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NOTE

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A method to determine lumen volume and collapse degree of pulp fibers by using bottleneck effect of mercury porosimetry

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Abstract On applying mercury porosimetry to wood blocks or paper sheets, the "bottleneck" effect due to pits of fibers occurs and thus lumen volume can be determined from the weight increase due to the remaining mercury. However, in addition to the mercury in the lumen, some mercury drops may also remain in the space between fibers within a paper sheet. The mercury between fibers increased with an increase of basis weight. Thus, a large number of paper sheets of low basis weight, such as $10\,g\cdot m^{-2}$, should be used to determine the lumen volume of pulp fibers. Furthermore, in the case of fibers from mechanical pulp with many open cut fibers, mercury can retract from the open lumen such that the bottleneck effect due to pits does not occur. Therefore, the degree to which fibers are cut should also be considered for lumen volume determination. Although quantitative estimation of open cut fibers is difficult, the percentage of open cut fibers is quite low for the long fiber fraction. Thus, the remaining mercury for the long fiber fraction can be adopted as the lumen volume at least for practical purposes. Compared with the original lumen volume of the wood, the volumetric degree of fiber collapse was also estimated. Plausible values of almost 100% for lightly beaten KP and about 85% for slabwood thermomechanical pulp were obtained for the degree of collapse.

Key words Bottleneck effect · Collapse · Lumen · Mercury porosimetry · Pulp fibers

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Introduction

Recently, lumen loading has again become noteworthy[1,2] as a novel paper-converting method. In this method, assurance of lumen volume is a key factor. Furthermore, fiber collapse, which is expressed as a decrease in cross-sectional area of lumen or in lumen volume, is very important to increase fiber–fiber bonding area,[3] relating to the mechanical properties of paper. Fiber collapse occurs during the pulping and sheet-making processes including beating.[3,4] The decrease of lumen volume has been estimated by image analysis of cross-sectional views of pulp sheets[5] and by a specific method developed by Page[3] using a pressure-sensitive adhesive tape. The method proposed in this study to determine lumen volume and degree of fiber collapse is an application of the bottleneck effect of mercury porosimetry, which was originally used to examine pore structure.[6,7]

In standard operation of a mercury porosimeter, a cumulative intruded mercury volume is measured by applying pressure from about 0.005 to 70 MPa. From the relation between pressure and the intruded mercury volume, the pore structure is examined. The pressure is finally returned to atmosphere pressure, 0.1 MPa from 70 MPa, and then the measurement is completed. The weight increase of the sample specimen during the measurement gives the volume of mercury remaining within the specimen.

For lumen pores within wood or paper, mercury in general penetrates through pits of fibers.[8] Once mercury has filled a lumen pore by applying high pressure, the mercury does not retract when the pressure is decreased to atmosphere pressure and remains in the lumen pore because the pits of the fibers act as "bottlenecks."[6] Thus, the weight increase of the wood or paper sample through porosimetry measurement should include the mercury remaining in the lumen pores. For wood samples, the pore volume is exactly the lumen pore volume. On the other hand, almost all pores in paper exist between fibers and the weight increase of paper samples includes the mercury remaining between fibers and the mercury in lumen pores that are originally derived from wood.[9]

In the case of kraft pulp (KP), fibers generally retain their original shape;[10] however, almost all of the lumen volume is lost before refining due to their softness.[7] Refining of KP fibers further raises their softness and may bring about more loss of lumen volume. On the other hand, mechanical pulp fibers are relatively rigid but are severely damaged. However, thermomechanical pulp (TMP) fibers are known to include more less-damaged fibers as their long fiber fraction.[10] In the damaged or open cut fibers, the bottleneck effect of pits does not work. Thus, the rigidity and damage of TMP fibers might be important factors for lumen volume determination.

In the present study, a series of handsheets with various basis weights from radiata pine TMP and lightly beaten KP, and radiata pine wood blocks were used as samples. The weight increase after mercury porosimetry measurement was examined to find appropriate conditions to determine lumen volume and degree of fiber collapse, compared with the original lumen volume in wood.

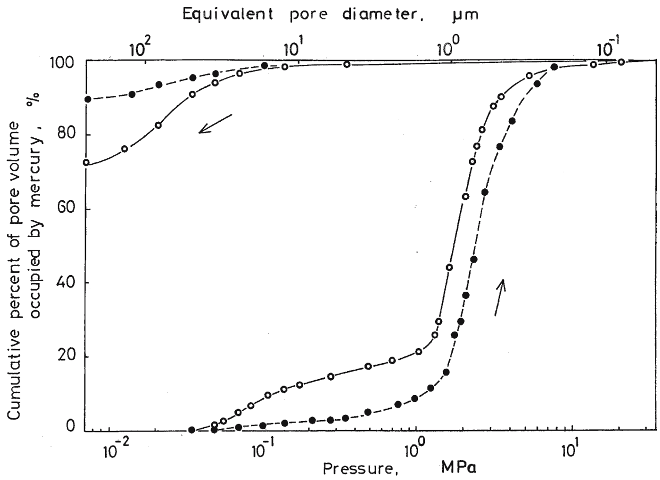

Fig. 1. Mercury penetration and retraction curves for wood blocks with and without adhesive tape sealing the transverse section. *Open circles*, without tape; *filled circles*, with tape

