Impact of fiber structure on edge-wicking of highly-sized paperboard

LEBO XU, JEREMY MEYERS, AND PETER W. HART

ABSTRACT: Coffee edge-wicking testing was conducted on two groups of highly-sized paperboard manufactured at two mills with similar manufacturing processes, but with vastly different local fiber sources. Although the Hercules size test (HST) indicated similar internal size levels between the two types of board, the edge-wicking behavior was noticeably different. Analysis of fiber structure revealed that the board with more edge-wicking had fibers with thicker fiber walls, which kept the fiber lumen more open after pressing and drying on a paper machine. It was demonstrated that liquid penetration through voids between fibers in highly-sized paperboard was limited, because the fiber surface was well protected by the presence of sufficient sizing agent. Nevertheless, freshly exposed fiber walls and lumens at the cut edge of the sheet were not protected by sizing material, which facilitated edge-wicking. The correlation between fiber structure and edge-wicking behavior was highlighted in this work to inspire development of novel sizing strategies that protect the freshly cut edge of the sheet from edge-wicking.

Application: The current work shows the contribution of fiber structure to the edge-wicking process for highly-sized paperboard after all the surfaces are protected with size. Understanding the impact of fiber structure on edge-wicking should inspire the development of novel sizing strategies to further reduce edge-wicking.

Paper is easily wet by solvents similar to water due to the intrinsic hydrophilicity of cellulose fibers. With treatment of hydrophobic material, known as size, wetting can be delayed, which makes the manipulation of the wetting behavior possible during the papermaking process. The desired level of water resistance is governed by the end use of the paper product, which may include high moisture environments or direct water contact.

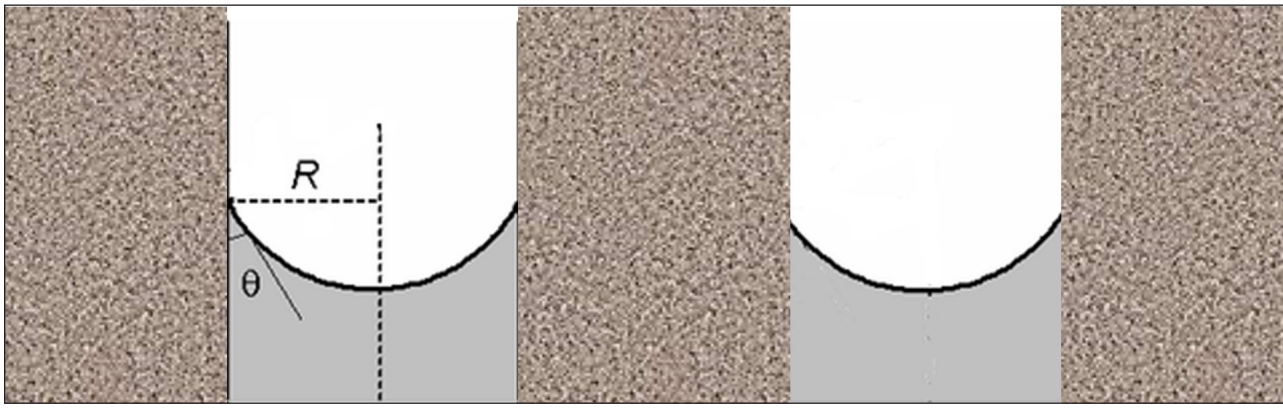
Previous research has suggested four major mechanisms for water transport in paper [1-3]:

1. Capillary penetration in the void between fibers.
2. Diffusion on the fiber surface.
3. Diffusion through fibers.
4. Vapor phase transport.

For applications such as printing and general packaging, liquid penetration perpendicular to the paper surface is a major concern. In this case, capillary penetration in the void spaces between fibers is the most important wetting mechanism.

