Effects of Sugar Addition Before Drying on the Wet-Flexibility of Redispersed Kraft Fibers

Min Zhang, Martin A. Hubbe, Richard A. Venditti, and John A. Heitmann

Department of Wood and Paper Science
North Carolina State University
Raleigh, NC 27695-8005

ABSTRACT

Paper made from recycled, chemically pulped fibers typically has lower strength than paper made from virgin fibers. This was confirmed with respect to tensile and compression strength for an unbleached softwood kraft pulp. It was also confirmed that the addition of sucrose, at high concentration, to virgin pulp before drying could improve the recycled paper strength, compared to a control with no sugar added. The use of glucose was found to be slightly more effective than sucrose. Fibers treated with the sugars were found to have higher flexibility and water retention values than untreated fibers that had been subjected to the same drying conditions.

INTRODUCTION

Since the 1960's there has been increasing interest in factors responsible for the loss of inter-fiber bonding ability when chemical pulps, such as kraft or sulfite, are dried and then resuspended in water [1-7]. One explanation for this behavior is that the fibers and fines become less able to swell in water [1,2, 8-10], a phenomenon often attributed to irreversible closure of micropores in the fiber cell wall [10-13]. Other investigators have considered changes in the wet flexibility of fibers [8,11,13]. Fibers with higher local conformability at their surfaces would be expected to form less relative bonded area under fixed conditions of wet pressing and drying [15-16]. Recently it has been proposed that some or all of the observed loss in bonding ability is due to chemical factors at the fiber surfaces [17], possibly involving changes in the ability of surface-bound polymer chains to inter-diffuse [18].

In 1963, Higgins and McKenzie [1] found that a high concentration of sucrose (20% solution by weight) present during drying could reduce the loss of bonding ability when kraft or neutral sulfite pulps were dried. They attributed this effect to the action of sucrose in forming reversible hydrogen bonds with fibers during the drying process. It was proposed that such bonds could be more readily broken on re-wetting than the intra-fiber bonds formed during drying. At that time the concept of fiber pores as described by Stone and Scallan [10,19], using a solute exclusion method, had not yet been published. Modern concepts of cell wall architecture and fiber swelling are to a great extent founded on the results obtained using this technique [20].

Given the known nano-scale pore size distribution of never-dried, unbleached kraft fibers [19,21-22], relative to the even smaller size of monomeric sugar molecules [19], it is reasonable to expect the sugar molecules to enter the cell wall with a solution and remain there during the evaporative drying of paper. Due to the high water-solubilities of sugars and their lack of ionic charge it has been assumed, to a first approximation, that such molecules are neither enriched nor depleted at the wetted fiber surfaces, relative to their bulk concentration [10]. Such an assumption implies that the concentration of sugar in solution that enters the fiber wall ought to be similar to the bulk concentration. The low volatility of sugars, including glucose and sucrose, ensures that the molecules are left behind when water evaporates. Extending the surface tension mechanism proposed by Campbell [23], the receding films of water within fiber pores during the drying of paper are expected to cause pores in the fiber wall to collapse [24]. Sugar remaining within collapsed pores would be expected to form hydrogen bonds with the adjacent cellulosic materials [23]. The important question then is how the presence of monomeric sugars would affect reopening of the pores when the fibers are reimmersed in water. Despite their chemical similarity with the monomeric sugars, only the cellulose molecules of the cell wall have a known ability to form insoluble crystalline zones due to cooperative hydrogen bonding along their highly regular molecular chains [25-28]. By contrast, monomeric sugars lack the size and regularity required to form insoluble crystallites. The importance of such mechanisms is underscored by observations that paper properties can be affected by surface derivatization procedures [29,30]. Such derivitization tends to interrupt the structure of cellulose chains.

