

Equilibrium moisture content in wet pressing of paper

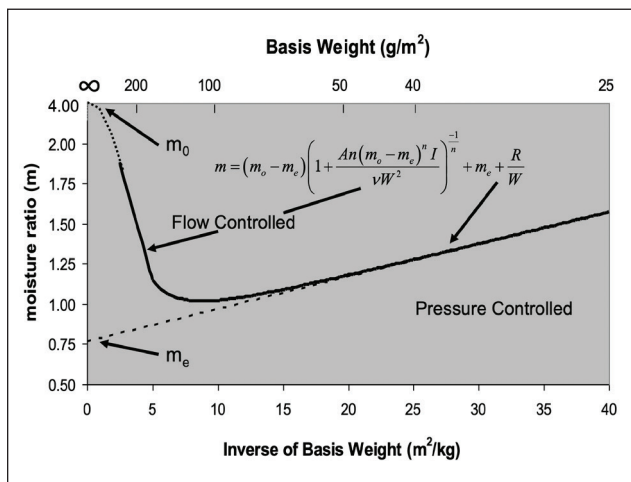
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ABSTRACT: Equilibrium moisture is a limiting factor in achieving high solids in the later stages of pressing or pressing low basis weight grades. We have developed a model that relates equilibrium moisture directly to the pore size distribution of fibers as measured by the solute exclusion technique. The model shows that chemical pulping and refining increase equilibrium moisture by increasing pore volume at given pore sizes in fibers, which leads to lower pressed solids and greater energy expenditure in the dryer section. Means to increase equilibrium moisture without compromising pulp strength are briefly discussed.

Application: The ultimate dryness possible in wet pressing is related to the structure of the fiber wall. Understanding how to modify this structure to improve water removal without affecting pulp strength is the key to higher pressed dryness.

In earlier work, we developed a model of wet pressing called the Decreasing Permeability Model (DPM) [1]. The key premise of this model is that the applied pressure on a wet web acts on all the water between and within fibers. At the moisture levels typical of pressing, fiber walls are too malleable to provide structural support.

Water removal in pressing can be qualitatively divided into two regimes: flow-controlled and pressure-controlled (Fig. 1) [2,3]. In the flow-controlled regime, water is primarily removed from the spaces between fibers. As water removal proceeds, the remaining water flows through spaces of ever decreasing permeability. This is represented



1. Water removal in wet pressing can be qualitatively divided into two regimes. The first term of the Decreasing Permeability Model (DPM) dominates the flow-controlled regime, while the equilibrium moisture, m_e , and rewet, R , dominate the pressure-controlled regime. W is the basis weight, I is the press impulse (pressure $\times$ time), and v is the kinematic viscosity of water. Specific permeability, A , and compressibility factor, n , are furnish specific coefficients [1-3].

mathematically by the first term in the DP model equation (Fig. 1). In the pressure-controlled regime, water is largely expressed from the walls of the fibers. This is represented by the second term in the equation, the equilibrium moisture, m_e , which is governed by a different mechanism and is the subject of this paper. Equilibrium is reached when moisture no longer changes with time but can be lowered if the applied pressure is increased. The third term is rewet, R , which is water that has been pressed from the paper into the felt but returns to the paper as pressure is released and by film splitting when the paper separates from the felt [4,5].

For lightweight or heavily pressed papers, the moisture ratio after pressing depends on only the equilibrium moisture and the amount of rewet [6]. Equilibrium moisture represents the lowest possible moisture content when pressing at a given pressure over infinite time. This is the limit in pressing and hence understanding the factors that control it is important.

BACKGROUND

Measurement of equilibrium moisture

A common way of determining equilibrium moisture is by platen pressing over a long period of time at different pressures. Such studies [1] have shown that the relationship between equilibrium moisture m_e and pressure p , (MPa) has the form:

$$m_e = cp^{-a} \quad (1)$$

Values of parameters c and a from several studies are summarized in **Table I**.