Experimental

Sample

Radiata pine slabwood TMP before and after classification to long fiber (passing 14 mesh and retained on 30 mesh), and short fiber fractions (passing 50 mesh and retained on 200 mesh) were the same as those investigated previously.[11] Bleached KP from radiata pine was supplied by the Pulp and Paper Research Organization of New Zealand (PAPRO-NZ) after lightly beating to CSF 550 ml. A series of the handsheets from TMP with various basis weights were prepared according to TAPPI T-205. Handsheets with very low basis weight (<10 $g \cdot m^{-2}$) were prepared using a 150-mesh fabric on the wire screen of the standard handsheet mold.

Wood (radiata pine) samples were prepared as the wood blocks [5 (T) × 10 mm (R) × 25 m (L)] and their transverse sections were sealed with an adhesive tape for some wood samples.

Instrumentation

Micromeritics 905 and 9305 porosimeters with pressure ranges of 0.0035 – 69 MPa were used. Measuring conditions of mercury porosimetry and the observation conditions of scanning electron microscopy were the same as those used previously.[9,12]

Results and discussion

Mercury intrusion and retraction from wood block (bottleneck effect)

The intrusion and retraction hysteresis curves throughout the pressure range from 0.0035 to 69 MPa for wood blocks with and without an adhesive tape seal on their transverse section are shown in Fig. 1. For the wood blocks without seals, some mercury intruded into the lumen pores of cut fibers facing the transverse section at a pressure of about 75 kPa, which corresponds to a lumen diameter of about 20 μm. The major intrusion occurred at about 1.5 MPa, corresponding to the pit pore diameter (ca. 1 μm). Mercury intrusion was complete at 10 MPa and all lumen pores were occupied by mercury. Furthermore, some mercury retracted on decreasing the pressure, and the retracted volume almost corresponded to the lumen volume of the open cut fibers facing the transverse section.

On the other hand, for sealed wood blocks, almost all of the mercury intrusion occurred at 1.5 MPa, corresponding to the pit pore diameter, and little mercury retracted on decreasing the pressure. These results suggest that the mercury intrusion volume at the maximum pressure could be the total lumen volume of intact fibers and the open cut fibers facing the transverse section. Furthermore, the intruded mercury volume at 75 kPa and then retracted at 25 kPa for the sealed wood block could be the lumen volume of open cut fibers in the wood block. This means that the majority of the intruded mercury, which is in the lumen volume of intact fibers of the wood block, remains in the sample after releasing to atmosphere pressure. These results provide a typical example of the bottleneck effect of mercury porosimetry,[6] as expected. As a result, the intact lumen volume can be estimated from the mercury volume, which is derived from the weight increase of the sample with the mercury after releasing to atmosphere pressure.

Effect of basis weight of paper specimen on lumen volume determination

The intrusion and retraction curves for paper differ from those of wood as described previously.[7,12] Almost all of the pores in paper are those between crossing fibers. Furthermore, the majority of the intruded mercury retracts from

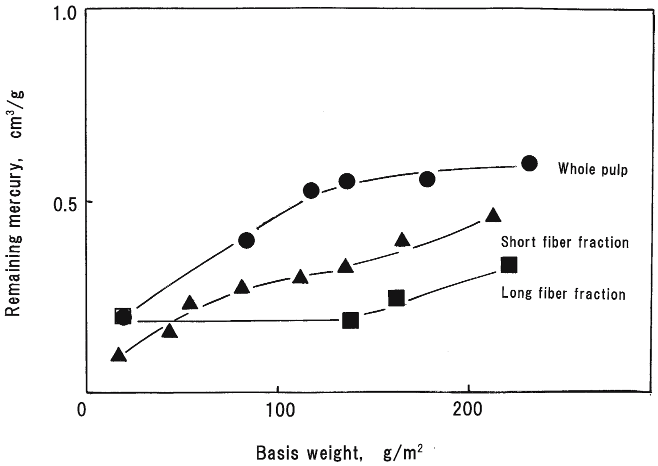

Fig. 2. Effect of basis weight on remaining mercury for the handsheets from radiata pine slabwood thermomechanical pulp (TMP)

the pores within paper on decreasing the pressure and the degree of mercury retraction decreases with an increase in the degree of beating. The mercury still remaining in paper after decreasing the pressure to atmosphere pressure has been interpreted to be that isolated and disconnected from the bulk continuum mercury between fibers of paper during the mercury retraction. A part of the mercury is that trapped in lumen pores by the bottleneck effect.[9] Therefore, the intruded mercury volume at the maximum pressure is much more than the lumen volume and the remaining mercury volume after releasing to atmosphere pressure, which was estimated from the weight increase, may not give the lumen volume. However, a decrease in basis weight of a paper sample may cause a decrease of the isolated mercury between fibers; that is, the remaining mercury estimated from weight increase may be restricted to the lumen pore volume. In the case of very low basis weight, mercury between fibers directly connects to surrounding mercury and the isolation of mercury during pressure decrease may not occur. Therefore, the remaining mercury at the lowest basis weight is preferable for lumen volume estimation of pulp fibers.