Capillary penetration in paper voids is well described by the Lucas-Washburn equation, which is widely used to describe liquid penetration in a porous substrate [4-5]. Originally, the equation was developed to describe capillary flow in an array of parallel cylindrical pores, as shown in **Fig. 1**. The Lucas-Washburn equation can be expressed as follows:

$$l = \sqrt{\frac{\gamma \cdot R \cdot \cos\theta}{2\eta}} \cdot \sqrt{t} \qquad (1)$$



1. Scheme of capillary flow in an array of parallel cylindrical pores.

where l (m) is penetration depth, γ (dyne/cm) is liquid surface tension, R (m) is radius of cylindrical pore, θ (°) is contact angle, η (cp) is liquid viscosity, and t (s) is penetration time. One can see that the properties of both the liquid (γ,η) and the substrate (R,θ) have an impact on liquid penetration. To make paper more resistant to water penetration, many sizing materials (such as rosin, alkenyl succinic anhydride [ASA], alkylketene dimer [AKD], etc.) were developed to modify the surface of fibers to reduce the surface tension. The modified surface gives a larger water contact angle, which translates to a smaller $\cos\theta$ value in the Lucas-Washburn equation, and therefore less water penetration (smaller l value). The pores in the scheme represent voids between fibers, not the fiber lumen. Liquid flow through the fiber (inside the fiber wall and in the lumen) is not considered by the Lucas-Washburn equation.

Edge-wicking is liquid penetration into the paper sheet from the in-plane direction rather than the cross direction. It is a concern for certain paper packaging applications where a cut paper edge is exposed to a liquid, such as with paper cups and juice or milk cartons. Edge-wicking can vary from a minor cosmetic problem, such as stained or discolored raw edges of a paper cup, to more serious package failures around seams. The current industrial trend is towards packaging applications that are considerably more challenging with respect to edge-wicking; for example, liquid packaging manufacturing additives may contain aggressive surfactants. Harsh processing conditions (peroxide or steam treatment) may be employed to sterilize paper packages in filling lines. Factors such as these adversely affect edge-wicking and can potentially lead to package failure. In many cases, plastic laminate, such as polyethylene, is used as a popular approach in addition to a sizing program for enhanced water resistance in the perpendicular direction to the paper surface. However, lamination does not address the edge-wicking issue, as lamination material does not cover the fiber at the cut edge of a sheet of paperboard.

Over the last fifty years, substantial efforts have been made to reduce edge-wicking [6-13]. Most of these efforts have focused on chemical modification of the fiber surface via sizing programs. With sufficient sizing, capillary penetration of water into the voids between fibers is significantly reduced and may no longer be the dominant contribution to overall liquid transport into the paper sheet. In fact, some studies have indicated that liquid diffusion within the fiber wall and in fiber lumens is the major mechanism of liquid transport in highly-sized paper [14-18]. Based on these findings, fiber structure, rather than chemical surface modification, should have a major impact on edge-wicking in highly-sized paper, in which the fiber surface has been well protected by sizing material. However, to our knowledge, no work has been done to reveal the effect of fiber structure on edge-wicking of highly-sized paper.

For the current work, several highly-sized paper samples were obtained from two North American mills with vastly different fiber sources. These samples were produced to meet the same grade specification. Even though these samples were made with a similar sizing program and achieved the same Hercules size test (HST) targets, the samples exhibited significantly different edge-wicking behavior. These samples were compared to each other to help understand how the physical fiber structure contributes to edge-wicking. The mechanism of edge-wicking in highly-sized paper was also investigated. By understanding the impact of fiber and sheet structure on edge-wicking behavior, we hope to find ways to mitigate edge-wicking and develop new sizing strategies beyond the internal size program currently employed in mills.

EXPERIMENTAL

All paper samples were conditioned at TAPPI standard temperature and humidity (TAPPI T 402 sp-08 "Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products") prior to being tested. General paper property tests, such as grammage and caliper, were measured with TAPPI T 410 om-13 "Grammage of paper and paperboard (weight per unit area)" and TAPPI T 411 om-15 "Thickness (caliper of paper, paperboard, and combined board)," respectively. Sizing of paperboard was evaluated by HST. The test was conducted at room temperature with reflectance of 80%. Green dye ink with 20% (by weight) formic acid was used in the test.

