

Cellulosic ethanol byproducts as a bulking agent

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ABSTRACT: Financial enhancement of biomass value prior to pulping requires subsequent use of remaining materials; e.g., high value use of remaining stock material after cellulosic ethanol production would improve the economics for cellulosic ethanol. In this work, use of enzymatic hydrolysis residual solids (EHRS), a cellulosic ethanol byproduct, were investigated as a bulking agent. The study examined the effect across several grammages, 20 g/m²–200 g/m², and EHRS substitution levels, 10%, 15%, and 20%. EHRS retention was examined with light scattering. EHRS loading increased bulk while reducing tensile strength, compression strength, and stiffness.

Application: Byproducts of cellulosic ethanol production can be used as a bulking agent.

The pulp and paper industry has the necessary infrastructure to process large amounts of woody biomass, whose increased use is one component of an effort to reduce the prevalence and severity of U.S. forest fires. Additional revenue streams that utilize existing capability, perhaps with minor modifications, are attractive to the industry. One potential revenue stream is cellulosic biofuel such as bioethanol, a renewable fuel, from underutilized woody materials not suitable for pulp production.

A substantial amount of research has been conducted using woody biomass for biofuel production in the past two decades [1], which included the utilization of underutilized woody biomass like pine beetle killed lodgepole pine [2] and forest harvest residue [3]. In cellulosic biofuel production, woody materials undergo a chemical pretreatment step, similar to pulping and carried out in a pulp digester, and then are enzymatically hydrolyzed to sugars. The sugars are then fermented to biofuel such as bioethanol. Significant amounts of woody materials, or enzymatic hydrolysis residue solids (EHRS), remain after enzymatic hydrolysis. EHRS consist of lignin and some unhydrolyzed polysaccharides such as cellulose and hemicelluloses. The amount of polysaccharides in EHRS depends on the degree of enzymatic hydrolysis. Because biofuel is a commodity product with low economic value, an economically viable production of biofuel requires the utilization and valorization of EHRS. Combustion of EHRS is one option, as it contains a substantial amount of lignin (higher heating value than carbohydrates). However, using EHRS as a bulking material may be a better choice in terms of valorization and environmental impact.

Rising pulp cost is a constant concern within the paper industry, and researchers are investigating methods of increasing material bulk while maintaining properties. Here, we investigate the use of EHRS to increase paper bulk and examine changes in the resulting mechanical and optical properties.

Papermakers understand that an inverse relationship exists

between tensile strength and bulk. Some cellulosic material, such as microfibrillated cellulose, has been used to increase tensile strength [4–8] and reduce permeability but at the expense of increased drying energy. Special additives can be used to improve retention and drainage [5,7]. Sometimes, a significant amount of additive material (e.g., 5% by weight or greater) was needed before the effect was noticed [6].

However, processes that improve strength typically reduce bulk. Creative selection and processing of fines may alter the severity of the strength-bulk relationship [9]. Milled dry kraft fibers are effective debonders and bulking agents [10]. Lee et al. [11] used wood powder as a bulking agent. In a mill trial making duplex board, the wood powder addition was used as an alternative to old corrugated containers (OCC) and because it provided similar bulk with reduced drying costs. High-yield pulps have been used to increase bulk and opacity in kraft pulps but can also improve formation [12]. Lee et al. [13] found that another organic filler, brewer's grain, also increased bulk and reduced drying energy, and its particle size was an important controlling feature.

Cannell and Cockram [14] provide a good review of the many variables that the mill manager must control to achieve good printability, high opacity and good strength. Long fibers improve tear but have low printability and poor formation. Conversely, short fibers have good surface smoothness and opacity but low tear. Small fiber diameters improve printability, opacity, and softness; large fiber diameters increase bulk and reduce smoothness and printability. Thick fiber walls reduce tensile strength, cause greater linting, and reduce smoothness.

In this work, we present a framework for investigating the effect of incorporating processed biomass in a paper network. This framework can help choose potential users of the specified processed biomass by examining its effect on a range of paper grammages and biomass loading levels. EHRS served as a model processed biomass for this proposed framework. We



1. Scanning electron microscope (SEM) images of hydrolyzed material. Background features are artifacts of imaging.

believe that EHRS can contribute physical properties to improve opacity and increase bulk. Specifically, this work examines the use of EHRS as a bulking agent in a kraft pulp. Because bulk increases were the objective of this work, a high yield pulp was used; if this study showed EHRS increased bulk, it can be inferred that the same would be true for less bulky base pulps. Beneficial use of EHRS can provide additional revenue to the papermaker, improve the economics of cellulosic ethanol production, and increase the use of forest biomass, a renewable resource.

