# Removal of water from pulps by pressing

## Part 1: Inter- and intra-wall water

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*ABSTRACT: Water removal from pads of wet pulp under pressure was investigated. Water pressed from the pad was quantitatively separated into two fractions—that expressed from the porous structure of the cell wall, and that expressed from spaces between fibers. Water from both locations starts to be expressed from the pad at low pressure. However, almost all of the water between fibers (and in the lumen) is removed at pressures below 2 MPa, leaving the water in the fiber walls as the predominant form of moisture remaining at higher pressures. The water content of pulp pads pressed to high pressure was correlated linearly with the fiber-saturation point (FSP), the initial level of moisture in the swollen fibers. Thus FSP is a measure of pulp pressability.*

*KEYWORDS: Bibliographies, cell walls, fiber saturation point, fibers, pressing, pressure, pulps, water removal.*

In his classic paper on the physics of water removal, Campbell stated that "the art of papermaking consists of the manipulation of a water suspension of fibers so that the water is removed in such an adroit fashion that those fibers are left, water-free, in a layer or sheet as intended by the artist" *(1)*. While equating the whole art of papermaking with water removal is perhaps a poetic exaggeration, water removal does require three successive and major processes. On the paper-machine wire, the bulk of the water is removed by drainage. In the press section, mechanical forces are used to induce a further portion of the water to flow out of the sheet. Finally, in the dryer section, heat is applied to evaporate the residual water.

Removal of water from a paper web by pressing is considerably less energy intensive than by evaporative drying *(2–4)*. Obviously, it is desirable to remove as much water as possible before a sheet leaves the press section. However, optimization of the pressing process will require a better understanding of the location and mechanisms of water retention in the fibrous sheet *(5, 6)*.

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In a saturated web of fibers, water is located in the interstices between fibers, in the fiber lumens, and in the porous structure of the fiber wall. One intuitively understands that, on pressing a web, the first water to be displaced from the web is the portion that is squeezed from the spaces between the fibers. However, it is not obvious at what stage, if any, water is forced from the other locations and to what extent this occurs. The earliest literature on the matter refers to the water removed by commercial presses as flowing exclusively from the spaces formed by the network of fibers *(1, 7, 8)*. More recent works suggest that some water is also expressed from the fiber walls *(9–11)*. However, this concept is qualitative and lacking in direct experimental data.

The evolution of thought on water removal during pressing has followed developments in efforts to estimate the amount of water in the fiber walls, a quantity that has proved to be elusive.

In 1906, the term "fiber-saturation point" was introduced to describe the moisture content within the cell walls of wood *(12)*. Up to the middle 1960s, the fiber-saturation point (FSP) of all wood and other cellulosic fibers was believed to share a common value of about 0.3 g water/g dry fiber *(13)*. With the presses of the day, pressed sheets retained between 0.7 and 1.0 g water/g dry fiber *(10)*. Thus the water contained within the fiber walls appeared to be a minor fraction of the water retained in a pressed sheet. This information, coupled with the concept

that the capillaries inside the fiber wall are finer than those between the fibers, was probably the reason to believe that the fiber-wall water was strongly held and undisturbed during pressing *(1, 6)*.

In the mid-1960s, it was shown that the previously accepted value of the FSP was too low, particularly in the case of wood pulps *(14)*. The FSP for all commercial pulps exceeds 0.8 g water/g dry fiber and can be as high as 2.0 g/g *(15, 16)*. With these values, it was eventually recognized that "at the end of the pressing cycle, the amount of water held by the sheet . . . is in most commercial presses below that . . . originally held by the fiber wall. In the majority of cases, therefore, water is pressed out of the fiber wall during pressing operations" *(5)*.