Edge-wicking of paperboard was evaluated by a hot coffee test. A sample paperboard was laminated with plastic film (Scotch thermal laminating pouch, 3M; Maplewood, MN, USA) on both surfaces. The laminated board was cut into strips of 1 in. x 5 in. Freshly brewed coffee with a powdered creamer (creamer concentration 3.6 v/v%) was used as the testing liquid, and the temperature of the coffee/creamer mixture was maintained at 90°C. The test strip was inserted 3 in. into the hot coffee/creamer mixture for a specified amount of time. When the specified time was reached, the strip was removed from the solution and wiped dry for evaluation. Tested strips were scanned to form color images (TIFF format). The color images were converted to binary images with ImageJ software (1.50i) (National Institutes of Health; Bethesda, MD, USA). The black pixel of a binary image represents coffee stain. Coffee penetration depth was measured on each binary image with the ImageJ software. The total number of black pixels counted by the ImageJ software represents the total amount of absorbed coffee. This value is referred to as the absorption index of the sample.

The amount of hydrophobic material within each of the paper samples was determined by extraction from the different samples obtained from different mills. The total amount of extracted hydrophobic organic compounds was compared to determine the efficiency of various internal sizing programs. Paper samples of identical weight were extracted with chloroform using an Accelerated Solvent Extractor (ASE100, Thermo Fisher Scientific; Waltham, MA, USA). The extraction was conducted at 100°C for 1 h. After evaporation of the solvent, residues were weighed to determine the amount of hydrophobic organic compounds in each sample.

Sample	Grammage, g/m²	Caliper, mils	Density, grammage/caliper	HST, s	Extracted Hydrophobic Material
Sample N	306	17.9	17.1	896	1.12 eq.
Sample S	333	18.0	18.5	842	1.00 eq.

I. Characterization of samples from mill N and S (HST = Hercules size test).

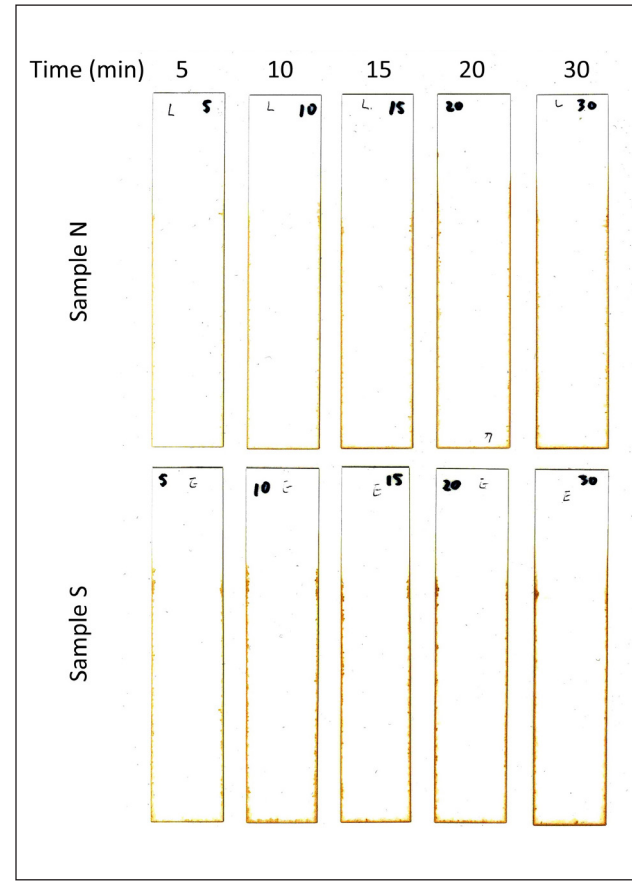
Scanning electron microscope (SEM) images were collected with a Cryo microtome without embedding medium to maintain the void spacing between fibers. Optical microscope images were collected on a Leitz Ergolux microscope with Olympus Stream Essentials software (Olympus; Tokyo, Japan). Fiber dimensions were determined with an OpTest Fiber Quality Analyzer (FQA) (OpTest Equipment; Hawkesbury, ON, Canada) via standard methods.