MATERIALS

Handsheets examined in this work were composed of a mixture of kraft pulp fibers and hydrolyzed cellulosic solids. The kraft pulp was made from lodgepole pine with a 60 min ramp from 80°C–170°C followed by a 25 min cook at 15.7% active alkalinity and 30% sulfidity. The resulting pulp had 67% yield, 106 kappa no., 19.8% solids as chips, and 16.7% solids once shredded.

EHRS was the residue of enzymatic hydrolysis of a lodgepole pine pulp produced from a bisulfite pretreatment [15] at sodium bisulfite loading of 6 wt% on wood (oven dry basis) at 180°C for 30 min in a laboratory pulping digester. The pretreated lodgepole pine wood chips were refined in a 12 in. laboratory disk refiner (Sprout-Waldron Operation, Koppers Company; Muncy, PA, USA) with a disk plate pattern D2-B505 having a plate gap of 1 mm. Added water served to facilitate refining and wash pulp and resulted in pulp discharging consistency of 10%. Pretreatment removed approximately 25% of lignin, over 50% of xylan, and 75% of mannan, which created a pulp enriched with glucan (approximately 50%). As a first trial, the pretreatment condition was mild and at a slightly acidic condition that was not optimized for sugar production but rather to result in a solids similar to chemithermomechanical pulp (CTMP). Low dosages of cellulase (Celluclast 1.5 L, Novozymes, Sigma-Aldrich; St. Louis, MO, USA) of 7 filter paper unit (FPU)/g glucan and beta-glucosidase (Novozyme 188, Sigma-Aldrich) of 10 CBU/g glucan were used. Enzymatic hydrolysis was conducted at 50°C at a solids loading of 12%. Coupled with low severity pretreatment, the low enzyme dos-

age produced aglucan saccharification of approximately 27%, or approximately 30% monomeric sugar yield from carbohydrates when hemicelluloses were taken into account. Enzymatic saccharification of xylan and mannan was minimum. Resulting EHRS was enriched with lignin, although a substantial amount of cellulose and small amount of hemicellulose remained.

Figure 1 shows three example scanning electron microscope (SEM) images of hydrolyzed material. The material had many different sizes and aspect ratios. No additional refining was used before incorporating in the kraft pulp used for the handsheets. Because of the high yield of the EHRS we expected it would act as a debonder.

HANDSHEET PRODUCTION

Handsheets of a range of grammages, 20 g/m²–200 g/m², loaded with different amounts of EHRS, 10%, 15%, and 20%, were produced. The range of grammages and EHRS loading levels were used to identify potential paper products that would benefit from increased bulk. Solids contents of the pulp and EHRS were determined after their respective production. Appropriate amounts of pulp and EHRS were mixed prior to disintegration according to the amount of EHRS loading, either 0% (control), 10%, 15%, or 20% EHRS on a dry weight basis. After weighing for correct amounts of pulp and EHRS, the mixture was disintegrated for 5000 revolutions. The handsheets were made according to TAPPI Standard Test Method T 205 sp-12 “Forming handsheets for physical tests of pulp,” with the exception that water at 27°C was used in pulp disintegration instead of at 20°C. Precise grammage control was not used during production because individual handsheet grammage was determined and used to present results on a specific weight basis. For each nominal handsheet weight, two handsheets were made and weighed, and if necessary, small measured additions of water or pulp mixture were added to the drier tank. Handsheets were ring-dried overnight at 23°C and 50% relative humidity (RH). Before testing, the handsheets were preconditioned for 12 h at 27°C and 30% humidity and reconditioned at 23°C and 50% RH for 12 h.

It is not possible to determine with any precision the

amount of EHRS in the handsheets. We expected that during initial drainage of the handsheet, a portion of the EHRS and pulp fines would pass through the screen. Eventually the filter cake effect would prevent additional losses. The net effect would be a lower amount of EHRS and fines in the final material than was introduced in the handsheet mold.

Handsheet caliper was measured using TAPPI Standard Test Method T 411 om-10 "Thickness (caliper) of paper, paperboard, and combined board." Because of the pulp coarseness here, this method tended to overestimate caliper, especially in the higher grammage handsheets.