The technique used to measure FSP is relevant to the discussion presented in this report. FSP is determined by first adding a polymer solution to a sample of moist pulp. The polymer must have a molecular diameter greater than the largest pore openings in the fiber walls. Water associated with the fibers in excess of the FSP dilutes the polymer solution, whereas water within the fiber walls does not. The FSP is calculated from the weights of materials involved and the change in concentration of the polymer solution *(14)*. The most frequently used polymer is dextran, with a molecular weight of $2 \times 10^6$. This polymer has the necessary property of not being adsorbed by cellulose surfaces *(17)*, and its high optical rotation provides a means of determining the concentration of its solutions with accuracy.

In 1977, Carlsson and co-workers utilized the properties of dextran to demonstrate unambiguously that the effluent expressed from a pad of pulp fibers actually contained some water from within the fiber walls *(18)*. They formed the pad from a suspension of fibers in a solution of dextran. Effectively, they had used the polymer to "label" the water external to the wall, i.e., that in the lumens and interfiber spaces, while leaving the water inside the fiber walls free of polymer. The pad was then placed in a static press, and the concentrations of dextran in successive effluents were measured. At a certain stage of pressing, the concentration of dextran in the effluents fell rapidly below that of the original interfiber solution. The solution of dextran had been diluted by "pure" water, the origin of which could only be the fiber walls.

In the current work, we use essentially the same experimental procedure as Carlsson, but we extend the scope of the procedure by introducing a simple calculation. Using this calculation, we subdivide the effluents from the pressed pad into the fractions coming from the intra- and inter-wall locations. These amounts are then used to back-calculate how the distribution of water between the two locations changes over a range of applied pressure. This technique for examining the sequence of water removal is applied to a wide variety of pulps in an effort to better our understanding of pressability.

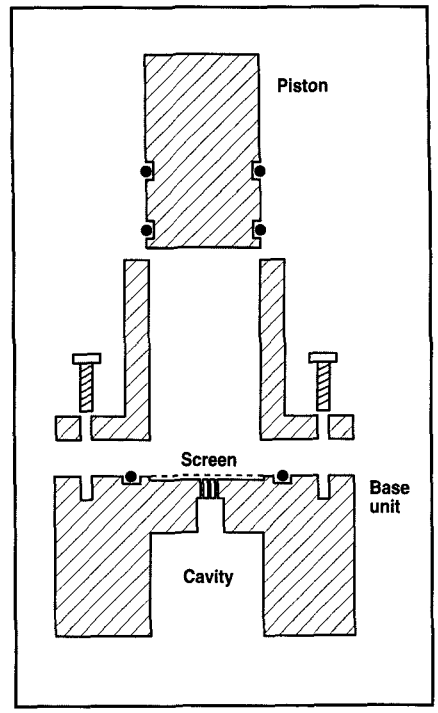
## Experimental

### Pulps

A series of kraft pulps was prepared from pin chips of black spruce. Yields ranging from 45% to 90% were obtained by varying the time of cooking. The characteristics of these pulps were tabulated in a previous work *(19)*. For the present work, these pulps were supplemented with a series of acid sulfite pulps of a similarly wide yield range and made from the same source of chips. A 100% yield sample was obtained by fiberizing the pin chips in a Waring blender.

Fines passing through a 100-mesh screen were removed from all pulps using a Bauer–McNett classifier. Since the properties of pulps can be influenced by the nature of the counterions of the acidic groups, all the results reported in this article were obtained after ion-exchange of the fines-free pulps to sodium form as a reference condition *(19)*. All pulps were kept and tested in the never-dried state unless otherwise mentioned.

1. Cross-sectional view of the pressing device




### Apparatus

**Figure 1** shows the stainless-steel pressing device designed for the water-removal experiments. The diameter of the cylindrical piston is 5 cm, and the pressing chamber has a volumetric capacity of 100 mL. The pad was formed on a 100-mesh wire screen that covers the five, centrally located, 1-mm-wide drainage holes in the base unit. Effluent from the pad was collected in weighing jars that fitted into the cavity of the base unit. For the application of pressure, the assembled device was placed beneath the crossarm of an Instron (Model 1137) operating in a compression mode. A load cell (Series 2512-134) capable of applying loads up to 15,000 kg was used.