RESULTS AND DISCUSSION

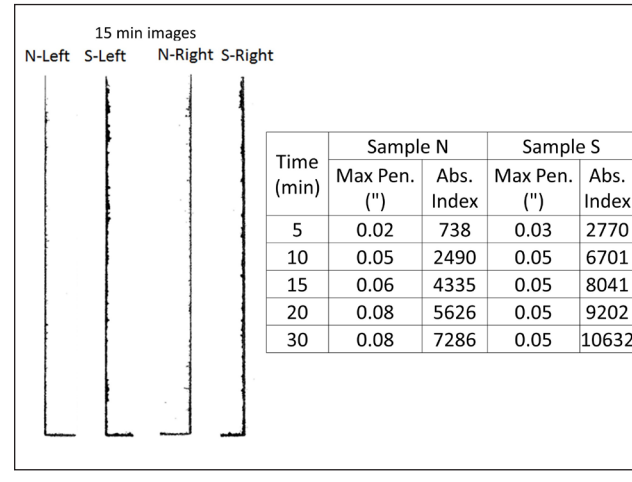
Paper samples of the same manufacturing grade were collected from two North American mills with significantly different fiber species. Both mills used essentially the same internal sizing program and other wet end additive programs. Samples from both mills met the same product specifications for a specific grade of cup stock. Both mills were integrated pulp and paper facilities that used local wood species to make pulp for papermaking. The wood species employed in pulp production was one of the major differences between the two sets of paper samples obtained from these two mills.

The physical and internal sizing characteristics of both sets of samples were determined. The results are presented in **Table I**. The two groups of cup stock had similar grammage, caliper, and bulk density. Sample N had a slightly longer HST time than that of sample S. The solvent extraction test showed that sample N contained slightly more hydrophobic material than sample S, which could explain the longer HST time of sample N. In general, internal sizing levels of the two groups of samples were approximately the same.

The coffee edge-wicking test was performed on both paper samples. Images of the tested samples are shown in **Fig. 2**. Visually, sample S had more edge-wicking. For quantitative analysis, samples from both sets were scanned to collect digital images for image analysis. Maximum penetration depth, absorption amount (abs. index) and a working binary image sample of 15 min samples were given in **Fig. 3**. The two groups of samples had similar maximum penetration depth at a certain testing time. Sample S had a much higher coffee absorption amount (total dark area calculated through image analysis) than sample N across all testing time ranges. The data suggested that the visual edge-wicking difference was due to a higher amount of adsorbed coffee rather than the penetration depth. Even though internal sizing levels were similar for the two sets of samples (based on HST value), the edge-wicking behavior was quite different. The HST test, a perpendicular penetration test, did not predict edge-wicking (in-plane

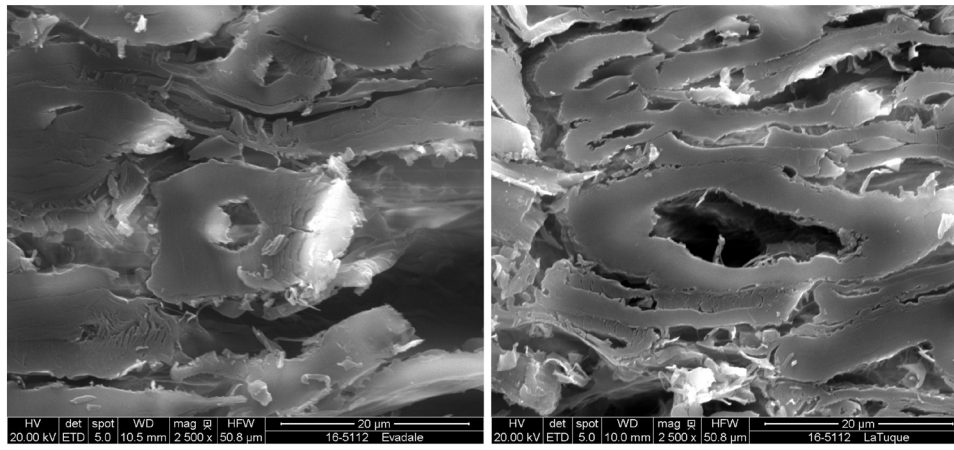


2. Optical images of tested samples from coffee edge-wicking test.



Time (min)	Sample N		Sample S	
	Max Pen. (")	Abs. Index	Max Pen. (")	Abs. Index
5	0.02	738	0.03	2770
10	0.05	2490	0.05	6701
15	0.06	4335	0.05	8041
20	0.08	5626	0.05	9202
30	0.08	7286	0.05	10632

