CELLULOSE MICROFIBRIL ANGLE AS A DETERMINANT OF PAPER STRENGTH AND HYGROEXPANSIVITY IN *PINUS TAEDA* L.

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ABSTRACT

To determine the importance of cellulose microfibril angle (MFA) for paper sheet mechanical properties, we prepared unbleached kraft pulps from ten individual, 10-year-old *Pinus taeda* (loblolly pine) trees similar in wood density, coarseness, cell-wall thickness, and fiber length but differing in MFA. MFA correlated ($R^2 = 0.81$) linearly with zero span tensile strength of dry sheets. MFA was shown to be a major determinant of handsheet tensile strength, stretch, modulus of elasticity, stiffness, and hygroexpansivity in unrefined and refined pulps at a probability level of ≥97%. These results strongly suggest that breeding for southern pine trees with decreased MFA in the juvenile wood is highly desirable for paper mechanical products.

Keywords: Elastic modulus, hygroexpansivity, kraft pulp, loblolly pine, microfibril angle, paper properties, stiffness, tensile strength, wood fiber, wood properties, zero span tensile strength.

INTRODUCTION

Wood fiber hygroexpansivity, tensile strength, and elasticity are important determinants of the tensile and compressive strengths, bending stiffness, and modulus of elasticity, as well as tangential and longitudinal shrinkage of solid wood and paper materials (Harris and Meylan 1965; Cave and Hutt 1968; Page 1969; Page et al. 1977; Page and Seth 1980; Page and El-Hosseiny 1983; Seth et al. 1986; Mark and Gillis 1993; Megraw et al. 1998). The tensile strength of paper is a function of fiber density, length, perimeter, and bond strength, (Bergman and Rennel 1967; Page 1969). Fiber strength (tensile and modulus of elasticity) is determined by cellulose microfibril angle (MFA) in *Picea mariana* (black spruce) and *Picea glauca* (white spruce) (Page et al. 1972; Page et al. 1977; Page and El-Hosseiny 1983). MFA is defined as the helical angle made by the cellulose microfibrils

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in the S_2 layer of the secondary wall relative to the longitudinal axis of the cell. In softwoods, because the S_2 layer constitutes ~90% of the mass of the tracheid, its properties strongly influence the overall physical properties. MFA is a principal determinant of the ultimate tensile strength, for undamaged or defect-free fibers. When the fibers are damaged or have defects introduced during pulping and handling, the measured fiber tensile strength falls below this maximum value. Additional work developed the theoretical basis for the effect of MFA on fiber strength (El-Hosseiny and Page 1975) and elastic modulus (Page et al. 1977). The shape of the stress-strain curve for individual fibers was also explained by the MFA. During the tensile test, the fibril angle decreases, thus reducing shear stress till late in the stress-strain curve, when the reduction in MFA produces a steepening of the curve (Page and El-Hosseiny 1983).

The importance of MFA in determining single fiber tensile strength and elastic modulus has also been demonstrated for pine tracheids. In an extensive study of one 48-year-old *Pinus taeda* (loblolly pine) tree, the mechanical properties of individual tracheids as a function of growth ring and tree height were measured and compared to the MFA (Groom et al. 2002a, 2002b; Mott et al. 2002). Mature tracheids were found to be significantly stiffer and stronger than juvenile tracheids, primarily due to reduction in MFA. These results support Page's theory that strongly suggests that MFA is an important determinant of paper sheet tensile (Page 1969) and elastic modulus (Page et al. 1977) in loblolly pine.

For pine species, which have much thicker S1 and S2 cell-wall layers and significantly greater MFAs than spruce, results demonstrating the importance of MFA in controlling paper tensile strength and modulus of elasticity are conflicting. Early work showed that MFA was not a significant determinant of sheet tensile strength, but did significantly affect stretch in *Pinus radiata* (Watson and Dadswell 1964). More recent results with 13- and 15-year-old plantation grown *Pinus radiata* trees showed that MFA had a strong influence on handsheet tensile strength ($P = 0.001$), tensile stiffness index ($P = 0.003$), tear index ($P = 0.008$), and stretch ($P = 0.02$) (Kibblewhite et al. 2003).