### Pressing experiments

To start an experiment, ca. 25 g of wet pulp (oven-dry weight 4 g) and 100 mL of a 2% solution of dextran (mol. wt. $2 \times 10^6$, Pharmacia Ltd., Sweden) were weighed into a bottle and mixed by

I. Dewatering data for a pad of nylon fibers

| Press load, MPa | Optical rotation, degrees | Total volume of effluent, mL | Volume of effluent from fiber walls, mL |
|---|---|---|---|
| 0.00 | 3.948 | 90.38 | 0 |
| 0.06 | 3.954 | 12.12 | 0 |
| 0.15 | 3.957 | 1.68 | 0 |
| 0.40 | 3.952 | 1.67 | 0 |
| 0.70 | 3.935 | 0.84 | 0 |
| 1.0 | 3.991 | 0.55 | 0 |
| 2.0 | 3.950 | 0.97 | 0 |
| 14.3 | 3.934 | 1.98 | 0 |
| 26.2 | 3.969 | 0.31 | 0 |
| 41.8 | 3.848 | 0.20 | 0 |
| 53.8 | 4.106 | 0.10 | 0 |

II. Dewatering data for a pad of kraft fibers*

| Press load, MPa | Optical rotation, degrees | Total volume of effluent, mL | Volume of effluent from fiber walls, mL |
|---|---|---|---|
| 0.00 | 3.656 | 66.35 | 0 |
| 0.01 | 3.652 | 28.55 | 0.03 |
| 0.16 | 3.607 | 19.54 | 0.26 |
| 0.31 | 3.276 | 2.12 | 0.22 |
| 0.61 | 2.914 | 1.60 | 0.33 |
| 0.91 | 2.436 | 0.67 | 0.22 |
| 1.98 | 2.403 | 1.04 | 0.64 |
| 14.2 | 0.287 | 1.17 | 1.08 |
| 26.2 | 0.204 | 0.25 | 0.23 |
| 41.7 | 0.000 | 0.17 | 0.17 |
| 53.4 | 0.000 | 0.09 | 0.09 |

*Pulp yield =69%

shaking for about 45 min. The pressing device minus the piston was assembled, and the suspension was drained through the screen. The initial filtrate was collected and its optical rotation measured for the later calculation of the initial water content in the fiber wall (i.e., the FSP).

With the piston in place, the pressing device was then inserted into the Instron. In ten steps, the pressure on the piston was successively increased to a maximum of 10,800 kg. Each pressure step was maintained for 15 min, during which time the flow of water ceased. Low compression velocities were used to avoid generating any large hydrostatic pressure in the pad. The liquid expressed at each load was collected in a separate jar. The liquids were weighed, and their optical rotations measured using a polarimeter reading to 0.001 degrees. Where the effluent was small in volume, it was quantitatively diluted to produce sufficient volume to fill the polarimeter cell. At the end of the experiment, the pad was removed from the pressing device and weighed in both wet and dry conditions.

## Calculations

### Total water content of pad

The curve for the total water content of the pad as a function of pressure is readily constructed. The weights of the effluents (per unit weight of dry fiber) are merely added in a cumulative manner to the final moisture content of the pressed pad.

### Water content of fiber walls

Measurements made mainly during pad preparation provide all the information necessary to calculate the initial water content of the fibers (FSP) in the usual way *(14)*:

$$\delta_0 = [(w+q)/p]\ \{1 - [w/(w+q)]\ (r_s/r_0)\} \quad (1)$$

where

$\delta_0$ = initial water content of fiber walls

$p$ = dry weight of pulp sample

$q$ = weight of water in pulp sample

$w$ = weight of dextran solution added to pulp sample

$r_s$ = optical rotation of stock solution of dextran

$r_0$ = optical rotation of initial filtrate collected during pad formation.