MATERIAL TESTS

Increased bulk generally reduces stiffness and strength and increases opacity. Tensile, ultrasonic, and compression testing were used to examine the effect of EHRS loading, to demonstrate the disruption in the fiber network caused by EHRS, and to probe the level of EHRS loading that would create a non-commercially viable product. Opacity evaluation was used as a non-mechanical evaluation technique to probe EHRS retention.

Tensile tests

Tensile tests were performed according to TAPPI Standard Test Method T 494 om-88 "Tensile properties of paper and paper board (using constant rate of elongation apparatus)," with a few exceptions, on an Instron (Norwood, MA, USA) Model 5865 test machine equipped with pneumatic grips. All specimens were 15 mm wide and were cut with a Thwing-Albert (West Berlin, NJ, USA) Precision Cutter. Specimens at nominally 20 g/m² were supported by an envelope paper during cutting. Specimens from handsheets at 80 g/m² and higher were tested with line clamps, 100 mm gauge length, at a rate of 20 mm/min. Because line clamps caused failure at the clamps for handsheets below 80 g/m², those specimens were tested with textured, flat platen grips. The flat platen grips covered a larger portion of the specimen and made it necessary to reduce the gauge length to 75 mm. To keep the recommended test period within TAPPI Standard Test Method T 494 om-88 guidelines, the rate of elongation was reduced to 10 mm/min for the specimens tested with the flat platen grips. Grip elongation and applied force were collected at 10 Hz.

Only data from specimens whose failure occurred away from the grips were analyzed. Grip elongation was converted to strain using the appropriate gauge length, and tensile applied force was converted to force/width using specimen width. Converted data were analyzed by fitting to the empirical equation suggested by Andersson and Berklyto [16] and given by:

$$\sigma = C_1 \tanh[C_2(\epsilon - \delta)] + C_3(\epsilon - \delta) \quad (1)$$

where:

σ = stress, kN/m

ϵ = strain, grip displacement/gauge length, m/m

C_1, C_2, C_3 = empirical constants

δ = strain offset, empirical constant

Fitting to Eq. (1) was performed in Matlab via the built-in *nlinfit* function. The initial portion of each tensile test, where grip slippage was evident, was not used in the fitting. For handsheets at nominal 20 g/m², data below 2 N were not used; for nominal 40 g/m² and 60 g/m², data below 3 N were not used; for all other grammages, data below 4 N were not used. The upper data cutoff was at maximum tensile force, i.e., tensile strength. Failure strain, ϵ_{max} , was the strain at the tensile strength. Because initial stiffness is quite sensitive to grip slippage and different low force cutoffs were used for different grammages, initial tensile stiffness was not a reliable comparison in this work. Tensile energy absorption, TEA, was determined by integrating Eq. (1) and evaluating at $E = \epsilon_{max}$, giving $TEA = C_1/C_2 \ln(\cosh(C_2 * \epsilon_{max})) + C_3 \epsilon_{max}^2/2$.

Ultrasonic testing

Ultrasonic testing measures the time-of-flight of a longitudinal sound wave (supersonic pulse) to travel through the sheet between two probes of fixed distance. For each handsheet, ultrasonic tests were performed with a Nomura Shoji (Tokyo, Japan) SST-250 paper tester in which the transmission probe oscillated at 25 kHz. A central circular region, with 15-cm diameter, was examined for each specimen. Ultrasonic velocity was measured from 0 to 175° in 5° increments, giving 36 measurements for each handsheet. Specific stiffness was the mean ultrasonic velocity squared, $Q_{specific} = (\text{mean}(\text{velocity}))^2$. Ultrasonic velocities of nominal 20 g/m² handsheets are not reported because they were a web of discrete fibers and violated the continuum requirement for ultrasonic stiffness identification.

Compression strength

For handsheets above 100 g/m², compression strength was measured with a Büchel Stripcompression Tester BK-155 (Veenendaal, Netherlands).