The effect of MFA on the dimensional stability of paper is unclear. Paper expands or shrinks with changes in moisture content. The ability to minimize or control such dimensional changes is particularly important for printing and packaging papers. The change in paper dimensions due to relative humidity changes is known as hygroexpansion. Hygroexpansivity is a complex phenomenon, affected by many factors. One study (Uesaka and Moss 1997) examined the effects of fiber morphology including MFA on hygroexpansion. The hygroexpansion of a single fiber in the longitudinal direction was found to have a significant effect on the hygroexpansion of paper. The hygroexpansion of a single fiber then is controlled by the secondary wall structure. With a limited data set and range of MFA for a bleached Douglas-fir (*Pseudotsuga menziesii*) kraft pulp, it was found that juvenile and top wood fibers with a higher MFA showed greater hygroexpansivity. To date the effect of MFA on hygroexpansivity of paper made from pine species has not been demonstrated.

In this study, ten individual 10-year-old loblolly pine trees with a range of MFA were selected for processing into bleachable grade kraft pulps with the objective of determining the importance of MFA on handsheet zero span tensile index, stiffness, hygroexpansivity, elastic modulus, and stretch.

MATERIALS AND METHODS

The specific gravity and MFA of 10-year-old *Pinus taeda* L. loblolly pine trees were measured from wood cores taken at 1.5 m, as part of a clonal progeny test. Ten trees with similar mean specific gravities (0.394–0.412), but different MFAs in rings 3, 5, and 7 were selected (Table 1). For the five trees with high MFAs in rings 3, 5, and 7, the MFA between the rings and for earlywood and latewood within each ring was relatively constant, varying by only 3–4°. The five trees with relatively low MFAs showed the more typical cambial age profile where MFA decreased from ring 3 to ring 7 by 9–13° (Mc-

TABLE 1. *Wood properties.*

Tree no.	Wood specific gravity Rings 3–8*	Chip specific gravity 3.0–6.1 m	% latewood Rings 3–7	Mean microfibril angle 1.5 m#	3.0 m	6.1 m	3.0–6.1 m weighted
			Low MFA				
1	0.42	0.382	26.8	30	15	17	16
2	0.41	0.358	25.2	24	18	18	18
3	0.39	0.345	21.9	28	20	21	21
4	0.39	0.361	31.5	32	23	20	22
5	0.36	0.344	17.0	29	22	21	22
			High MFA				
6	0.38	0.332	17.2	48	27	28	28
7	0.46	0.374	28.6	45	32	24	29
8	0.40	0.321	24.2	46	30	30	30
9	0.41	0.380	16.9	49	31	29	30
10	0.41	0.354	17.4	47	37	25	34

* Mean basal area weighted SG for rings 3–8 at 1.5 m.
# Mean basal area weighted MFA from rings 3, 5, and 7.

Millin 1973; Megraw et al. 1998) and the MFA of the latewood was usually lower by 3–6° than the MFA of the earlywood (Megraw 1997; Groom et al. 2002b).

The ten 3.0–6.1-m sections of the trees were harvested in May 2001 from the same controlled site of timberland in North Carolina and transported to the Institute of Paper Science and Technology in Atlanta, GA, for pulping and testing. All logs were debarked by hand, then chipped in a 1.5-m Carthage chipper. The chips were screened on a RADER™ disc screen and the 2–8 mm-thickness fractions retained. The chip specific gravity was determined by water displacement in a pycnometer according to TAPPI T258. Chip moisture was determined by drying in a 105°C oven.

Pulping was done in 10-L laboratory batch digesters equipped with external circulation and electric heaters (Courchene et al. 2000). The chip charge was 1000 o.d. g. White liquor was made in the laboratory from solutions of NaOH and Na_2S. The liquor concentration was determined with a standard ABC test using an automatic titrator (Table 2). Duplicate cooks were made for each chip source. After the cooking liquor was drained, the chips were removed, disintegrated in a tank, and the pulp was screened through 0.15-mm slots with a 610-mm × 305-mm flat screen. The accepted pulp was collected and thickened in a centrifuge. The pulp weight and consistency were measured and the screen yield was calculated. The kappa number of the screen accepts was measured according to TAPPI T236. The screen rejects were collected, dried, and weighed for calculation of total yield.