Parameters c and a have physical significance. Parameter c is the equilibrium moisture at pressure 1 MPa and is therefore a useful basis for comparing equilibrium moisture

c	a	Pulp Type	Comments	Source
1.36	0.17	Unbleached kraft	69% yield	Laivins and Scallan [7]
0.915	0.22	Kraft and mechanical	Mixed	Holstege [8]
0.881	0.26	Unbleached softwood kraft	Unbeaten	Maloney et al [9]
0.946	0.33	Unbleached softwood kraft	Beaten	Maloney et al [9]
2.14	0.23	Bleached kraft 470 CSF	163 g/m ²	Busker and Cronin [10]
1.55	0.17	Bleached kraft 470 CSF	60 g/m ²	Busker and Cronin [10]
0.632	0.20	Thermomechanical		Maloney et al. [9]

I. Equilibrium moisture based on data from the literature [1].

content at a common pressure. Parameter *a* is an indicator of the responsiveness of equilibrium moisture to pressure. This is illustrated by taking the first derivative of Eq. 1 and substituting to obtain:

$$\frac{dm_e}{m_e} = -a \frac{dp}{p}$$

(2)

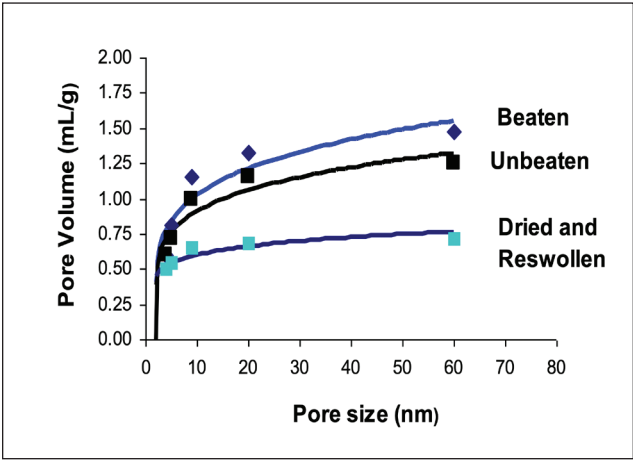
This shows, for example, that for *a* = 0.2, a 1% increase in pressure gives a 0.2% decrease in equilibrium moisture content. The near constancy of *a* at value of around 0.2 suggests that it is related to the cell wall structure of pulp fibers.

Structure of cell wall

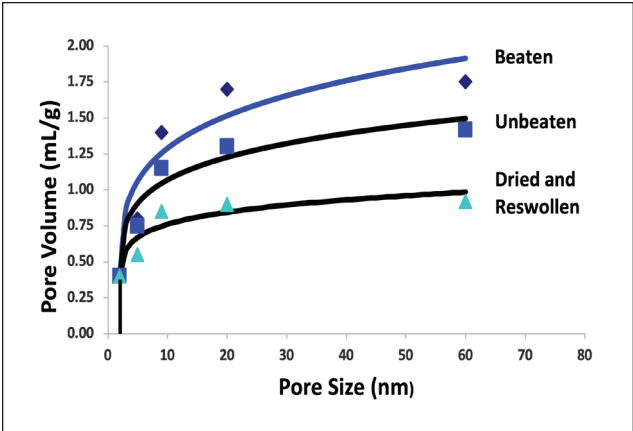
Pore size distribution in wet pulp fibers has been the subject of many studies using different approaches. Examples are nuclear magnetic resonance (NMR), solute exclusion, polymer adsorption, and thermoporosimetry [11]. Each approach relies on assumptions and therefore has shortcomings. NMR and thermoporosimetry measure water whose properties have been altered through intimate contact with the pore walls. Fiber saturation point measurements have shown that the NMR and thermoporosimetry techniques only measure a portion of the available water for pressing, which means that these methods underestimate the amount of water that can be pressed out [20].

On the other hand, solute exclusion and polymer adsorption techniques measure penetration of large molecules into the cell wall from an ambient solution. In essence, these measure pores that are “accessible” to the fiber exterior. Our focus is pressing water from within fibers, and therefore these approaches are of direct interest.