3. Sample working binary image of 15 min samples and image analysis results of the samples.



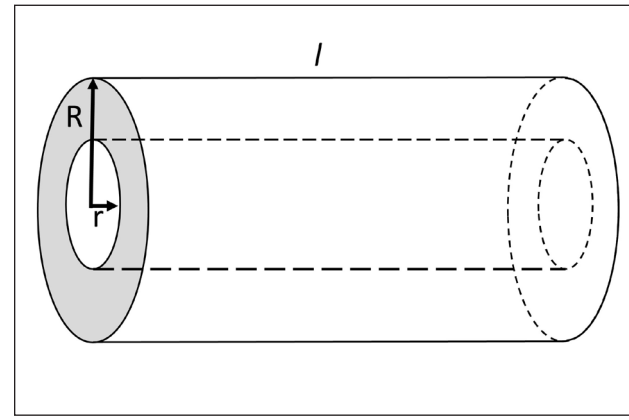
4. Cross-section images of fibers in sample S and N.

penetration) very well in the case of highly-sized paper grades. The results suggest that edge-wicking of highly-sized paper is affected by something other than internal size levels.

As previously mentioned, the fiber source (wood species) was a major difference between the two groups of samples. To assess the fiber structure of these sheets, cross-section SEM images were collected from both sample groups. Fiber cross-section images, **Fig. 4**, showed different fiber characteristics in the two types of paperboard. Fibers in paper sample S had thicker fiber walls, and most of the fiber lumens remained open after pressing and drying on the paper machine. Paper sample N had fibers with thin fiber walls, and those fibers were more flexible and collapsible than the thick-walled fibers found in sample S. Most of the fibers in sample N collapsed during the papermaking process, which resulted in relatively closed lumens.

Though clear structural differences were highlighted in the SEM images, the number of observed fibers was limited due to the complexity of SEM analysis. To confirm the fiber structural difference between the two types of samples, the samples were repulped and subjected to FQA testing. The average fiber wall thickness for each set of samples was estimated based on FQA data. Considerably more fibers were measured with the FQA compared to the SEM fiber measurements, which provided a more representative analysis of the structural differences in the fibers from sample S versus sample N.

The fiber structure can be simplified by assuming the fibers exist as straight tubes, **Fig. 5**, and the mass of a fiber



5. Scheme of simplified fiber structure.

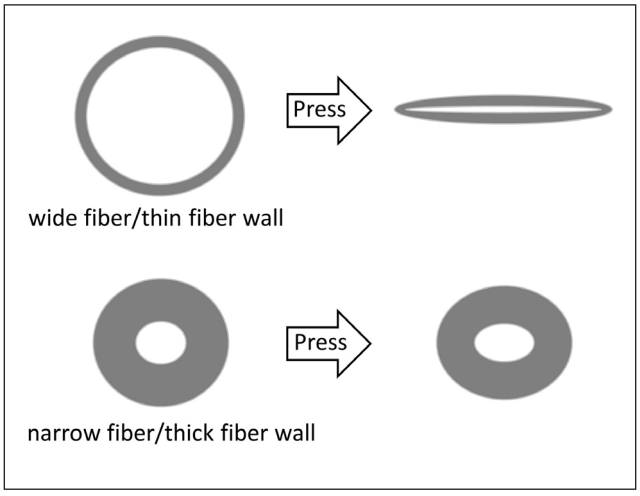
can then be expressed by Eq. 2, where R (µm) is radius of fiber, r (µm) is radius of lumen, ρ (g/cm³) is cellulose density in the fiber wall, l (mm) is fiber length, and C (mg/m) is coarseness (mass of fiber with unit length):

$$(\pi R^2 - \pi r^2) \times \rho \times l = C \times l \quad (2)$$

The fiber radius (R) and coarseness (C) can be obtained from FQA testing, and a cellulose density of 1.5 g/cm³ can be assumed; one can then solve the equation for lumen radius (r), with results shown in **Table II**. Fiber wall thickness is the difference between the fiber radius (R) and lumen radius (r). Based on this analysis, fibers from mill S have thicker walls