Optical testing

For control and 20% EHRS loaded handsheets above 40 g/m² nominal, absorption and scattering coefficients were identified using the procedure given in [17,18] because the material was unbleached, nearly opaque, and contained lignin. Most opacity measurements are intended for bleached material and would be unable to differentiate between materials here due to the significant light scattering. The procedure [17] is able to identify the Kubelka-Munk coefficients, k (specific absorption coefficient), and s (specific scattering coefficient) by examining transmission and reflectance in the infrared spectrum. The k values were similar for both control and 20% EHRS loaded materials

	Kraft Pulp	EHRS
Fines ^{1,2} , %	3.5	29.11
Mean length ² , mm	2.04	0.35
Mean curl index ²	0.43	0.30
Mean kink index, 1/mm	0.40	1.20
Mean fiber width, μm	36.7	40.3
Coarseness, mg/m	N/A	0.29
¹ Length weighted.		
² Fibers < 0.20 mm.		

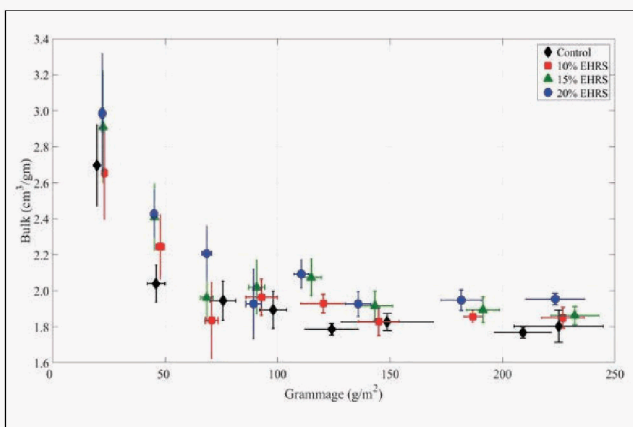
1. Fiber and fines analysis.

because they were of similar color and contained similar amounts of lignin. Results for the Kubelka-Munk specific scattering coefficient, s , are in the Discussion section.

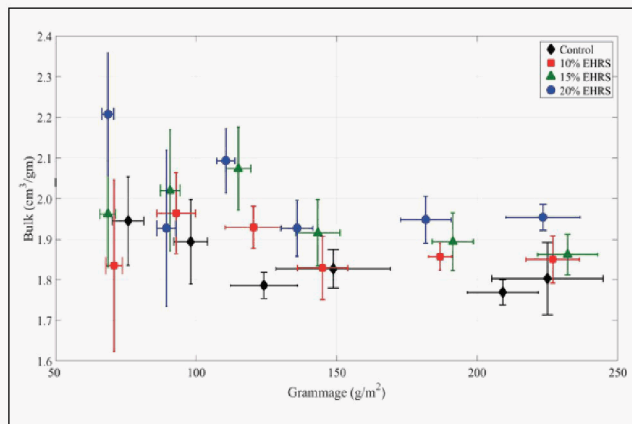
RESULTS

The physical characteristics of the kraft pulp and EHRS material were examined with a Fiber Quality Analyzer (OpTest Equipment; Hawkesbury, ON, Canada) and are listed in Table I. Pulp preparation was designed to produce few fines and maintain fiber length. EHRS material had a large amount of fines and short fibers. Pulp curl and kink indices indicate the pulp fibers were straight with few abrupt angles. EHRS curl index indicates the material was composed of generally straight fibers, whereas the kink index indicates that some folding of fibers was present. As expected, fiber widths were similar for each material. The EHRS material was quite coarse but was likely influenced by the large amount of short fibers.

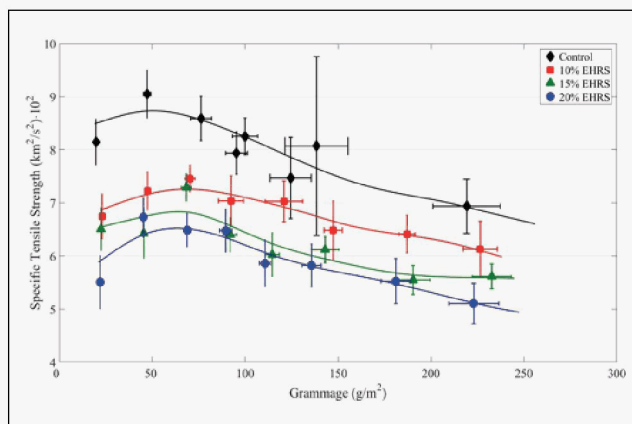
Figure 2 and Figure 3 show that the addition of EHRS did add bulk; Fig. 3 uses the same data, but includes only those materials greater than 40 g/m². Each data marker is located at the mean grammage and mean bulk for each handsheet set; er-



2. Enzymatic hydrolysis residual solids (EHRS) material increased handsheet bulk Horizontal and vertical error bars span a standard deviation from the mean value in all four directions.