In pioneering work, Stone et al. [12] studied cell wall structure using a solute exclusion technique. Pulp fibers are placed in solutions of molecules of known size, and the concentration of the solution is measured. Solution concentration increases when the size of the solute molecule exceeds that of the pores because the molecules cannot penetrate the pores. Pore size distribution is measured as cumulative pore volume up to a given pore size. Examples are shown for a softwood kraft (Fig. 2) and sulfite pulp (Fig. 3) [12]. For wet fibers, this cumulative pore



2. Pore size distribution of softwood kraft pulp measured by means of solute exclusion from [12]. The curves are from fits to the surface tension model (Eq. 5) with coefficients given in Table II.



3. Cumulative pore volume of sulfite pulp as a function of pore size from [12]. The curves are fits to the surface tension model with coefficients given in Table II.

volume is in effect the cumulative water in all the pores up to a specific pore size. The key assumption is that pores are either fully accessible or fully inaccessible to solute molecules. This assumption has been questioned. Measurements on porous glass have shown that the solute molecules must be significantly smaller than the pore size

for complete penetration [13-15]. In addition, some voids within the cell wall are unavailable because of the “ink bottle” effect [14].

Based on the data of Stone et al. [12], Page [16] postulated the existence of additional pore volume in the form of “cracks” in fibers. These are fissures visible under a microscope having a size range 100 to 1000 nm. They may contain a significant amount of water that is not intimately connected with the fiber wall and likely pressed out in flow-controlled pressing. Page also noted that the cumulative water in pore sizes up to 50 nm corresponded to the fiber saturation point (FSP), defined as water within fiber cell walls.

To overcome shortcomings of the solute exclusion technique, Alinec and van de Ven [15] proposed a polymer adsorption technique to measure cell pore size. Here, polymer molecules of known size penetrate the pores of fibers and coat pore walls to the extent that penetration by additional molecules is blocked. Pore size is determined by comparing this penetration level to that found for glass of known pore size. Their findings suggest an absence of pore sizes in the range of 2–40 nm, which contradicts those of Stone et al. [12], as well as pore size distribution measurements of dry fibers using mercury porosimetry and nitrogen sorption [17]. Moreover, Alinec and van de Ven proposed that when a dilation effect is taken into account, the solute exclusion technique gives a mono-disperse pore size of 10 nm.

The most recent and comprehensive study of cell wall structure is that of Maloney and coworkers [18-21]. They employed a thermoporosimetry technique to measure water in “micropores,” defined as thermodynamically-altered water having a depressed freezing temperature [20]. Micropore water was found in a continuous distribution of pore sizes from 1 to 100 nm. They defined a second range of pore sizes called “macropores” as the difference between water in micropores and the FSP (determined by solute exclusion). Lastly, they identified a subgroup of micropores less than approximately 1 to 2 nm that contain “non-freezing water.” It was observed that refining mostly increased the volume of macropores, leaving the volume of micropores largely unchanged [20].

It is beyond the scope of this study to resolve differences in the findings of past work cited here previously. However, we note that solute exclusion and thermoporosimetry both show a continuous distribution of pore sizes from a few nm. In addition, pressing measurements by Carlsson et al. [22] have shown that solute exclusion can be used to quantify water removal from pores of different dimensions as pressing proceeds. Consequently, we will employ the findings from the solute exclusion method as a measure of “pressable” water from the fibers. Indeed, response to wet pressing, the inverse of penetration from an exterior source, may shed further light on pore structure.

ANAYLSIS

For pore sizes less than 50 nm, the cumulative pore volume follows a log-normal distribution in pore size [12,16,23]. Although it has been hypothesized that surface tension of water in this pore size range controls equilibrium moisture, the mechanism was considered too complex to model [24,25]. We now propose such a model.

We employ the basic assumptions of the DPM [1] that: 1) diminishing rate of water removal in wet pressing is caused by increasing resistance to flow, not diminishing pressure acting upon water, and 2) there is no true structural pressure that shields water from pressure, meaning that all water in the fiber is subject to the applied pressure. We assume that this assumption holds down to the nanometer scale, in essence treating the problem as a flaccid fiber wall limiting case. Consequently, at equilibrium, flow ceases due to zero permeability. This comes about when pores have no pathway to the fiber surface or when pathways have been closed by the applied pressure. The validity of these assumptions for the extreme cases of pressing in this study are discussed at the end of this paper.