To construct a plot of fiber-wall water content as a function of pressure, the amount of water expressed from the fiber wall at each increment of pressure must be known. Since the initial filtrate consists solely of liquid from the lumens and the spaces between fibers, its optical rotation is that of the liquid in these locations. Subsequent effluents are effectively a mixture of two components—interwall water, with a rotation $r_0$, and intrawall water, with zero rotation. Thus, an effluent of rotation $r$ is considered to consist of a fraction of interwall water $(r/r_0)$ and a fraction of intrawall water $[1-(r/r_0)]$. With these assumptions, the drop in the water content of the fiber walls, $\Delta\delta$, at each increase in pressure is

$$\Delta\delta = f[1 - (r/r_0)]/p \quad (2)$$

where

$f$ = weight of effluent.

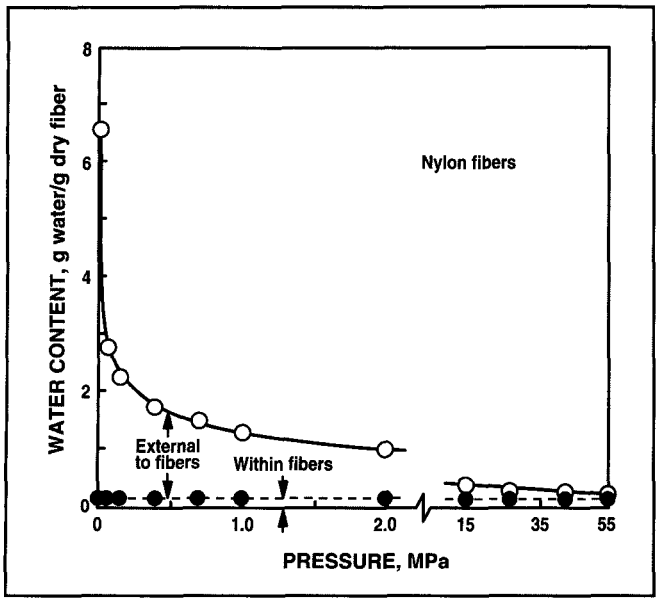
Hence the water content ($\delta$) of the fiber walls at a given pressure is obtained from the initial fiber-saturation point less the cumulative amount of water squeezed from the fiber walls.

$$\delta = \delta_0 - \Sigma\Delta\delta \quad (3)$$

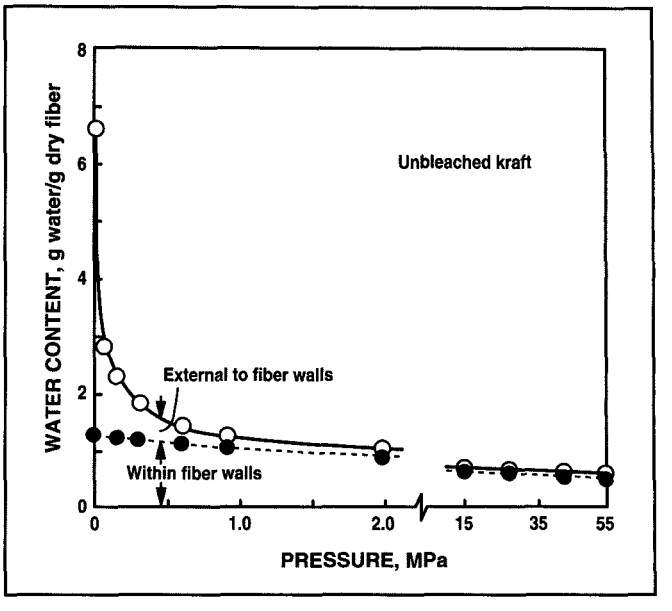
## Results and discussion

In a typical experiment, a series of pressures is applied to a pad prepared from a suspension of pulp fibers in a dextran solution. The effluent expressed at each pressure is collected individually and analyzed for dextran. The concentration of dextran is compared with the concentration found in the filtrate collected during the formation of a pad, and the fraction of water originating from the wall is then calculated.