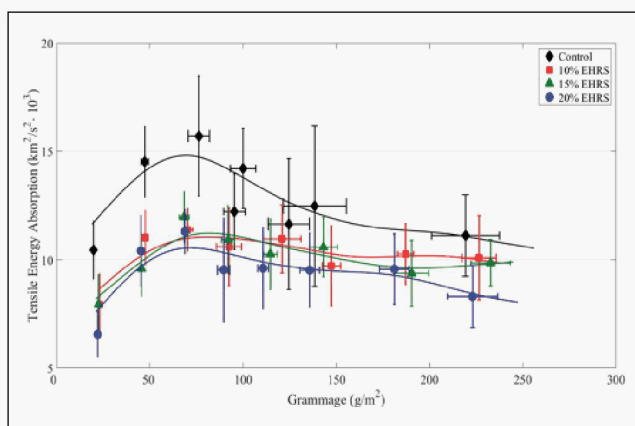
3. EHRS material increased handsheet bulk on reduced x-axis scale. Horizontal and vertical error barn span a standard deviation from the mean value in all four directions.



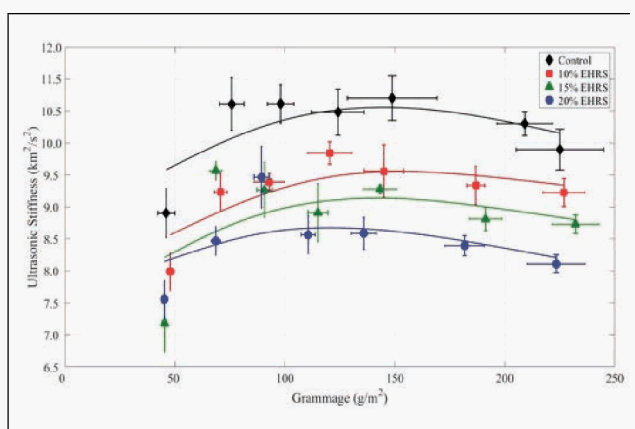
4. Specific tensile strength for each handsheet. Lines are smoothed cubic splines intended to guide the reader's eye. Horizontal and vertical error barn span a standard deviation from the mean value in all four directions.

rors bars denote standard deviation. Significant bulk increases were not evident until grammages were above 100 g/m², at which point each additional amount of EHRS increased bulk. It is not possible to use microscopy to verify amount of EHRS retention. A combination of a coarse furnish and the lack of filter caking yielded inconsistent bulk improvements at low grammages. At high grammages, the increased amount of fibers reduced the number of pores and improved fines retention.

Figure 4 shows the specific tensile strength results for the materials in this study. The general trend for each material in Fig. 4 agrees with data of l'Anson and Sampson [19], which they related to Wiebull's failure theory. At low grammages, the material is nearly discrete and stress transfer between fibers is inefficient, e.g., creating bending moments between two perpendicular bonded fibers. Additional fibers improve stress transfer efficiency. Finally, inefficiency occurs with more fibers as probability of weak zones increases. For the control material, weak zones occurred in regions of poor bonding. Tensile failure strain, not shown, had a large amount of scatter and no general trends were observed. So, although specific tensile



5. Tensile energy absorption (TEA). Lines are smoothed cubic splines intended to guide the reader's eye. Horizontal and vertical error bars span a standard deviation from the mean value in all four directions.



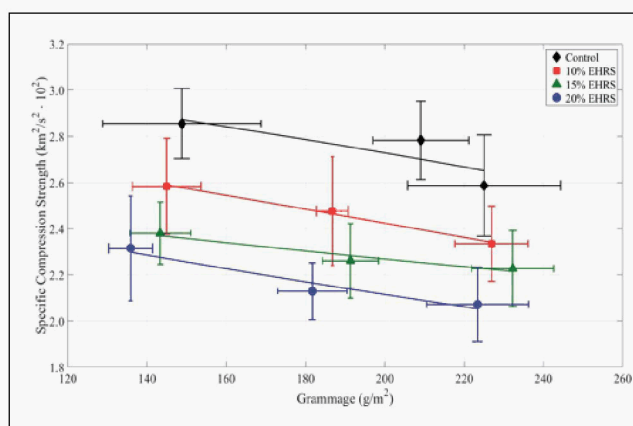
6. Ultrasonic stiffness. Lines are smoothed cubic splines intended to guide the reader's eye.

strength was reduced, stretch was largely unaffected.