Fiber flexibility tests have the potential to provide additional evidence to test the hypothesis that sugars modify the process of fiber-wall pore collapse or make such pores more likely to reopen upon rewetting. A fiber with closed pores is expected to have a more conformable surface, compared to one in which the pores remain more open [31,32]. As an approximation it will be assumed here that fiber flexibility results can be used as an indication of changes in local conformability. Many techniques have been devised to measure wet fiber flexibility [16,33-39]. Among these, the method of Steadman and Luner [36] appeared especially well suited to the needs of the present study, due to the relative simplicity of the measurement and the fact that the technique measures the wet flexibility of a relatively large number of fibers more quickly and reproducibly compared with other techniques. Steadman and Luner [36] used this technique to find that there is a linear, but not unique, relationship between sheet apparent density and average wet fiber flexibility for a number of pulps and pulp treatments. Paavilainen and Luner [39] also used the technique to show that at least half of the wet fiber flexibility value was lost on air-drying. The goal of the experiments to be described herein was to determine whether the addition of monomeric sugar molecules to never-dried kraft fibers has a significant effect on the wet flexibility after those same fibers have been dried and then resuspended in water.

EXPERIMENTAL

Materials

Pulp: Never-dried, unbleached pine kraft pulp was obtained from the Mansfield, Louisiana mill of International Paper. The sample was taken from the high-density storage, after the pulp had passed through the blowline and de-shive refiners. Upon its receipt from the mill, the pulp was screened and centrifuged to 30% consistency. The fibers were fluffed and then refrigerated during storage. These procedures made it possible to carry out experiments from a highly consistent master batch of fibers. The unrefined pulp had a freeness of approximately 760 ml CSF.

Chemicals: Reagent-grade sucrose (Fisher, Lot.701806) and anhydrous α–D-glucose (Aldrich, catalog number 158968) were used without further purification.

Equipment: Microscopic observations and digital images were obtained with an Olympus BH2-UMA microscope with Sony 3CCD Color Video Camera (Model: DXC-970MD). The images were captured and evaluated using Image-Pro Plus software (Version 4.0, Media Cybermetrics). Micro glass slides (75mm x 50mm x 1mm, Aldrich catalog number Z162698) and stainless steel wire (25.4 µm, Cybermetrics) were used to prepare wired slides for the fiber flexibility tests.

Procedures

Although the procedure for refining pulp and handsheet making were described earlier [40], the detailed procedures for them have been reproduced here because the procedure was modified slightly.

Refining by Valley Beater: Sub-batches of centrifuged pulp were taken from cold storage and refined with a laboratory Hollander-type beater (Valley Machinery, Inc.), following the procedure of TAPPI method T-200. The pulp was dispersed in the beater for 30 minutes (zero load) then refined for 20 minutes. The pulp then was screened, centrifuged to approximately 30% consistency, fluffed, and then refrigerated again until use. The freeness of the pulp refined in this way before making handsheets was approximately 700 ml CSF.

Handsheets: Before making handsheets, the refined pulp was dispersed in a disintegrator for 5 minutes. As shown in Fig. 1, the refined pulp was added to either a sugar solution or to dilution water (for control tests). The final concentration of sugar solutions was 2% dry mass on dry mass. The pulp samples were mixed with chemicals for 12 hours with stirring before a pad of fibers was formed on a Büchner apparatus with vacuum applied. In each case, ten pads were required and each pad weighed around 2.4 grams (O.D.). The pads were dried in a TAPPI standard conditioning room (50.0% ± 2.0% RH and 23.0 ± 1.0ºC) for at least 12 hours before physical properties were tested. For making recycled handsheets, the procedure of TAPPI Standard Method T-205 was used, with the following modifications. Instead of the standard slurry pulp consistency 0.3%, a 0.6% consistency was used. As a result the basis weight of the handsheets was 120 g/m^2, not 60 g/m^2. At the higher basis weight the samples are not expected to fail in a "buckling" mode during STFI compression strength tests [41,42]. Instead, they are expected to fail by the desired shear/internal bond failure.