TABLE 2. *Cooking conditions.*

Active alkali	18.5%
% sulfidity	30%
Liquor:wood	4:1
Cooking temperature	170°C
Target H factor	1400

Coarseness and fiber length were determined with a Fiber Quality Analyzer™ from OpTest (Hawkesbury, Ontario) using standard OpTest protocols. The MFA of the wood was measured at 1.5-, 3.0-, and 6.1-m heights by X-ray diffraction (Megraw et al. 1998). Care was taken to avoid any compression wood. Briefly ~6-mm × 6-mm x 3-mm (tangential × longitudinal × radial) wood samples were dissected from the earlywood and latewood regions of each ring, and the MFA was determined by X-ray diffraction through the tangential face. The fibril angle from wood represents an average of ~400 fibers. The MFA of the ten pulps was estimated by differential interference contrast microscopy after ultrasound treatment in cobalt chloride (Peter et al. 2003). Images of ≥100 randomly picked tracheids from each pulp were captured with a Pixera Professional digital camera, and the MFA of each tracheid was determined with a two-dimensional fast Fourier transformation method (Jackson and Rothbard 1999). Care was taken to get 20–25 latewood fibers per pulp sample.

Standard basis weight handsheets of 60 o.d. gm^{-2} were made according to TAPPI T205. The zero span tensile strength of the dry sheets was measured according to TAPPI T231. The hygroexpansivity of 100-mm long × 15-mm wide strips was measured with a Varidim tester from TechPap (Grenoble, France). Samples were first conditioned at 50% relative humidity (RH) for 30 min. The RH was then cycled from 50 to 15%, back to 50%, then to 75%, and back to 50%. The test duration at each RH set point was 2 h.

Three pulps were also selected and refined to 2500 revolutions in a PFI mill according to TAPPI T248 and standard handsheets made. These handsheets were tested for apparent density (TAPPI T220), Taber stiffness (TAPPI T566), and tensile properties (TAPPI T494). The elastic modulus was determined by in-plane ultrasonic measurements utilizing an automated robot and a standard IPST procedure. The sheets were preconditioned from 20% to 50% relative humidity prior to testing, and all tests were done with five replicates.

Statistical analysis of the results was done by standard regression analysis using Number Cruncher Statistical Software™.

RESULTS AND DISCUSSION

Wood properties

The log from 3.0–6.1 m was harvested for kraft pulping and subsequent paper testing. The chip specific gravity (SG) is not well correlated with the wood SG at 1.5 m (Table 1). This difference may be due to methods used to measure SG, X-ray densitometry for the wood core vs. w:v for the chips, and the fact that the chips represent more of a composite sample from the 3.0–6.1-m logs that were used for pulping, whereas the wood core measurements represent only the 1.5-m position in the tree. The chip SG was similar for both the low and high MFA trees (Table 1). The ten trees were split into low and high MFA groups based on the 3.0–6.1-m weighted average MFA (Table 1). All of the trees in the low MFA group corresponded to the trees with low MFAs selected from the 1.5-m MFA data. As expected, the MFAs in the 3.0–6.1-m log were lower than the MFAs from 1.5 m (Table 1) as MFA is known to decrease with increases in tree height (Megraw 1998; Mott et al. 2002). The five trees in the low MFA group had basal area weighted averages of 16–22 degrees, while the five trees in the high MFA group had basal area weighted averages of 28–34 degrees.

Pulp properties

The properties of the unbleached kraft pulps were similar for the two MFA groups, except for their MFA (Table 3). Interestingly, although under constant pulping conditions there was some variation in kappa number and yield, MFA correlated well with pulp yield ($r = -0.79$). The low MFA trees had slightly higher pulp yields, even though the mean SG of the chips from the low (0.358) and high MFAs (0.352) were similar (Table 1). One potential explanation for the higher pulp yield in the low MFA trees is that they had higher cellulose levels or reduced lignin levels compared with the high MFA trees. The MFA measured in the pulps by differential interference contrast (Table 3) correlated well ($r = 0.86$) with the 3.0–6.1-m basal area weighted X-ray MFA measurements in the wood (Table 1). The length-weighted tracheid lengths fell into two groups, with three samples approximately 2.4 mm and the rest between 2.0–2.2 mm. These two groups were not correlated with the two MFA classes (Table 3); however, overall MFA and tracheid length were weakly correlated ($r = -0.66$). This weak negative correlation is probably explained by the observation that MFA is typically higher and fiber length is shorter in the juvenile fibers and MFA becomes lower and fiber length becomes longer towards the bark as cambial age increases (McMillin