There is also a third resistance. At equilibrium, many fiber surfaces are exposed to air. This introduces a surface tension resistance to flow at fiber/air interfaces. Assuming that surface tension controls equilibrium, we may link equilibrium moisture to a critical pore size by a force balance between pressure and surface tension resistance:

$$p_e \cdot \frac{\pi}{4} d_e^2 = \pi d_e \sigma$$

where σ is the surface tension (assuming zero contact angle), which gives the Young-Laplace equation:

$$p_e = \frac{4\sigma}{d_e} \quad (3)$$

Equation 3 implies that all pore sizes larger than d_e will have an equilibrium pressure less than p_e and therefore water will be expelled from the total of these pores at pressure p_e . In contrast, all pores smaller than d_e will require a pressure larger than p_e to expel water and therefore water will remain in the fiber. Thus, d_e represents the cumulative water in all pores up to this size.

We may substitute Eq. 3 into Eq. 1 to link equilibrium moisture content to pore size, d_e :

$$m_e = c(4\sigma)^a d_e^a \quad (4)$$

In Eq. 4, moisture m_e is all the water at equilibrium. However, based on our interpretation of Maloney’s findings, not all this water is available to be pressed out. In particular, water can’t be pressed from pores smaller than

k	a	c	d_o nm	R^2	Pulp Type	Comments	Source
0.85	0.20	1.1	1.98	0.90	Softwood sulfite	Beaten 13000 revs	PFI
0.75	0.17	0.93	1.98	0.92	82% α cellulose	Unbeaten	Stone et al. [12]
0.58	0.13	0.30	1.95	0.86		Dried and reswollen	
0.66	0.21	0.86	1.90	0.87	Softwood kraft	Beaten 13000 revs	PFI
0.61	0.19	0.78	1.91	0.93	88% α cellulose	Unbeaten	
0.45	0.13	0.31	1.10	0.96		Dried and reswollen	
0.61	0.39	1.0	1.60	0.97	Spruce sulfite	53.6% yield	Stone and Scallan [26]
0.96	0.23	1.29	2.45	0.99		66.6%	
1.00	0.11	1.15	3.14	0.98		75.3%	
0.70	0.074	0.77	2.00	1.00		87.7%	
0.42	0.50	0.79	1.29	0.99	Spruce kraft	44.6%	Stone and Scallan [26]
0.88	0.19	1.12	1.89	0.97		69.1%	
0.83	0.13	0.98	1.64	0.90		76.4%	
0.73	0.058	0.79	1.86	0.95		89.3%	
1.21	0.11	1.39	2.00	1.0	Spruce chlorite	70% yield	Ahlgren [27]
0.86	0.14	1.03	1.90	1.0		75%	
0.78	0.044	0.82	1.98	1.0		80%	
0.57	0.070	0.623	1.41	1.0		84%	
0.53	0.017	0.54	2.00	1.0		94%	
0.44	0.030	0.46	1.98	1.0		100% (wood)	

II. Coefficients for Eq. 5 using chemical pulp data from the literature.

d_o . This suggests another mechanism may be important on this scale, as discussed later in this paper. Our interest is equilibrium resulting from mechanical pressing; therefore, we propose a “pressable equilibrium water” as m_e^* and link this to pore structure by:

$$m_e^* = k(d_e - d_o)^a \quad (5)$$

Thus, $m_e^* = 0$ when $d_e = d_o$ and Eq. 5 can be shown to retain the form of Eq. 2 and thereby retains the physical significance of a .