Water Retention Value (WRV): The water retention value (WRV) was used as a measure of the internal fiber swelling capacity of the pulp [43]. The procedure followed TAPPI test UM 256 with some modifications. Prior to measurement, the pulp was dispersed in a disintegrator for 5 minutes (15,000 revolutions), thoroughly washed in a large excess of de-ionized water, and allowed to stand in water overnight. Following further washing, the pulp was collected on a vacuum filter and dewatered to 25% solids. Moist samples of pulp (equivalent to 0.16 g dry mass) were placed in sintered centrifuge tubes (pore size 0.22 μm, volume 3 ml, provided by MSI). Samples were centrifuged at 900 g for 30 minutes according to TAPPI UM 256. After centrifugation, the moisture content of the samples was determined by weighing immediately and also after drying at 105 °C for 2 hours and cooling in a desiccator jar (anhydrous calcium sulfate) for 30 minutes.

Refining by PFI Mill: As shown in Fig. 2, a PFI mill was used to refine the relatively small batches of pulps needed for flexibility analysis. Thirty grams of pulp were taken from cold storage and refined with a PFI mill (9000 revolutions). The procedure for refining pulp by PFI followed TAPPI test T248cm-85. The refined pulp (10% consistency) was put in a zip-lock plastic bag and stored in a cold room for future use. These conditions yielded freeness values of approximately 420 ml CSF.

Fiber Flexibility: In the fiber flexibility test a sparse layer of fibers, mainly lying separate from each other, was formed on the wire of a standard sheet machine. Around 5 ml of pulp slurry (1% consistency) was used to make the fiber layer. The layer was then couched onto a #4 Whatman filter paper using standard sheet technique, corresponding to TAPPI Method T-205. A glass slide having several thin stainless steel wires (diameter=25 μm) wrapped across its surface in a parallel pattern was laid onto the filter paper. The filter paper and slide were then pressed against each other between two sets of water-saturated blotters in a regular handsheet press. After a two-minute pressing at 50 psi, the glass slides were separated from the blotters and filter paper and viewed in a microscope under incident light. The areas of intimate contact between the fibers and the glass appeared dark, and the area that is not in contact, for instance as the fibers cross over the wire, was less visible. The explanation of the incident light image is based on the theory of thin film interference [44]. It makes sense that the no-contact length is related to the flexibility of the fiber, and as the fiber becomes more flexible this distance will decrease. The detailed procedure for preparing slides appears in the literature [36]. The image analysis software was used to measure the width and less-visible length of the fibers crossing over the wires. Around 100 fibers were chosen for a standard set of flexibility tests.

Preparation of Samples for Fiber Flexibility Determination: In the study of the effects of drying and wet pressing on fiber flexibility of refined pulp, four portions (2.4 grams oven dry each) of the refined pulp were taken from cold storage. Each portion was soaked in deionized water for 2 hours before being dispersed in a disintegrator for 5 minutes. After soaking, different procedures were adopted for specific experiments.

Effects of drying and wet pressing: Further steps in the procedure for determining the effect of drying and wet pressing on fiber flexibility are outlined in Fig. 2. In the case of control experiments the fiber slurry was washed in a large excess of deionized water before standing in deionized water for 4 hours before measurement of fiber flexibility.

To study the effect of wet pressing, fibers soaked in the same way were formed into handsheets, wet pressed using the standard TAPPI pressing method, and then dispersed immediately in a TAPPI disintegrator for 5 minutes. This was followed by another washing in a large excess of deionized water. The washed fiber slurry then was kept in deionized water for an additional 4 hours before performing a fiber flexibility test.

To study effects of oven drying, handsheets were made from dispersed fiber suspensions and dried in an oven for 10 minutes at 105 °C before being put in a TAPPI standard conditioning room for 12 hours. The handsheet was soaked in deionized water for 6 hours before being dispersed in a disintegrator for 5 minutes. Subsequent soaking and flexibility measurement steps were the same as given above. The procedure in the case of air-drying was identical, except that the handsheets were not oven-dried but were put directly in a TAPPI standard conditioning room for 12 hours.

Effects of sugars: To determine the effect of sugars on the flexibility of refined fiber, four 1.2 g amounts (based on O.D.) of the refined pulp were taken from cold storage. The pulp samples were soaked in deionized water for 2 hours before being dispersed in a disintegrator for 5 minutes individually. After that, different procedures were adopted for specific experiments.