TABLE 3. *Pulp properties.*

Tree no.	Kappa no.*	Total yield, %*	LW* fiber length#, mm*	Coarseness mg m^{-1}*	Cell wall thickness, μm	Pulp MFA
			Low MFA			
1	28.2	45.7	2.38	0.188	3.59	22
2	27.7	46.1	2.37	0.173	3.63	25
3	27.6	45.2	2.12	0.157	2.56	28
4	26.7	43.6	2.11	0.173	3.19	30
5	28.2	45.1	2.02	0.160	2.99	32
			High MFA			
6	29.0	44.0	2.43	0.202	2.96	31
7	27.7	41.9	2.15	0.205	3.20	32
8	26.1	41.3	2.08	0.155	2.54	37
9	30.6	42.9	2.11	0.179	2.82	39
10	25.6	41.9	2.06	0.183	3.03	34

* The average value from the duplicate cooks.
\# Length weighted (LW) fiber length.

1973; Megraw 1997). MFA and tracheid coarseness were not correlated. As expected, MFA and wall thickness were negatively correlated (r = –0.69) in agreement with the results of Hiller (1964). This negative correlation is most likely explained by the observation that latewood tracheids have lower MFAs and thicker cell walls than earlywood tracheids (McMillin 1973; Megraw 1997).

Paper sheet properties of unrefined pulps

Theory and comparison with data from other species predict that reduction in the pulp MFA will increase the tensile and compressive strength (Page 1969; Seth et al. 1986) and reduce the hygroexpansivity (Uesaka and Moss 1997) of paper sheets. Zero span tensile strength is a relative measure of fiber strength (Page 1969) and strongly depends on cellulose content and tracheid MFA in spruce (Gurnagul et al. 1992). Figure 1 and Table 4 show that paper sheets made with fibers of decreasing MFA have greater zero span tensile strengths. MFA and zero span tensile strength (ZST) were highly correlated with a probability level of 99%. The regression equation is as follows:

$$ZST = -2.63 \times MFA + 216.4; \quad R^2 = 0.8 \qquad (1)$$

ZST was not correlated with fiber length, coarseness, cell-wall thickness, or handsheet density. These results confirm that tracheids with lower MFA have greater tensile strength (Page et al. 1972; Groom et. al 2002a) and that paper sheet strength increases with reductions in tracheid MFA for loblolly pine as predicted (Page 1969; Page et al. 1972).

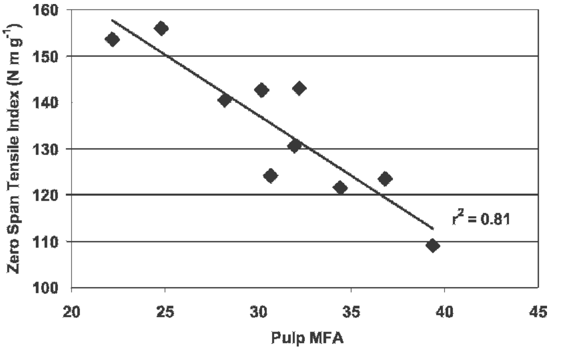

FIG. 1. Zero span tensile index of unrefined handsheets as a function of the MFA for all ten pulps.

To test the importance of MFA on hygroexpansivity, handsheets made from pulps with different MFAs were compared. The length of a 100-mm strip was reduced when relative humidity (RH) went from high to low, and when RH was raised, the strips expanded (Table 4). In order to compare results across several cycles, a hygroexpansion coefficient (HC) was calculated according to the formula:

$$HC = | \text{percent change in length/percent change in RH} | \qquad (2)$$

TABLE 4. *Zero span tensile strength and hygroexpansivity for unrefined handsheets.*

Tree #	Pulp MFA	Zero span tensile index Nm g^{-1}	Hygroexpansivity, % length change			
			RH 50→15%	RH 15→50%	RH 50→75%	RH 75→50%
			Low MFA			
1	22	154	–0.14	0.14	0.12	–0.09
2	25	156	–0.14	0.13	0.10	–0.09
3	28	140	–0.13	0.14	0.14	–0.12
4	30	142	–0.16	0.17	0.12	–0.12
5	32	143	–0.20	0.20	0.16	–0.14
			High MFA			
6	31	124	–0.21	0.20	0.16	–0.14
7	32	131	–0.21	0.20	0.17	–0.15
8	37	124	–0.19	0.17	0.19	–0.14
9	39	109	–0.23	0.22	0.22	–0.20
10	34	122	–0.21	0.20	0.17	–0.15