Next, we consider parameter c , which was defined earlier as the equilibrium moisture at $p_e = 1$ MPa. Over most of our range of interest, $d_e \gg d_o$, and therefore $d_e - d_o \approx d_e$ and $m_e^* = m_e$. Thus:

$$c = \frac{k}{(4\sigma)^a} \quad (6)$$

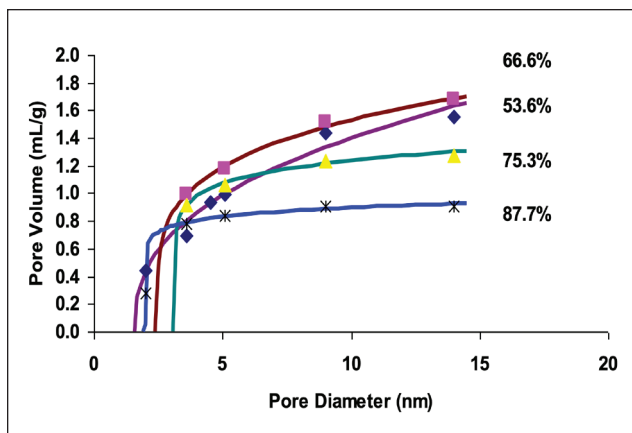
Assuming $\sigma = 70 \times 10^{-3} \text{ N/m}$, we obtain:

$$c = 0.28^{-a} k \quad (7)$$

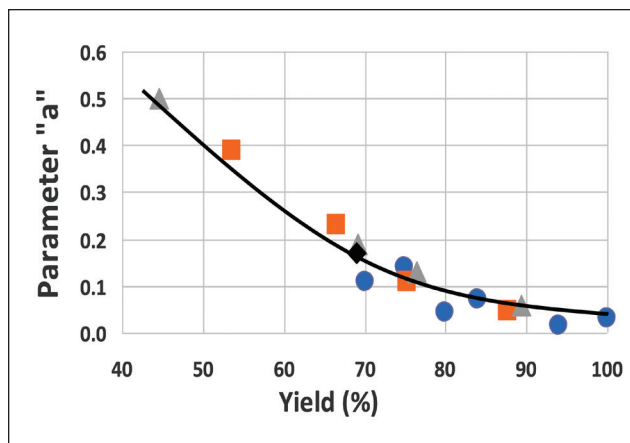
COMPARISONS TO THE EXPERIMENT

To test our model, we determined values of a , k , and d_o by means of nonlinear least squares fits of Eq. 5 to the data in Figs. 2-3 and references [26,27]. Values of c were determined from Eq. 7. Reference [26] measured the pore size distribution of kraft and sulfite pulps by solute exclusion as a function of yield. Reference [27] describes work in which lignin was removed by sodium chlorite extraction. The coefficients for Eq. 5 are shown in **Table II** and the fitted lines are shown in **Fig. 2-6**.

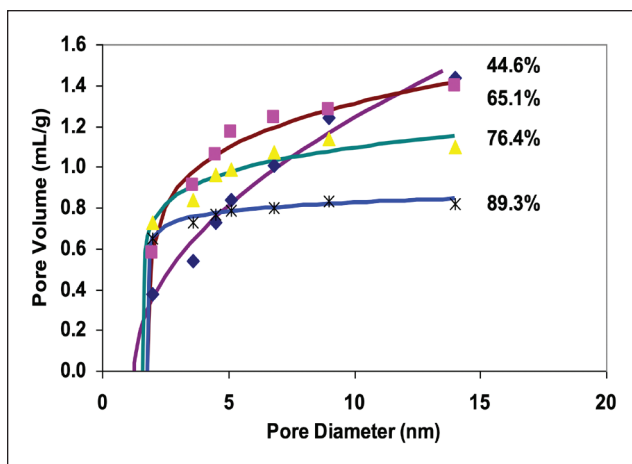
It is apparent in Table II that values of a and c depend strongly on yield. This is clearly shown in plots of a vs. yield in **Fig. 7** and plot of c vs. yield in **Fig. 8**. We include with these data the result from platen pressing (Table I [1]), for