In the case of control experiments the fiber slurry was left standing in deionized water for 12 hours before making a fiber pad on a filter paper (Whatman #4). The fiber pad was dried in a TAPPI standard conditioning room for 12 hours, and then soaked in deionized water for at least 4 hours before being dispersed in a disintegrator for 5 minutes. The slurry was washed in a large excess of deionized water before the fiber flexibility was tested. The water retention value (WRV) was also measured.

To study effects of sucrose and glucose the fiber slurry was combined with a sugar solution and deionized water in order to produce a total volume of one liter having either 2% or 10% sugar by weight in water. The mixture was allowed to soak at room temperature for 12 hours before making a fiber pad on a filter paper (Whatman #4). After that, the procedure was the same as that of the control experiment.

RESULTS AND DISCUSSION

It is well known that the tensile strength and STFI compression strength of recycled handsheets made from unbleached softwood kraft pulp are reduced relative to the corresponding primary handsheets made from virgin fibers. This was confirmed in a recent study of unbleached kraft pulp, which also showed a corresponding decrease of the water retention value [40]. According to other results [10], there is irreversible pore closure during recycling, and this is assumed to contribute to the observed strength losses.

Effects of Sugars on Paper Strength

As shown in Fig. 3, adding sucrose or glucose to virgin pulp before the initial drying increased the tensile strength and compression strength of recycled handsheets, compared to the control experiments. A t-test showed that the increases in breaking length, STFI compression and WRV were significant at a 95% confidence level for glucose-treated fiber relative to the control. The relative improvement in strength upon the addition of the sugars before drying is in agreement with the previous findings of Higgins and McKenzie [1]. Further evidence supporting the assumed mechanism of strength loss (pore closure causing increased fiber stiffness) was obtained from the water retention value (WRV) tests. From Fig. 3, the WRV of fibers treated with sugars increased compared to the untreated recycled fibers. That is consistent with the explanation that sugars blocked irreversible closure of some pores.

According to Fig. 3, glucose had a slightly larger effect on breaking length and compression strength, compared to sucrose. Since the molecular weight of glucose is smaller than that of sucrose, glucose has a better chance to enter smaller pores than sucrose does. This may be one reason why glucose had a higher effect on paper strength than sucrose did. However, the present data are not sufficient to rule out other plausible explanations. For example, the structure of sucrose may be sufficiently different from the cellulose chains as to make it less able to adsorb onto fiber surfaces and affect inter-fiber bonding, compared to glucose.

Effect of Drying on Fiber Flexibility

One explanation for reduced bonding ability of recycled kraft pulps has been that pore closure stiffens the fiber, decreases its conformability, and thus results in less fiber-to-fiber bonded area during papermaking [8]. Others have speculated that crystallization also plays a role in this fiber hardening [10,45]. Fiber flexibility tests were carried out to determine whether fiber flexibility correlates with the increased strength and water retention value promoted by sugars.

Though the procedures used for fiber flexibility analysis herein followed the main steps given by Steadman and Luner [36], there were some differences with respect to the calculations. To summarize, the flexibility can be expressed as follows,

$$Fiber\,Flexibility = \frac{1}{EI} = \frac{72d}{PWS^4} \tag{1}$$

where E is the modulus of elasticity (Nm^{-2}), I is the moment of inertia (m^4), d is the wire diameter (m), P is the applied pressure (Pa), W is the projected fiber width (m), and S is a mathematical estimate of the loaded fiber span (m). A correct form to calculate the loaded fiber span (S) based on the Pythagorean Rule is given by Cresson [46]:

$$S = \sqrt{d^2 + \left(\frac{L}{2}\right)^2} \qquad (2)$$

where L is the less-visible length of fiber, as mentioned earlier.

A detailed derivation of equation (1) can be found in the paper of Steadman and Luner [36]. In the present study the geometric mean was used to represent the fiber flexibility of samples instead of the median used by others [35,36]. This modification of the original approach was justified by the fact that in many cases the maximum values were 1000 times the minimum values. According to Dickey [47] data that is spread over such a wide range is best evaluated by using logarithmic plots and by calculation of geometric means of the logarithms rather than median values. Statistical modeling can be carried out on the log scale to test hypotheses, whereas the distributional results and inferences for medians are not so well developed. For instance, a t-test can be used when determining the effect of sugars on fiber flexibility as long as the distribution of data on the log scale is reasonably close to a normal distribution.