The hygroexpansion coefficient as a function of MFA is shown in Fig. 2. MFA and the hygroexpansion coefficient were significantly correlated at a probability level of 99%. The regression equation determined for this relationship is:

$$HC = 1.92E{-}04 \times MFA; \;\; R^2 = 0.82 \qquad (3)$$

This significant result confirms the earlier work with Douglas-fir (Uesaka and Moss 1997), and extends to loblolly pine this initial finding that handsheets made with lower MFA tracheids have reduced hygroexpansivity and thus have improved dimensional stability. The improved dimensional stability of randomly oriented handsheets made from pulps with lower average MFAs suggests that this result may also translate to commercially produced sheets that have a machine and cross direction.

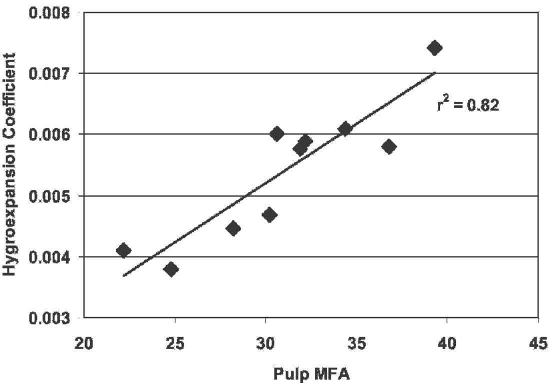

FIG. 2. Hygroexpansion coefficient of unrefined handsheets as a function of the MFA for all ten pulps.

Paper sheet properties of unrefined and refined pulps

By improving fiber-fiber bonding and by increasing sheet density, refining of the pulp has a large impact on the properties of paper sheets. The mechanical properties of handsheets made with unrefined and refined pulps were compared for three of the pulp samples, 1, 6, and 9 with a low, mid, and high MFA, respectively. The properties of sheets made with these unrefined and refined pulps are summarized in Table 5.

The tensile index of sheets increased with reductions in MFA and sheets made with the refined pulps had greater tensile strength than the sheets made with unrefined pulps (Fig. 3A). As MFA increased, sheets had greater stretch, with the refined sheets stretching more than the unrefined sheets at the respective MFA (Fig. 3B). Tensile energy absorption (TEA) index is calculated as the area under the stress strain curve and is a function of both the tensile strength and the stretch. TEA is a measure of the amount of energy that paper can absorb before breaking and is important for some packaging grades such as sack paper. The TEA index initially increased as MFA went from 22° to 31° due to the greater stretch but then decreased as the MFA increased

TABLE 5. *Properties of unrefined and refined handsheets.*

Tree #	Pulp MFA	PFI refining, revs.	Apparent density g cm^{-3}	Stretch %	TEA* index mJ g^{-1}	Tensile index N m g^{-1}	Elastic modulus GPa	Taber stiffness mN m
1	22	0	0.510	1.65	585	52.4	4.33	0.171
		2500	0.564	2.52	1340	79.0	7.53	0.123
6	31	0	0.427	2.58	863	46.3	3.44	0.143
		2500	0.653	3.53	1894	74.8	6.71	0.089
9	39	0	0.487	2.80	843	40.8	2.92	0.119
		2500	0.611	3.76	1735	61.7	5.64	0.079

* Tensile energy absorption index.