**2.** Effect of applied load on the water retained in a pad of nylon fibers. Retained water is depicted in two fractions: within the fiber walls and external to the fiber walls.




**3.** Effect of applied load on the water retained in a pad of kraft fibers. Retained water is depicted in two fractions: within the fiber walls and external to the fiber walls.




This is the same calculation that is commonly used for the analytical dilution of a "stock" solution. In our case, the "diluent" is the water expressed from the fiber walls, and the "stock" solution is the solution of dextran expressed from the spaces between the fibers. However, implicit in this interpretation is that the effluent contains all the water expressed from the fiber walls. It is possible that some water expressed from the walls enters interfiber spaces and is not carried out of the pad into an effluent. If this occurs, our calculation would overestimate the amount of water retained in the fiber walls. With this proviso, we report our findings on the changing composition of a fibrous pad with pressure. As will be seen, the calculation provides sensible results for both pulp and model fibers.

### Effluent analysis

In preliminary experiments, we tested our procedure with pads prepared from suspensions of fibers of known characteristics. For example, **Table I** gives the raw data obtained from a pressing experiment using nylon fibers. All of the effluents have essentially the same high optical rotation, indicating that water is not expressed from the fibers. This is in line with the nonporous character of these fibers. It follows that all the water pressed from the pad comes from the spaces between the fibers.

**Table II** gives the data for a pad prepared from a typical kraft pulp. The initial effluent (0.01 MPa) was the only one with the same optical rotation (and thus the same concentration of polymer) as the filtrate collected during pad formation. This suggests that the application of this first load must have redistributed the fibers to form a more compact pad without compressing the fibers. Subsequent effluents have lower and lower optical rotations and, hence, must contain progressively larger fractions of water from the fiber walls. At high loads, a small amount of liquid without an optical rotation is expressed. This water comes exclusively from the fiber walls. Thus, in contrast to nylon fibers, pulp fibers appear to contain considerable amounts of water within the fiber walls. Some of this water is expelled progressively as press loads are increased.

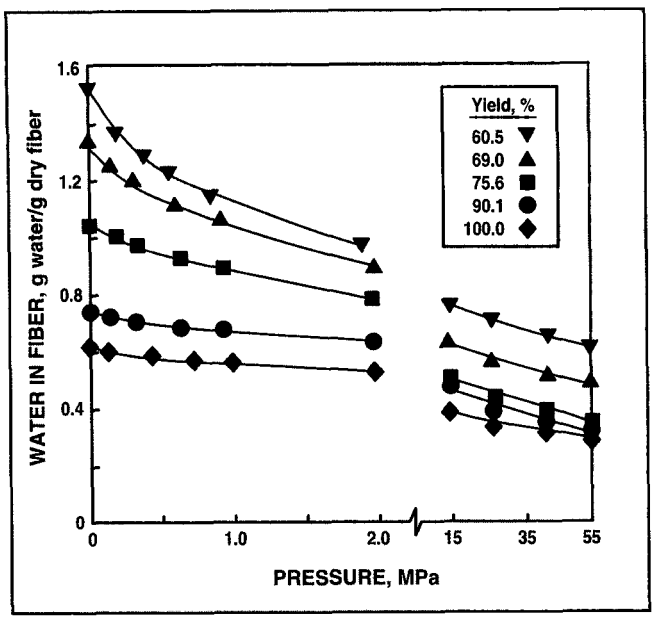
### Retention of water in fibrous pads

At each incremental pressure, the effluent volume is measured and the fraction of fiber-wall water is calculated. Using this information, we can plot the equilibrium water content within the fibrous pad as a whole and within the constituent fiber walls as a function of applied load. Typical plots for pads of nylon fibers and pulp fibers are shown in **Figs. 2 and 3**, respectively.