To provide context for experiments involving sugar addition, it is worth first considering what happens to fiber flexibility due to drying in the absence of chemical addition. Fig. 4 shows that wet pressing and drying reduced the flexibility of fibers and that oven drying had the most significant effect on the reduction of fiber flexibility. The limit bars in the figure represent 95% confidence intervals for the mean. The only difference in processing conditions between fibers represented in the two left-most histogram bars was the application of standard pressing, as in TAPPI Method T205. Though effects of wet-pressing on fiber properties have been reported by others [48], such effects were not found to be statistically significant in the present study of fiber flexibility. Since the fibers represented in the two right-most bars also had experienced the same wet-pressing operation, it can be argued that they should be compared relative to the “wet-pressing” bar to judge the effect of drying conditions. Only the oven-drying treatment had a significant effect relative to this control at the 95% level of confidence.

Fig. 5 shows that the flexibility of unrefined fibers had the same ranking according to the drying procedure as that of refined fibers but that refined fibers were more flexible than unrefined fibers. The freeness of the unrefined fibers was approximately 760 ml, whereas the refined fibers were approximately 420 ml CSF. The results of these flexibility tests are in agreement with the well-known fact that paper made from refined fibers has higher relative bonded area and higher paper strength than unrefined fibers. However it is worth noting, in the case of unrefined fibers, that oven-drying had the largest relative effect in terms of the logarithmic scale used for the analysis.

Effect of Sugars on Fiber Flexibility

Results in Fig. 6 show that refined fibers treated by sugars before drying were more flexible after drying and reslurrying than untreated refined fibers subjected to the same processing conditions. The first histogram bar labeled “control” in this case represents a condition of room temperature drying of fibers collected as a mat on a Büchner apparatus (no pressing). The remaining bars represent the same drying conditions, except that the vacuum-dewatered fiber mats remained permeated with part of the sugar solutions in which they had been suspended. The presence of 10% glucose or sucrose solution at the start of the drying process had a significant effect on fiber flexibility, compared to the control experiment, at a 95% confidence level. The effect of the 2% sugar solutions was not as evident. The fact that a large concentration of sugar was required to achieve a significant effect agreed with previous work that considered other aspects such as water retention values and inter-fiber bonding [1,40]. The same fact also is consistent with the earlier assumption that the monomers did not preferentially adsorb to an appreciable extent at the fiber surfaces, relative to their concentration in the adjacent solution.

CONCLUSIONS:

The presence of sugars during drying of refined, never-dried kraft fibers improved the strength of the corresponding recycled handsheets, compared to similar samples tested in the absence of sugars. The sugar treatment also caused the water retention values of the recycled fibers to increase relative to an untreated control. For the first time it was shown that sugar-treated fibers showed higher wet-flexibility compared to untreated fibers after drying, extensive rinsing, and soaking. Observed increases in flexibility, WRV and paper strength upon sugar treatments were in agreement with the concept that conformability of fiber surfaces during sheet formation or during subsequent drying play a significant role in determining the strength of inter-fiber bonding.

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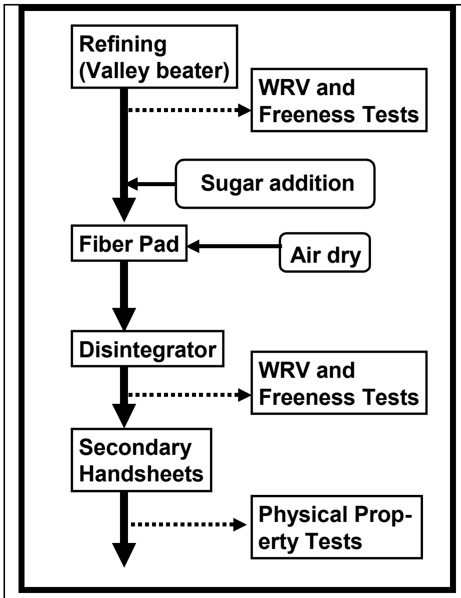