Fiber Source	*R*, µm	Coarseness, mg/m	*r*, µm	Fiber Wall Thickness, µm	(*R*-*r*)/*R*
Mill N	12.835	0.111	11.882	0.95	0.07
Mill S	9.235	0.111	7.857	1.38	0.15

III. Fiber structure parameters based on fiber quality analyzer (FQA).



6. Scheme of fiber's cross-section and its ability to collapse.

(1.38 μm) compared to fibers from mill N (0.95 μm), which is in agreement with the SEM analysis.

If the cellulose material of different fibers has a similar Young's modulus, a wider fiber with thinner fiber walls would be easier to collapse during pressing and drying, as depicted in **Fig. 6**. The ratio between fiber wall thickness (*R-r*) and fiber radius (*R*) can be used as a parameter to evaluate a fiber's ability to collapse. The smaller the value of this ratio, the easier for a fiber to collapse. The value of (*R-r*)/*R* for fibers from both mills is also shown in Table II. The fibers of mill N have about half the value of (*R-r*)/*R* versus the fibers of mill S; therefore, fibers of mill N are more easily collapsed and would tend to have closed lumens because of the fiber structure. This characterization is also consistent with the SEM observations.

Different fiber characteristics lead to different sheet structure as well. Cross-section SEM images of paper sample S and N are shown in **Fig. 7**, in which the white area is fibers and the black area is the voids between the fibers. It is clear that sample S has more fiber cross-sectional area than sample N.

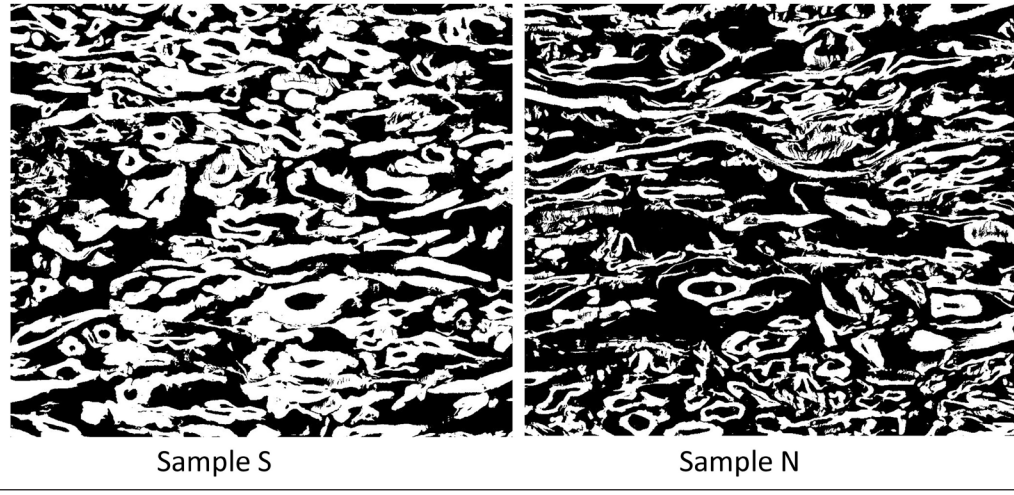
[Creamer] in HST Ink, v/v%	HST, s
5	>600
10	>600
20	>600