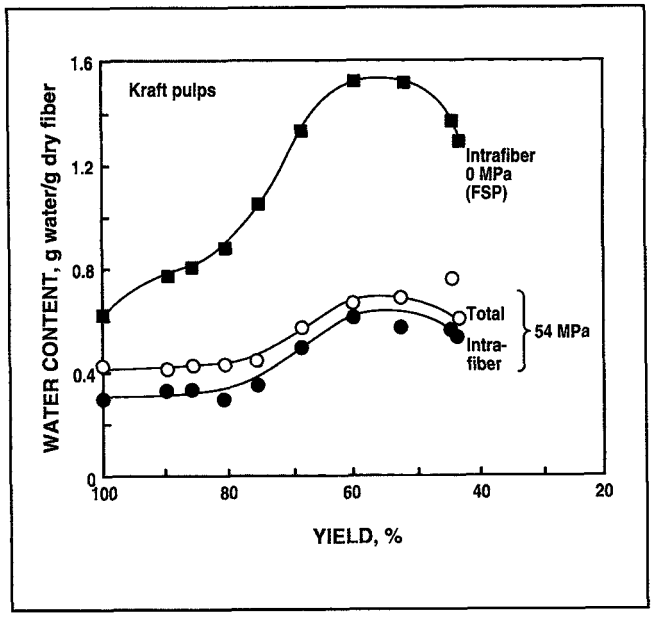
The dewatering curves for the pad of nylon fibers are shown in Fig. 2. Initially, a large amount of water is retained between the fibers, but this amount decreases to almost zero at the highest pressures. A very small and constant amount of water, namely 0.1 g/g, is retained in the fibers throughout the pressure range. This amount agrees with the reported moisture content of nylon fibers *(20)*. Thus our experimental procedure gives results that are consistent with the nature of the fibers in this pad. These are rigid fibers with a low moisture absorptivity, and the water removed from the pad is thus exclusively from spaces between fibers.

The location of the water retained in a pad prepared from a suspension of pulp fibers is rather different, as seen in Fig. 3. In this pad, a significant

4. Amount of water within fiber walls as a function of pressure for kraft pulps of different yield




5. Amount of water within fiber walls as a function of yield for kraft pulps at zero pressure (upper curve) and at 54 MPa (lower curve). The middle curve depicts the total water within the pulp pad at 54 MPa.




amount of water is retained in the fiber walls as well as in the spaces between fibers. Both locations show water loss at the lowest compressive load. However, the volume of water removed from the interfiber spaces is much larger than that expressed from the fiber walls. Following the initial dewatering, further removal of water becomes increasingly difficult. In fact, most of the water between the fibers and in the lumens is removed at low pressures, and then the curve for the pad approaches and finally parallels that for the fiber wall. At high pressures, therefore, flow of water from spaces exterior to the fiber walls ceases, and the water expressed from the pad comes exclusively from the walls.

The similar shape of the dewatering curves for the pad as a whole and for the fiber walls shows that the process of dewatering during wet pressing does not occur in distinct and separate phases, i.e., dewatering of the interfiber pores and then the fiber walls. Rather, wet pressing simultaneously compresses both the network structures of the pad and of the fiber wall, with both structures becoming less permeable and drier. This gives credence to a model for the removal of water as a flow through a rather arbitrary "structure" of decreasing permeability and decreasing water content *(21)*. Carlsson and co-workers also deduced that water permeates from a larger pore system and from systems of smaller pores without a sudden transition *(22)*.