Fig. 1. Procedure for handsheets in which virgin pulp was dried in presence of concentrated sugar solution

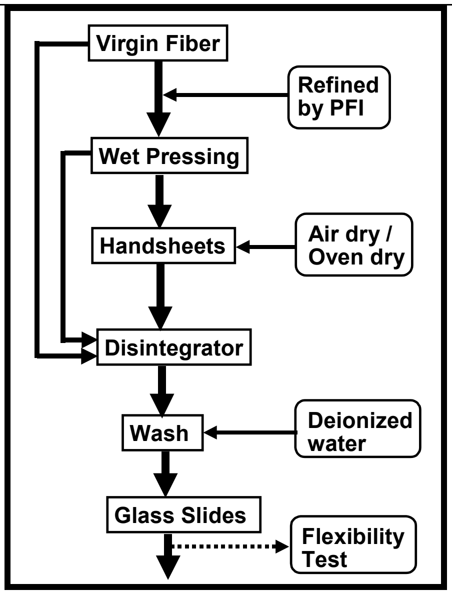

Fig. 2. Procedure for the effect of wet press and drying on fiber flexibility

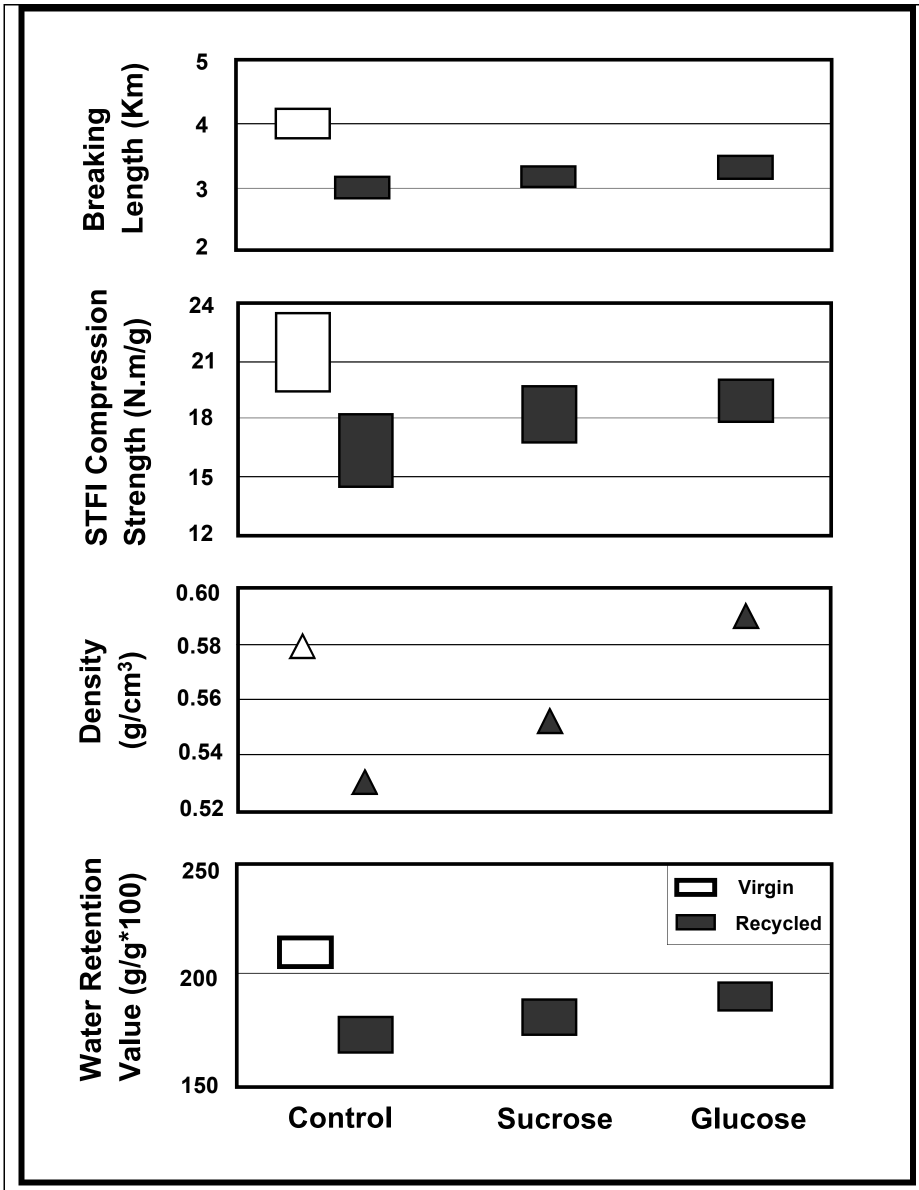

Fig. 3. Effect on fiber and paper properties of pretreatment with 