspension, and sheets were then prepared after stirring the mixture of fibers and PCC for 10 sec in the MBF. In the presence of C–PAM, the same amount of PCC was added to the stirred fiber suspension, and the mixture was stirred for 10 sec, followed by the addition of C–PAM, 0.05% by mass on o.d. fibers, after which the mixture was again stirred for 20 sec. A sheet was then prepared and the solids content and filler content of the sheet (TAPPI T 211 om–

Table 1 Conditions in ultra–fine friction grinder

Variables	Conditions
Gap [μm]	240&250
Rotating speed [rpm]	1,500
Pulp consistency [%]	5
Grit class of the grinding stones	46 (grit size 297–420 μm)

93) were measured to evaluate the interaction of filler with fibers.

3. Results and discussion

3.1 Controlling fibrillation and its characteristics

Fig. 1 shows the development of the degree of internal fibrillation of fibers as measured by FSP with increased beating in the grinder. The FSP was found initially to increase and then reach a plateau for the two different gaps used. Fibers seem to continue to promote external fibrillation in the plateau region, caused by rubbing of the fibers against each other and against the stone surface during repeated recirculation of the pulp through the gap. In other words, internal fibrillation seems to be dominant at the beginning, shown in the figure as a solid line, while external fibrillation becomes dominant in the plateau region of the FSP level shown as a dotted line. This is supported by the increased degree of external fibrillation in the plateau region of the FSP, indicated as a dotted line. This is shown in **Fig. 2.** The amount of external fibrils increases only slightly up to passage 6, but increases clearly between passages 6 and 22, the region where mostly external fibrillation seems to occur. The smaller gap is better for external fibrillation than the larger gap.

A light microscope image of the development of external fibrillation is shown in **Photo. 1.** Minor changes in external fibrillation can be seen in the region where internal fibrillation is dominant, between unbeaten pulp and passage 6, while increased external fibrillation is seen in the region where external fibrillation is dominant, between passage 6 and passage 22. The additional external fibrils form a supplementary net-

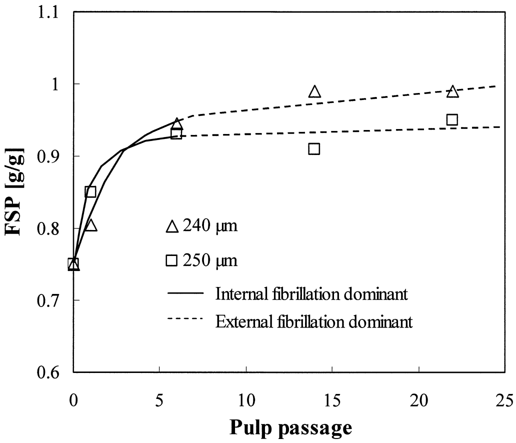

Fig. 1 Development of FSP (Pulp passage : Number of pulp recirculations through the gap in the grinder). R 100 fraction used for the FSP measurement.

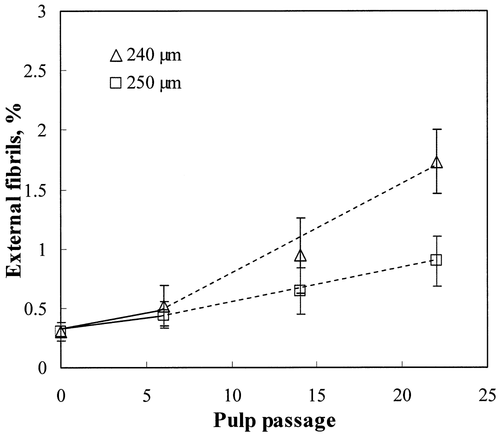

Fig. 2 Development of external fibrillation. R 100 fraction used for the measurement of external fibrillation.