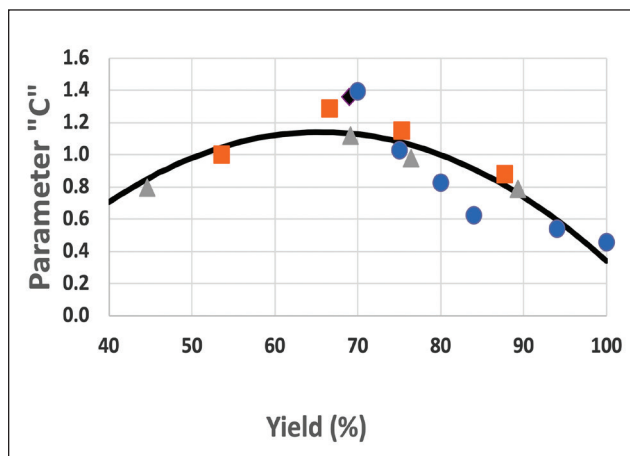
4. Pore volume as function of pore size diameter for spruce sulfite pulp as a function of yield from [26]. Lines are fits to the surface tension model (Eq. 5).



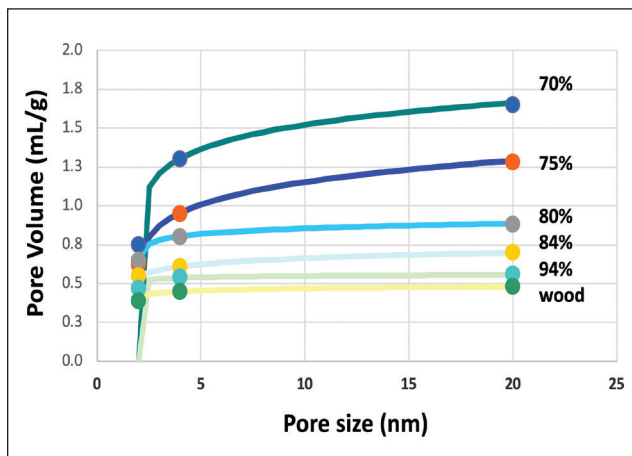
7. The a parameter from surface tension model (Eq. 5) is a measure of the responsiveness of equilibrium moisture to pressure. The a parameter increases as yield is lowered for kraft (▲), sulfite (■), and chlorite (●) pulping (Table II). This trend is consistent with the result for 69% yield, platen-pressed kraft (◆) (Table I).



5. Pore volume as function of pore size diameter for spruce kraft pulp as a function of yield from [26]. The trends as a function of yield are similar to those in Fig. 4.



8. The c parameter from the surface tension model (Eq. 5) is the equilibrium moisture at 1 MPa. The values of c from Table II for kraft (▲), sulfite (■), and chlorite (●) treatments as a function of yield reach a maximum at 65% yield. The fitted line is a quadratic equation with $R^2 = 0.74$

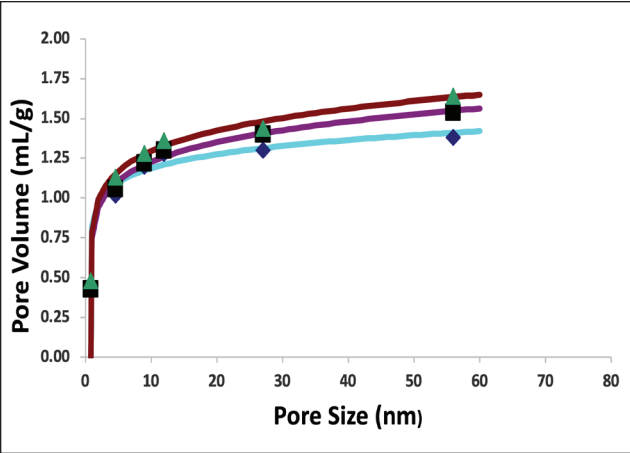


6. Effect of chlorite removal of lignin on the pore size distribution of spruce [27]. Pore volume is greater for lower yields. The fitted lines are for Eq. 5.

which we have information on yield. It lies on the trend line calculated from the solute exclusion, pore size distribution measurements.

The fitted line in Fig. 8 is a quadratic equation that quantifies the pulping mechanism described earlier. In the high yield range, water content increases with decreasing yield as water fills voids created by lignin removal and at lower yields, water content decreases with decreasing yield due to pore collapse.