2% sugar solutions, followed by drying and resuspension in water (vertical scale of rectangles shows 95% confidence intervals)

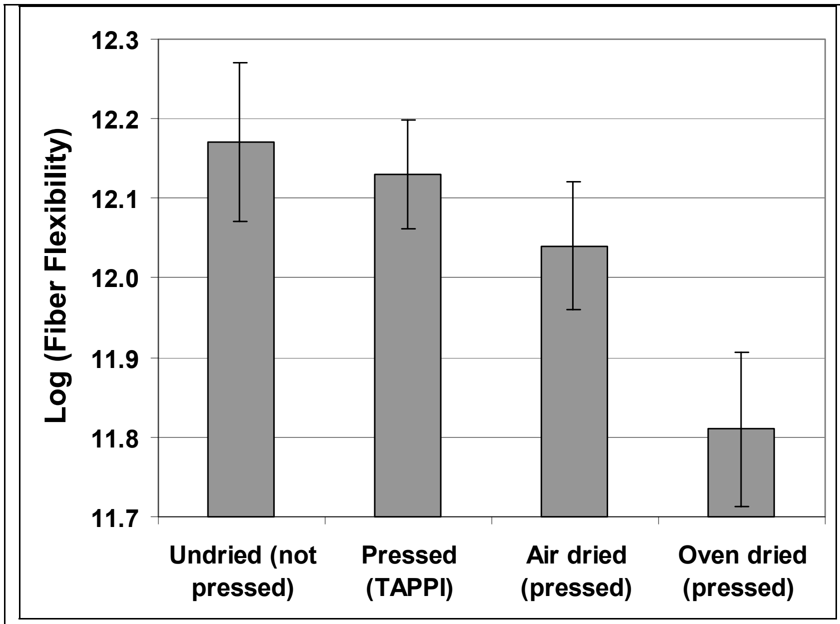

Fig. 4. Effect of wet pressing, with or without subsequent drying at either room temperature or 105 ^{o}C on the wet-flexibility of refined, soaked unbleached kraft fibers