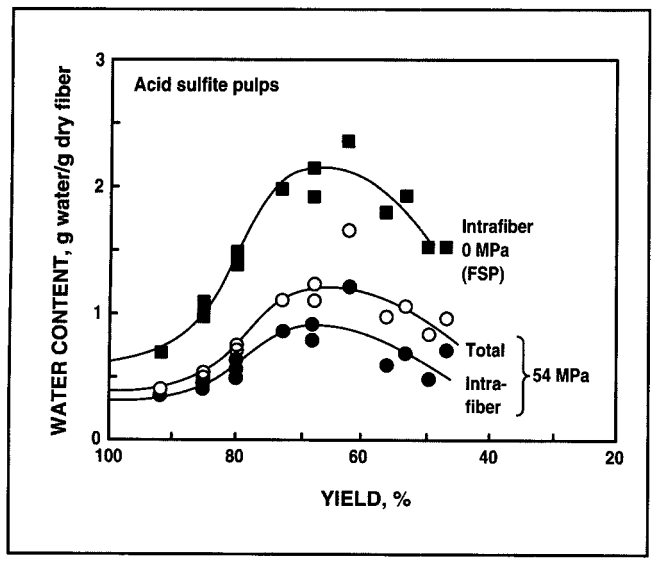
It is interesting that the pad always appears to contain some interfiber water and that, for pressures exceeding 2.0 MPa (300 psi), the amount is fairly constant. We considered the possibility that this water was an artifact resulting from ultrafiltration of large dextran molecules from the solution. However, this is certainly a minor effect, since a pressed pad contains less than 0.5% of dextran in the initial suspension of fibers. Carlsson also considered and dismissed the possibility of ultrafiltration of dextran *(18)*. The basis for this was that even with a load of 50 MPa applied onto a pad of viscose fibers, a solution of high-molecular-weight dextran could still be flushed through the pad. That the fibers do not perfectly conform under high pressure does not seem unreasonable. Claesson and Andersson have reported that the interstices between fibers in a dry sheet were not completely eliminated even at a pressure of 1550 MPa (15,000 atm) *(23)*.

## Evaluation of pulp pressability

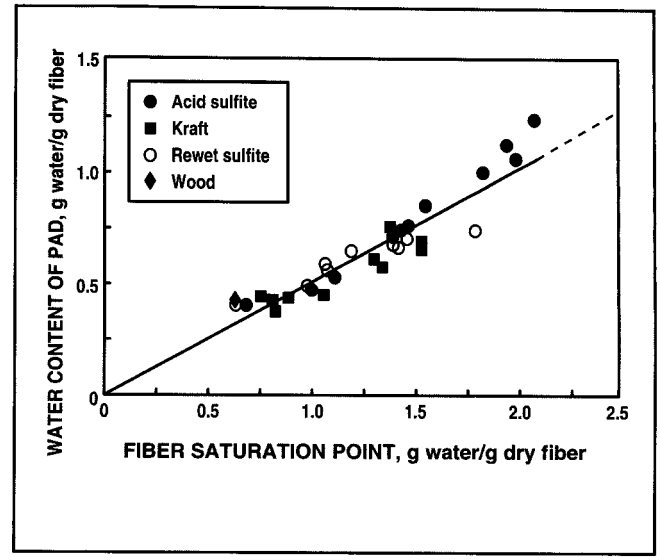
A long-sought goal of papermakers is the ability to predict the extent of water removal from a web by pressing. In this respect, the nature of the furnish is an important but often neglected variable *(24–26)*. We examined the dewatering curves for a wide variety of pulps in an effort to identify patterns of dewatering behavior.

Figure 3 shows that the water retained in a pad pressed to beyond 2.0 MPa (300 psi) is predominantly within the fiber walls, indicating that it is this water rather than the interfiber water that limits dewatering. **Figure 4** shows the variation in fiber-wall water content as a function of pressure for several different pulps. A feature brought out by examining any such family of curves is that the dewatering curves maintain the same order throughout the entire range of pressures. This order is determined by the initial level of swelling of water-swollen fibers, i.e., by the initial fiber-saturation point. Combining these observations, it ap-

6. Amount of water within fiber walls as a function of yield for acid sulfite pulps at zero pressure (upper curve) and at 54 MPa (lower curve). The middle curve depicts the total water within the pulp pad at 54 MPa.




7. Correlation of pulp FSP and pad water content at 54 MPa. The regression line is a best fit with a correlation coefficient of 0.94. (All pulps were never dried, with the exception of the rewet sulfite pulp.)




pears that the FSP is a furnish parameter that might be used to characterize the ease of water removal from a given pulp.