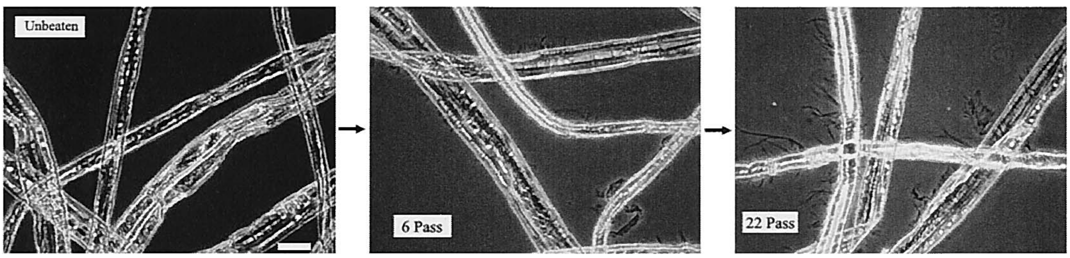

Photo. 1 Light microscope observation in phase contrast mode of external fibrillation of fibers. Pulp treated at 240 μm in ultra-fine friction grinder. Scale bar of 50 μm.

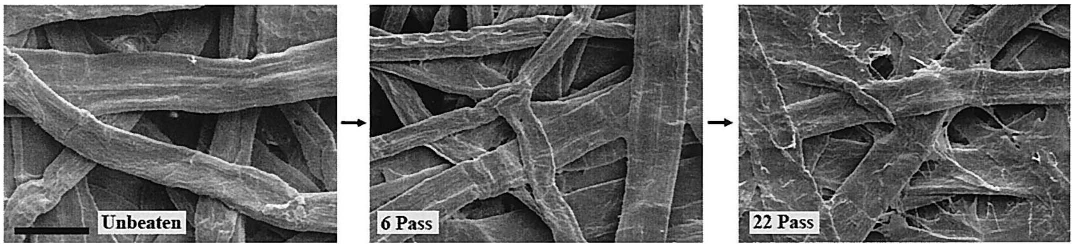

Photo. 2 SEM observation on the external fibrillation of a handsheet surface. Pulp treated at 240 μm in ultra-fine friction grinder. Scale bar of 50 μm.

Table 2 Changes in fiber properties. The R 100 fraction was used for measuring fiber length and curl while the whole pulp was used for measuring SR

	240 μm			250 μm		
Pulp pass	Curl (%)	Fibre length (mm)	SR	Curl (%)	Fibre length (mm)	SR
0	22.4	2.05	12	22.4	2.05	12
1	22.8	2.11	14	22.5	2.13	12
6	22.1	2.19	17	21.6	2.15	16
14	21.3	2.12	25	21.6	2.11	20
22	20.7	2.11	33	20.6	2.11	25

work shown as a film connecting between the fibers. This is shown in **Photo. 2.**

Table 2 shows the changes in fiber properties with increased beating in the grinder. As external fibrillation increases, fines generation also increases, resulting in increased SR. The narrower gap was found to increase the SR more than the wider gap. Insignificant changes in fiber length and curl were found with an increased number of pulp passages. The degree of external fibrillation (Fig. 2) increased more with the narrower gap than with the wider gap, revealing a direct relationship with fines generation in terms of the SR value.

3.2 Retention of filler

Fig. 3 shows the effect of fibrillation on the retention of filler in the absence and presence of C-PAM. Mechanical entrapment of filler in the absence of C-PAM is not influenced by the degree of internal or external fibrillation of fibers under high-vacuum dewatering, whereas colloidal interaction caused by C-PAM enhances filler retention with increased fibrillation of fibers. Filler retention is increased in the region where internal fibrillation is dominant, and it increases further in the region where external fibrillation is dominant. A higher degree of external fibrillation at the smaller gap was found to be better for retention of filler. Increasing only the degree of external fibrillation would allow increasing the filler retention, because a higher degree of external fibrillation increases the filler retention more. **Photo. 3** illustrates the important role of external fibrils for filler retention. More filler particles can be seen in the area of fibrils.