The effect of basis weight on the remaining mercury estimated from the weight increase is shown in Fig. 2 for the handsheets from the whole, long fiber fraction and short fiber fractions of slabwood TMP. The mercury remaining generally increased with an increase of basis weight as suggested previously, except for low basis weight handsheets from the long fiber fraction. Thus, a very low basis weight is essential for a paper sample to give a reasonable result for the lumen volume determination.

Lumen volume estimation and degree of collapse

According to the porosimetry procedure used previously,[7,12] one or three small specimens (1.3×2.7 cm square) of paper are loaded into the porosimeter cell. This procedure is suitable to avoid the formation of artificial pore volume[9] to investigate the pore structure of paper. However, this approach gives a small amount of mercury remaining, giving less accurate determination of lumen volume for paper with low basis weight. Therefore, loading of a large number of the specimens or folding of a long specimen into the cell is required to measure the remaining mercury accurately. The results of measurements of remaining mercury for a number of specimens with very low basis weight are shown in Table 1. Furthermore, the intruded mercury volume at the maximum pressure for the wood block was also shown rather than the remaining mercury volume because the former corresponds to whole lumen volume and the latter is the volume of the intact lumen. The whole lumen volume of a wood block can be roughly calculated to be $1.55\,cm^3 \cdot g^{-1}$, assuming that the density of wood and the fiber wall are 0.45 and $1.5\,g \cdot cm^{-3}$, respectively. The experimental value of $1.44\,cm^3 \cdot g^{-1}$ is fairly close to the calculated value. On the other hand, almost no remaining mercury for KP and some remaining mercury for TMP were observed. The difference of the remaining mercury for fractioned TMP could arise from the difference of inclusion ratio of open cut fibers, because the bottleneck effect may not work with open cut fibers. The long fiber fraction seemingly consists of intact fibers as shown in Fig. 3, whereas whole pulp and the short fiber fraction include many open cut fibers. Furthermore, the whole pulp also includes a fines fraction. Thus, the remaining mercury in whole pulp or in the short fiber fraction is less than that of the long fiber fraction. Therefore, the value of the long fiber fraction, $0.19\,cm^3 \cdot g^{-1}$, should be adapted for the estimation of lumen volume of TMP fibers, at least for practical purposes such as lumen loading. Given that a significant amount of the remaining mercury (about 45% of that for the long fiber fraction) was observed for the short fiber fraction, the precise estimation of lumen volume is difficult. Thus, the inclusion percentage of open cut fibers that provide no bottleneck effect is difficult to estimate quantitatively, even using image analysis. The scanning electron micrograph (shown in Fig. 3) suggests the percentage for the long fiber fraction is very low and is far less than 25%. Assumed to be 25%, the lumen volume can be calculated to be $0.25\,cm^3 \cdot g^{-1}$ as the remaining mercury volume/0.75 for TMP. Thus, the precise lumen volume of TMP fibers could be between 0.19 and $0.25\,cm^3 \cdot g^{-1}$. Defined as the volume ratio of the fiber lumen to the original lumen in wood, the degree of volumetric collapse of pulp fibers is nearly 100% for the lightly beaten KP fibers and could be between 83% and 87% for TMP fibers. These estimates of the degree of collapse seem to be reasonable, considering the cross-sectional view of the handsheets from these pulps[9–12] as shown in Fig. 3.

Table 1. Mercury volume as remaining mercury in test samples

Sample	Remaining mercury ($cm^3 \cdot g^{-1}$)
Thermomechanical pulp (slabwood)	
Whole	0.18
Long-fiber fraction	0.19
Short-fiber fraction	0.08
Kraft pulp	0.01
Wood	1.44

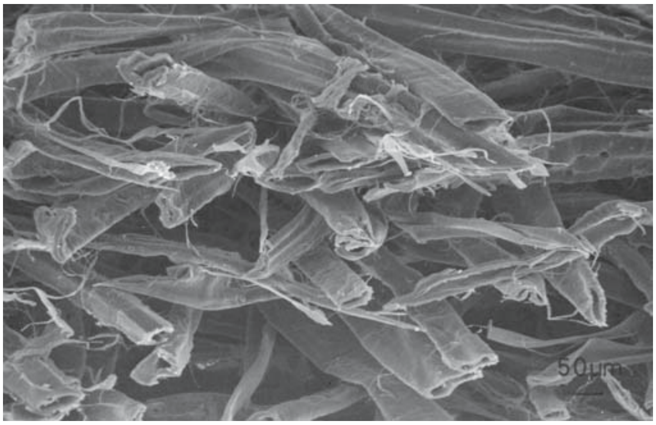

Fig. 3. Cross-sectional view of a handsheet from long fiber fraction of radiata pine slabwood TMP

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