III. Hercules size test (HST) time with different amounts of coffee creamer in the ink.

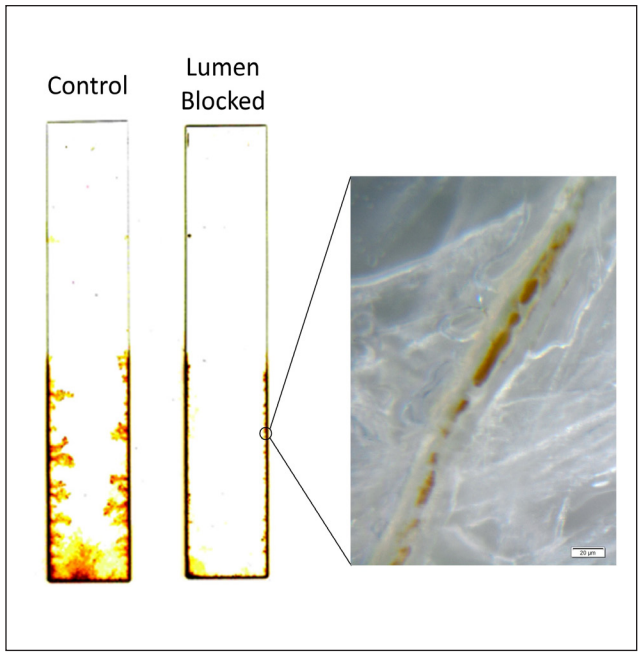
When cut (in the converting process, for example), the raw edge of sample S would have a significantly larger exposed hydrophilic fiber wall and lumen area with no sizing protection compared to sample N. Therefore, an aqueous solution would tend to edge-wick considerably more in sample S than sample N.

The creamer used in the hot coffee test is known to contain a certain amount of surfactant, which reduces liquid surface tension and makes coffee easier to wet heavily-sized fiber surfaces. To check whether creamer dosage in the edge-wicking test helped wet-sized fiber surfaces, we ran the HST test with additional creamer in the ink on sample S. The results are listed in **Table III**. The data indicate that the critical creamer concentration was between 20 v/v% and 30 v/v%. When creamer concentration was below 20 v/v%, internal size material was sufficient to lower the fiber surface energy enough to protect against wetting, which minimized liquid transport through the voids between the fibers. The coffee penetration into voids (black area in Fig. 7) was limited during the edge-wicking test with 3.6 v/v% creamer because the surfactant concentration was less than the critical concentration; therefore, new exposed fiber wall area and lumens (white area in Fig. 7) were likely the major pathway for liquid to wick into the paperboard.

A lumen "blocking" test was done by making handsheets with Bersize AEW (Bercen; Denham Springs, LA, USA), a



7. Sheet cross-section images of sample S and N.



8. Handsheet coffee edge-wicking samples. Note trapped coffee within a single fiber in the photo at right.

polymer dispersion with very small particle size that can enter and size the fiber lumens. To test the hypothesis that fiber wall area and lumens are the path for coffee edge-wicking in highly-sized paperboard, pulps from mill S with the same internal size dosage were used in the handsheet study. The hot coffee test was used to compare handsheets made with and without the blocking agent; the use of the blocking agent significantly reduced edge-wicking, as shown in **Fig. 8** (left). Further microscopic observations of the sample with the lumen blocking agent found that coffee was trapped in some fibers. The image in Fig. 8 (right) clearly showed that the fiber walls and lumens were a major pathway for edge-wicking in highly-sized paper.

CONCLUSIONS

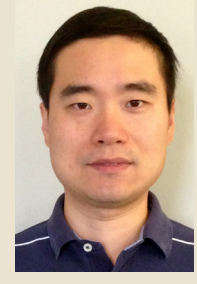
An internal sizing program, as typically applied under mill operating conditions, protects the fiber's outer surface to reduce liquid penetration between fibers. Such a sizing program typically reduces liquid penetration perpendicular to the surface of the paperboard. At low size levels, internal size is also an efficient way to reduce edge-wicking. At high size levels, the fiber's outer surface is well protected and liquid transport through voids between fibers is minimized; however, fiber cross-sectional area at cut edges is not protected by sizing material. The raw, cut fiber edges become an entry point, which results in increased edge-wicking. Transport of liquid through the fiber wall and lumen has been demonstrated to be a major pathway for edge-wicking in highly-sized paper. When internal size level is high enough to cover all the fiber surfaces, the HST test shows lower correlation with edge-wicking behavior than at lower size levels. In addition, further increases in the internal size level offer limited benefit towards the mitigation of edge-wicking in highly-sized paper. In this case, some new sizing strategies are desired for high performance liquid packaging grades, once a traditional internal sizing program provides full coverage of the surface of the fibers. In addition to new sizing technology, it may be possible to address the issue, at least in part, through either enzymatic fiber modification or decreasing the void volume by addressing the fiber cell wall. **TJ**

ABOUT THE AUTHORS

Edge-wick is an aesthetic and functional performance feature important to cup producers. Improving our understanding of the edge-wicking phenomenon can assist in the development of improved methods to control edge-wicking in our products. Our efforts here have developed an improved understanding of the cause of edge-wicking.