Solute exclusion measurements for an aspen bleached chemithermomechanical (BCTMP) pulp [28] are shown in Fig. 9 with fitted lines to Eq. 5. Beating increases the volume of water in the larger diameter pores (Fig. 9), and this water is more responsive to pressure as indicated by the larger value



9. Pore size distribution of aspen BCTMP after being treated for 0 (♦), 6000 (■), and 12000 (▲) revolutions in a PFI mill [28]. The platen pressed pulp (◆) falls on this line.

of a (Table III). Assuming that the yield of the aspen BCTMP was in the range of 70% to 85%, the values of the coefficients (a and c) fall on the trendlines in Figs. 7-8.

DISCUSSION

Qualitative pictures of the chemical pulping process

At low yield, Stone and Scallan [29] proposed a multilamellar cell wall model in which water is contained in slit-like openings between the lamellae as well as pores within them. They arbitrarily picked 2.5 nm as the dividing point between the small intra-lamellae pores and the large inter-lamellae pores [29]. Ahlgren and Goring [30] postulated that the voids left by removal of lignin by sodium chlorite have the same size and shape as the lignin molecules and that the larger molecules come out later in the pulping process. These hypotheses suggest a porous structure compatible with our assumptions of pressure acting on all water and pores having pathways to the fiber surfaces.

Link between equilibrium moisture and pore size

The good fits of Eq. 5 to the data in Tables II and III support the postulate of a strong link between equilibrium moisture and pore size. This finding also supports earlier views that water is preferentially pressed out from larger pores [18,22] and, lastly, that a lower limit exists on pore size from which water can be mechanically pressed. This

latter lower limit is about 2 nm, a value close to Maloney’s finding for non-freezing water [21].

Because of the excellent relationship between equilibrium moisture and pore size, we postulate that it may be possible to replace difficult solute exclusion experiments with much simpler platen pressing experiments, such as those in Table I, to obtain the pore size distribution of pulp.

Effect of drying

Drying closes larger pores as shown in Figs. 2-3 and by the lower values of the c and a parameters (Table II).

Effect of beating

Beating of chemical and high-yield pulp increases the value of the a parameter (Tables II and III), which means that removal of water in beaten pulp has a stronger responsiveness to pressure (Eq. 2). Beating also gave a larger value of c for kraft and sulfite pulp (Table II), which means there is more water in the beaten pulp since c is the equilibrium moisture at a pressure of 1 MPa. There was no trend for the high-yield pulp (Table III).

Effect of yield

As shown in Fig. 8, decreasing yield from the level of wood causes c to increase to a maximum, then decrease with further reduction in yield. Stone and Scallan [29] postulated that as material is removed at high yield, water replaces the voids left by the extracted lignin. In addition, smaller pores coalesce to form larger pores, and thereby shift the pore size distribution to pores of greater volume. As a consequence, equilibrium moisture increases as yield is decreased to about 65%. The size of this increase, 0.4 to 1.3, is similar to that found by both pressure-plate and non-solvent water techniques over this yield range. Stone and Scallan [31] proposed that additional removal of lignin leads to collapse of pores, which diminishes the water-holding capability of fibers and hence the moisture content. Figure 8 reflects this behavior.

As shown in Fig. 7, parameter a increases as yield decreases. This behavior reflects increasing responsiveness of equilibrium moisture to applied pressure as described by Eq. 1. We hypothesize that it occurs because, although greenwood contains substantial moisture, much of this water lies in pores having no pathway to an external surface. The result is little removal of moisture under pressure. Pulping opens pathways to external surfaces and thereby

k	a	c	d_{0r} nm	R^2	Pulp Type	Comments	Source
0.96	0.096	1.08	0.80	0.98	Aspen BCTMP		Hui et al. [28]
0.92	0.13	1.09	0.80	1.0		3000 revs	
0.97	0.13	1.14	0.80	1.0		12000 revs	

III. Coefficients for Eq. 5 using high yield pulp data from the literature (BCTMP = bleached chemithermomechanical pulp).

enables pressure to force water out. The final equilibrium moisture content consists of water in closed pores and water restrained by surface tension in exposed pores.