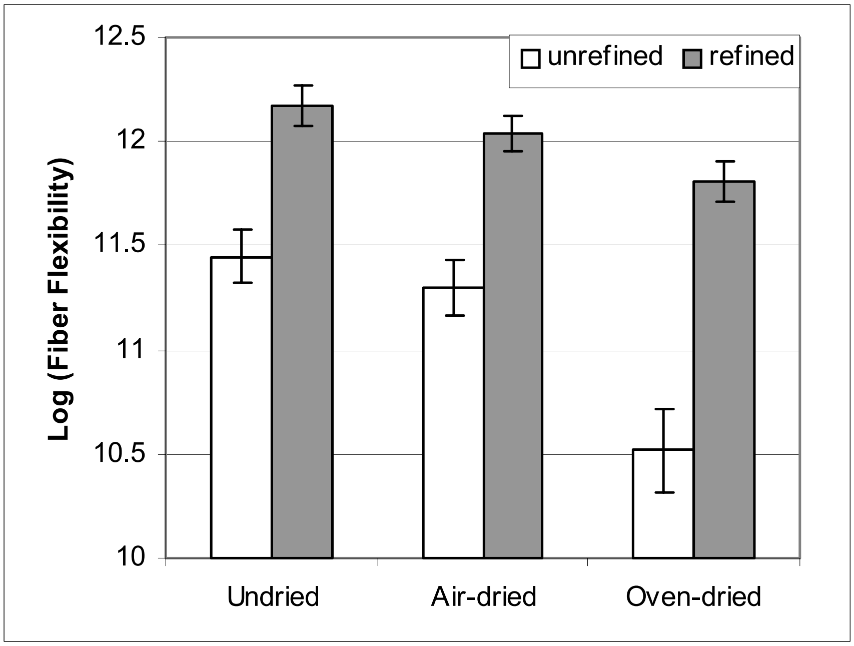

Fig. 5. Effect of refining on the flexibility of unbleached kraft fibers under the conditions of never-drying (control), air-drying at room temperature, or oven-drying at 105 ^{o}C for 10 minutes

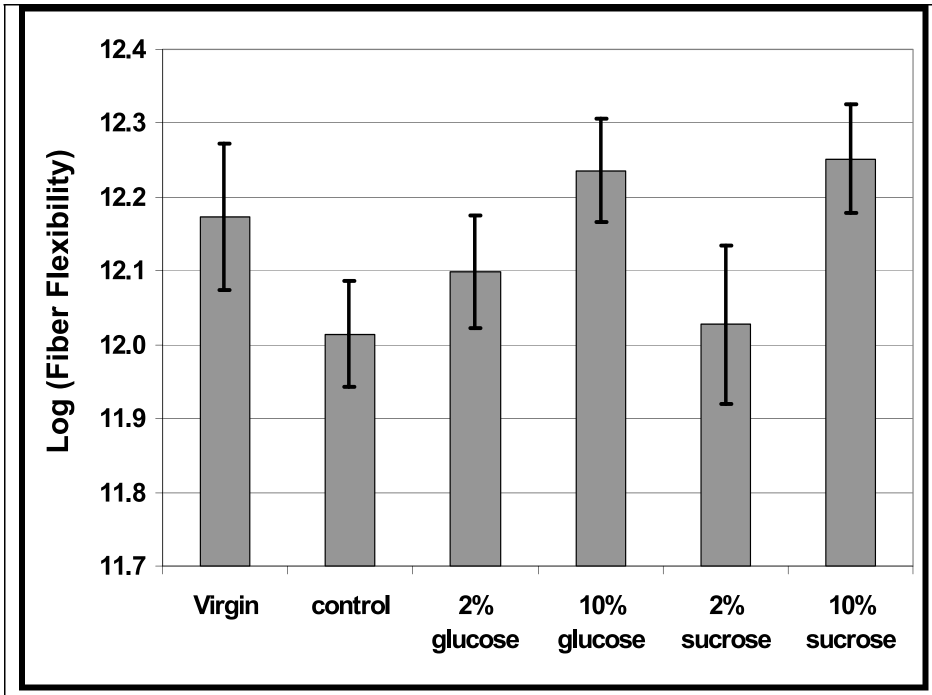

Fig. 6. Effect of various sugar treatments on the flexibility of unbleached kraft fibers dried at room temperature and then extensively washed and soaked in water

CAPTIONS:

Fig. 1. Procedure for handsheets in which virgin pulp was dried in presence of concentrated sugar solution

Fig. 2. Procedure for the effect of wet press and drying on fiber flexibility

Fig. 3. Effect on fiber and paper properties of pretreatment with 2% sugar solutions, followed by drying and resuspension in water (vertical scale of rectangles shows 95% confidence intervals)

Fig. 4. Effect of wet pressing, with or without subsequent drying at either room temperature or 105 ^{o}C on the wet-flexibility of refined, soaked unbleached kraft fibers

Fig. 5. Effect of refining on the flexibility of unbleached kraft fibers under the conditions of never-drying (control), air-drying at room temperature, or oven-drying at 105 ºC for 10 minutes

Fig. 6. Effect of various sugar treatments on the flexibility of unbleached kraft fibers dried at room temperature and then extensively washed and soaked in water