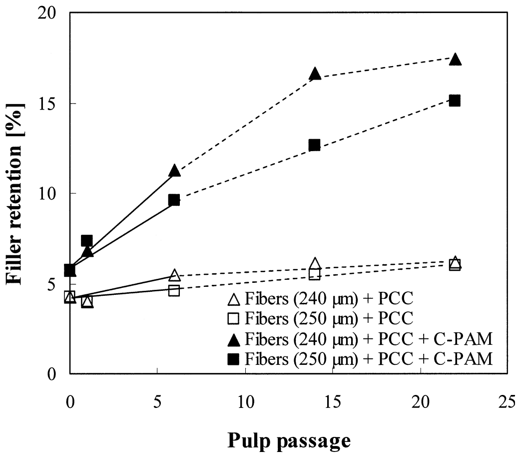

Fig. 3 Effect of fibrillation on the retention of filler.

When filler is added, both in the presence and absence of C-PAM, to fibers having a different degree of external fibrillation, the solids content of these sheets remains constant as shown in **Fig. 4,** although the retention of filler is increased with an increased the degree of external fibrillation. In other words, dewater-

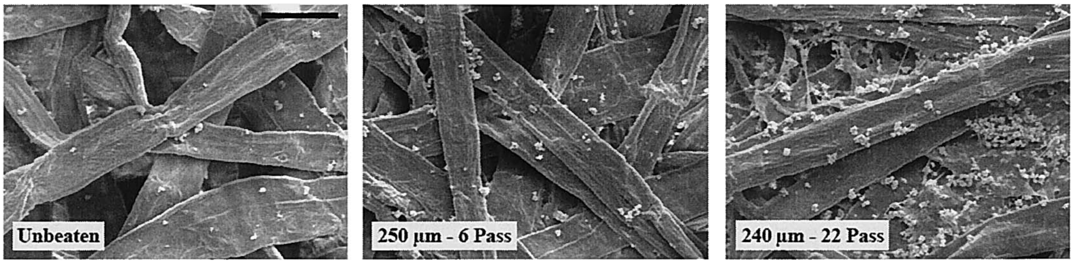

Photo. 3 SEM images of filler retained in the fiber network. 30% of filler addition. Scale bar of 50 μm.

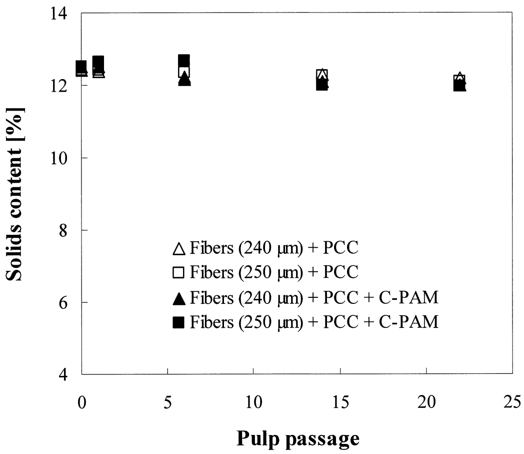

Fig. 4 Effect of fibrillation on solids content of wet sheet containing filler.

ing is not influenced either by fibers having a different degree of external fibrillation or by the amount of filler retained in the sheet.

4. Conclusions

In the experiments conducted in this study, external fibrillation was promoted from passage 6 to passage 22, while keeping internal fibrillation constant. This made it possible to evaluate the role of external fibrillation of fibers for the retention of filler during sheet forming in the MBF, which generates a high vacuum pulsation rate.

The mechanical entrapment of filler in the absence of chemical aid was not influenced by the degree of internal or external fibrillation of fibers under high-vacuum dewatering. However, the entrapment of filler was found to increase with increased internal fibrillation of fibers in the presence of chemical aid, and it can be further increased by promoting mostly external fibrillation of fibers. External fibrils still attached to the fibers therefore play an important role for the retention of filler.

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