Developing a theory that explained the edge-wicking performance of similar products produced at multiple mills was difficult. An interesting finding was that similar amounts of material wicking into the cut edge of the sheet could result in a passing test for one sheet structure, while another structure wicking a similar amount of material would fail the test. Surprisingly, the role of fiber structure upon edge-wicking was found to be greater than expected.

Hopefully, this work will lead to suppliers and papermakers developing enhanced methods to control edge-wicking. The next step is to share our enhanced understanding of edge-wick with suppliers to help develop improved solution methods.



Xu

Meyers

Hart

Xu is senior research chemist and Hart is director, Fiber Science and Innovation, for WestRock, Richmond, VA, USA. Meyers is assistant papermill superintendent, WestRock, Evadale, TX, USA. Email Hart at peter.hart@westrock.com.

ACKNOWLEDGEMENTS

The efforts of John Dejarnette (senior R&D researcher, WestRock), Nichole Kilgore (R&D researcher, WestRock) and Adele Panek (R&D researcher, WestRock) are appreciated for sample characterization, handsheet making, and FQA testing. The authors also appreciate support from the WestRock R&D Analytical Group, led by Paul Giusti (R&D manager, WestRock). SEM analysis was conducted by William Mallory (lead R&D researcher, WestRock). Solvent extraction analysis was conducted by Vitaly Khazan (R&D scientist/engineer, WestRock).

Bersize AEW was kindly provided by Bercen to support this research.

LITERATURE CITED

1. Salminen, P., "Studies of water transport in paper during short contact times," Ph.D. thesis, Åbo Akademi University, Turku, Finland, 1988.
2. Swanson, J.W., *The Sizing of Paper* (W.F. Reynolds, Ed.) 2nd edn., TAPPI Press, Atlanta GA, USA, 1989, p. 133.
3. Eklund, D. and Lindström, T., *Paper Chemistry: An Introduction*, DT Paper Science Publications, Grankulla, Finland, 1991, p. 192.
4. Washburn, E.W., *Phys. Rev.* 17(3): 273(1921).
5. Lucas, R., "Ueber das Zeitgesetz des Kapillaren Aufstiegs von Flüssigkeiten," *Kolloid-Z.* 23: 15(1918).
6. Hintz, H.L. and Webb, J.T., U.S. pat. 3,849,224 (Nov. 19, 1974).
7. Floyd, J.M., U.S. pat. 4,859,244 (Aug. 22, 1989).
8. Kern, N.T., U.S. pat. 5,308,441 (May 3, 1994).
9. Camden, R. and Claytor, P., U.S. pat. 5,766,732 (June 16, 1998).
10. Larsson, J., Aksberg, R., and Tufvesson, H., U.S. pat. 2012/0114959 A1 (May 10, 2012).
11. Propst, C.W. and Jones, J.C., U.S. pat. 8,333,872 B2 (Dec. 18, 2012).
12. Theisen, J.T., Unruh, B.C., Feit, S.L., et al., U.S. pat. 8,337,919 B2 (Dec. 25, 2012).
13. Meyers, J., U.S. pat. 20160186381 (June 30, 2016).
14. Verhoeff, J., Hart, J.A., and Gallay, W., *Pulp Pap. Mag. Can.* 64(12): P509(1963).
15. Van den Akker, J.A. and Windle, W.A., *Tappi* 52(12): 2406(1969).
16. Bristow, J.A., *Sven. Papperstidn.* 74(20): 645(1971).
17. Bristow, J.A., *Sven. Papperstidn.* 75(21): 847(1972).
18. Wedin, P., Senden, T., and Knackstedt, M., *Int. Pap. Coat. Chem. Symp., 6th*, SPCI, Stockholm, Sweden, 2006, Book of Abstracts, p. 127.