Chemical pulping at yields greater than 65% creates pores primarily between 5 nm and 15 nm as seen in Figs. 4-5. For yields below 65%, the pore size distribution is dominated by larger pores. In particular, the pores in the 5 nm to 10 nm range collapse, whereas the larger pores are less affected (Figs. 4-5). This creates a distribution dominated by large pores that offer less resistance to pressure and hence the higher value of the a parameter.

It is noteworthy that in contrast to the low value of a for wood ($a = 0.03$) in Table II, the value for TMP (about 95% yield) in Table I is about $a = 0.2$. This higher value is likely due to structural changes introduced by the robust action of the mechanical pulping process. Indeed, Maloney found that mechanical pulping produced small cracks in the cell wall that gave internal fibrillation [20]. This is also suggested by the value of $c = 0.63$ for thermomechanical pulp (TMP) in Table I, which is larger than the value of wood ($c = 0.44$) in Table II.

Validity of the model

The surface tension model assumes that at equilibrium, the applied pressure is fully supported by water within the fibers. This is an approximation, because for the smallest pores, there may be some mechanical support. In addition, some water will be contained in gels, which may have a different dewatering mechanism. However, we believe that the majority of the water within the fiber wall is exposed to external pressure given that even at the extreme pressure of 53 MPa, about one-third of the remaining mass is water [7].

SUMMARY AND CONCLUSIONS

The findings of this study support the hypothesis that surface tension and pore size distribution and their corresponding volume fraction are major factors determining equilibrium moisture produced by wet pressing conventional pulps. For chemical pulps, the equilibrium moisture reaches a maximum at about 65% yield. This finding is consistent with previous studies that suggest that water replaces removed material at high yields and pore collapse takes place at low yields. The finding is also consistent with there being a limit on the amount of water that can be mechanically pressed from wet pulp.

From a practical perspective, the important question relevant to this study is how to attain high solids content in wet pressing. The first requirement is complete pressing in the flow-controlled regime to reach the equilibrium-controlled range. Often, this is not the case. Once in the equilibrium range, water can be pressed from macropores but little from micropores. The macropore range is governed by species and pulping method. It is also strongly affected by pulp refining. Although the primary purpose of refining is paper quality, avoidance of unnecessary refin-

ing, or over-refining, may open possibilities to optimize the refining for both paper quality and pressing efficiency. This requires further study.

Other factors to reduce equilibrium moisture are beyond the control of papermakers but are worthy of mention here. In pulping, selective removal of xylan from the fiber wall with enzymes suggests that it may be possible to improve water removal without compromising pulp strength [32,33]. Raw material selection plays a role in equilibrium moisture: water retention value (WRV) decreases with increasing cell wall thickness for eucalyptus [34,35]; average fiber cell wall thickness depends on species growing location and tree age [36]; and sawmill vs. whole tree chips [36] may impact equilibrium moisture. Lastly, it may be possible in the future that new enzymes could surgically extract materials from wood so that the fiber is flexible and conformable without retaining a high amount of water. **TJ**

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We studied equilibrium moisture because although it can be measured, there was limited information on the factors that control it. Our work on modeling water removal in wet pressing identified equilibrium moisture as the ultimate limit in pressing. Over a period of several years, we explored various aspects of this subject by examining past literature on pore size distribution and water content in pulp fibers. We eventually focused on methods of measuring pore structure by penetration of water from outside fibers, with the view that such pores having a pathway into fibers were of direct relevance to pressing water out from fibers. In the end, we found we could develop a theory that was consistent with these observations.

Through our study, we discovered that we could explain the results of platen pressing experiments from the structure of pulp fibers. Using a simple theory, based on surface tension of water at the interface of pores and air, we were able to show that

equilibrium moisture is related to the wall structure of pulp fibers. The results were surprisingly consistent using data from several sources and using different techniques.

Equilibrium moisture defines the ultimate limit in wet pressing. It can be used to understand how to optimize an existing mill operation or to design new equipment.

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