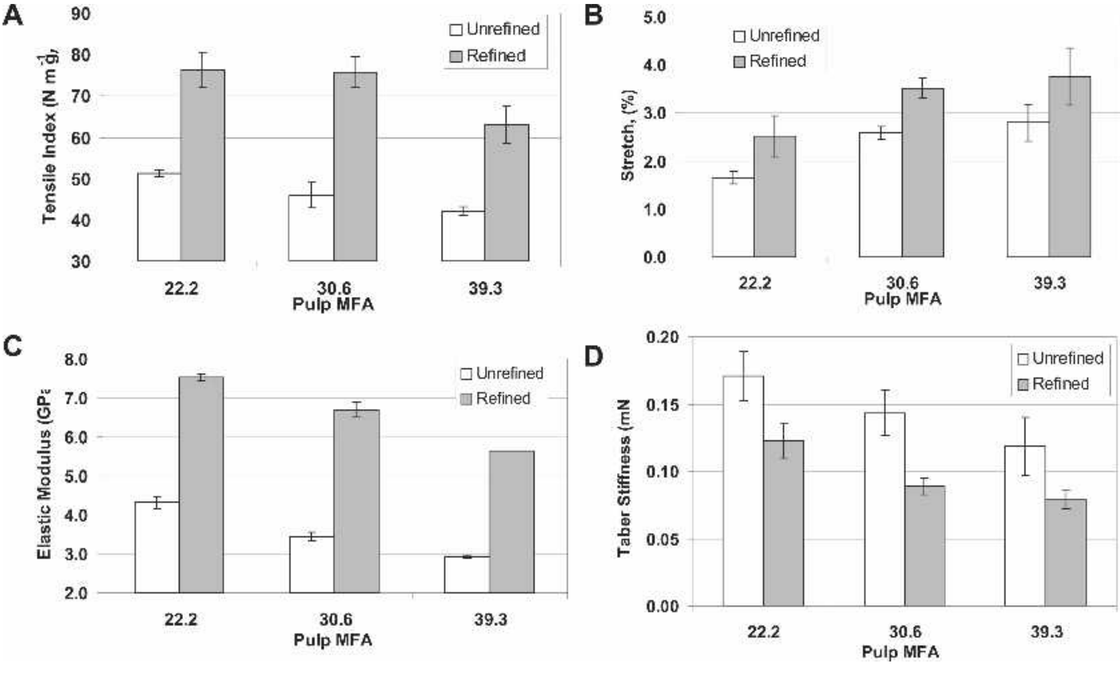

FIG. 3. Mean tensile index (A), stretch (B), MOE (C), and taber stiffness (D) with the 95% confidence interval of handsheets made with refined (grey) or unrefined (white) pulps 1, 6, and 9 as a function of MFA.

further to 39° due to the reduced tensile strength (Table 5). The MOE also increases with reductions in MFA (Fig. 3C), for both the refined and unrefined pulps. The MOE of the refined sheets is significantly higher than the unrefined sheets, due to improved bonding of the refined pulps (Fig. 3C). The increased Taber stiffness of the unrefined sheets, even though they have lower MOE's (Fig. 3D), is a function of the Taber stiffness calculation where MOE is multiplied by the sheet caliper to the third power. Thus, bending stiffness is very sensitive to sheet caliper, which is lower for the refined than the unrefined sheets as evidenced by the higher sheet densities (Table 5). The refined handsheets had lower Taber stiffness than the unrefined sheets (Fig. 3D).

Since the apparent density of the sheets varied (Table 6), regression analysis was used to determine the separate significance of MFA and sheet density on the measured mechanical properties. Two linear models, one with MFA plus a variable indicating refined or unrefined and another with sheet density, were evaluated for each mechanical property. The results in Table 6 show that MFA along with refining had a significant

TABLE 6. *Significance of factors determining handsheet properties.*

Model		Taber stiffness	Elastic modulus	Tensile index	Stretch
MFA	P*	0.005	0.00026	0.03	0.019
Refined	P	0.0028	0.00023	0.0019	0.022
	R^2	0.979	0.994	0.977	0.932
Handsheet density	P	0.067	0.048	0.032	0.14
	R^2		0.896	0.723	

P = probability level.

effect for each property at a probability level of 95% or greater ($P < 0.05$). The R^2 values indicate a good fit in each case. The handsheet density was only significant at the 95% level for elastic modulus and tensile index and the model fit was less than that for MFA. The finding that MFA significantly affects sheet tensile and elastic modulus confirms the theory that MFA is a direct determinant of sheet tensile and elastic modulus even in loblolly pine where the cell walls are much thicker than in spruce (Page et al. 1969; Page and Seth 1980).

CONCLUSIONS

In loblolly pine, tracheid cellulose microfibril angle is a major determinant of paper tensile strength, stiffness, MOE, stretch, and hygroexpansivity. Thus, breeding for trees with reduced MFA, especially in the juvenile wood will positively impact paper production leading to stronger sheets that have improved dimensional stability.

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