This concept was tested with data taken from the pressing curves of the whole series of kraft and sulfite pulps. **Figures 5 and 6** show the changes in water content as a function of yield for kraft and sulfite pulps, respectively. The upper curves in Figs. 5 and 6 show the variation of FSP with yield. Similar plots have been derived previously, and the shapes of the curves have been discussed *(15, 27)*.

For the kraft pulps (Fig. 5), there is a large increase in FSP upon pulping to 65% yield. This large increase in the amount of water within the cell wall apparently is not due to a physical expansion of the wall. Calculations showed that the increase in water content corresponded to the amount of solid material removed by pulping. As long as the removed solids were being replaced by an equal volume of water, the volume of the cell wall remained constant. Below 65% yield, both the FSP and the calculated volume of the wall decreased. We considered this to indicate that, following material removal in this range, the wet fiber wall actually contracted. More recently, the contraction has been attributed to the formation of hydrogen bonds between adjacent microfibrils in the wall *(19)*.

The same mechanisms were thought to operate in the case of sulfite pulping (Fig. 6), but it is likely that some additional effect contributed to the much sharper peak in the plot of FSP vs. yield. The peak may be the result of osmotic swelling caused by the introduction of hydrophilic sulfonic acid groups onto the lignin *(15)*. The concentration of these groups is highest in the middle yield range *(28, 29)*, which is where the FSP peaks.

The two lower curves in each of Figs. 5 and 6 show the water contents of the pressed pads and of their constituent fibers at different yields. At all yields, most of the water content of the pressed pads consists of water within the fibers. Further, it is apparent that the shapes of the plots after pressing are very similar to those before pressing, emphasizing the influence of FSP on pressing. In fact, the curves are more than similar. **Figure 7** shows that the water remaining in the pressed pad is a constant fraction of FSP. The proportionality shown in this figure was obtained from measurements at the highest pressure. However, comparable linear relationships passing through the origin exist at lower pressures. The pressure must, however, be high enough to collapse the fibers and remove most of the water between the fibers.

Why this proportionality is obtained is not at all clear, especially in view of the fact that some of the pores holding the water existed in wood, while other pores were created during pulping by the variety of mechanisms described previously. Nevertheless, the finding that a pulp's FSP (measured at atmospheric pressure) characterizes its pressability has practical value.

Our laboratory study was carried out with pads of high grammage using a static press and a residence time permitting equilibrium to be attained. These conditions are very different from those used in commercial presses. However, Busker and Cronin have examined water removal from sheets using the more practical conditions of a two-roll press standardized to a realistic load, speed, and sheet grammage. When a variety of pulps was evaluated under their pressing conditions, a correlation was found between the dryness of the sheets exiting from the press and the water-retention values of the pulps *(26)*. Comparable experiments were also carried out by Pikulik

*(30)*, and if his data gathered on different types of pulp are examined collectively, a common relationship between pressed moisture content and water-retention value also is found. Thus, since the water-retention value approximately corresponds to the FSP *(31)*, it appears that the relationship that we have observed under static pressing conditions also applies to dynamic conditions.

## Concluding remarks

The process of water removal from wet pulps was investigated using a novel method of analysis. The method provides a means of classifying the effluent from the pressed pulp into two components—that expressed from the fiber walls and that expressed from the spaces between the fibers and from the lumens.

The location of water in pressed pads has been a subject of considerable speculation in the literature. The quantitative data obtained in this study show that all pulp pads dewater in a similar fashion, with the water within the cell wall controlling and limiting the extent of dewatering in a press. If there were no water inside the fibers, paper could be rendered almost completely dry in the press section, and the size of the dryer section could be greatly reduced. However, there is a considerable amount of water within the fiber walls. The difficulty of dewatering a pulp varies with the amount of water within the fiber walls, which in turn varies with pulping process and yield.

In Part 2 of this report, we will examine in more detail the dewatering curves of fiber walls in an attempt to gain insight into the actual mechanism by which fibers resist water removal. ■

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