TEA results are shown in Fig. 5; even the smallest addition level of EHRS (10%) produced large TEA reduction. Higher levels of EHRS addition did not create more reduction. As in specific strength, TEA peaked near 60 g/m² and decreased with grammage.

Ultrasonic stiffness measurement requires a continuum [20], which was violated by the 20 g/m² handsheets, and those results are not included. The results for the 40 g/m² handsheets could also be reasonably questioned. In Fig. 6, the ultrasonic stiffness increased with grammage up to about 120 g/m². Removal of the data below 50 g/m² would indicate a gradual reduction of ultrasonic stiffness with increased grammage. This reduction likely occurs because the weak/soft zones of the handsheets increased with grammage, reducing the velocity and increasing the attenuation of the bulk ultrasonic wave.

For handsheets above 100 g/m², compression strength is shown in Fig. 7. The bond disruption caused by EHRS reduced compression strength and was generally in proportion



7. Compression strength Lines are smoothed cubic splines intended to guide the reader's eye.

to the addition of EHRS. The gradual decrease of compression strength with increased grammage is similar to the loss in tensile strength and reflects the greater likelihood for defects at higher grammages.

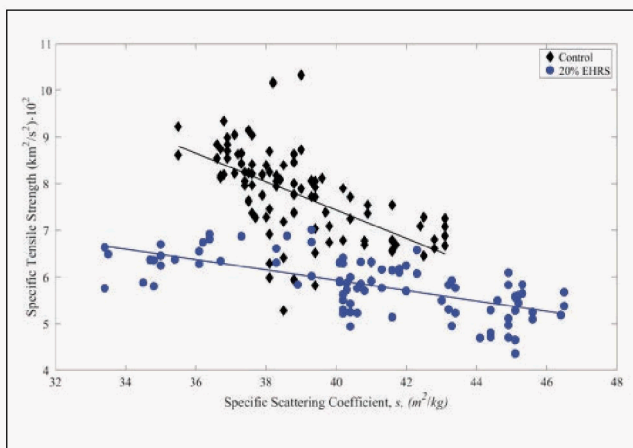
DISCUSSION

This study demonstrates some of the challenges to the paper-maker. Although several variables were examined, namely grammage and EHRS loading level, many other variables, namely processing and refining of control pulp and EHRS, were not examined. A more detailed examination of processing and refining may be helpful when addressing the requirements of a particular product. Prescribed refining of fines [9] has been used to maintain tensile index.

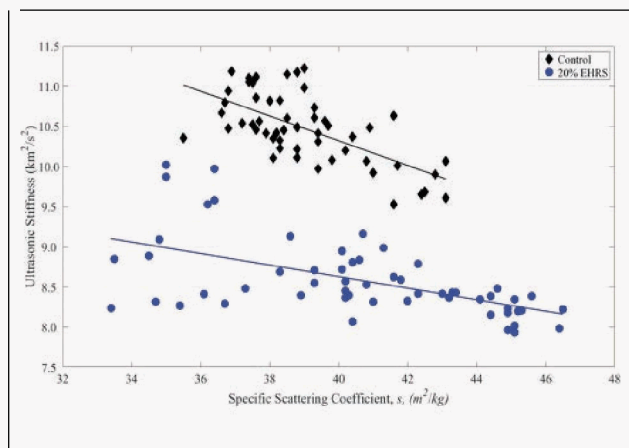
Economically beneficial use of EHRS is important to the success of biofuels. Unlike other bulking agents, such as thermomechanical pulp (TMP) fibers, wood powder, etc., EHRS is a low-value byproduct. Creation of bulk demonstrated here, while controlling physical and mechanical properties, adds value to EHRS and creates an additional revenue stream for cellulosic ethanol production.

It was not possible to directly determine the amount of EHRS in each handsheet. Loss of specific tensile strength shown in Fig. 4 provides good evidence that EHRS was retained in the sheet and that higher dosages of EHRS resulted in additional EHRS retention. Loss of specific tensile strength between control and 10% EHRS was similar to the loss between 10% EHRS and 20% EHRS materials. Given this behavior, it seems reasonable to expect that if 5% EHRS loaded handsheets had been produced, their specific tensile strength would tend to lie midway between the control and 10% loaded material and would likely be an appropriate loading level for commercial products.

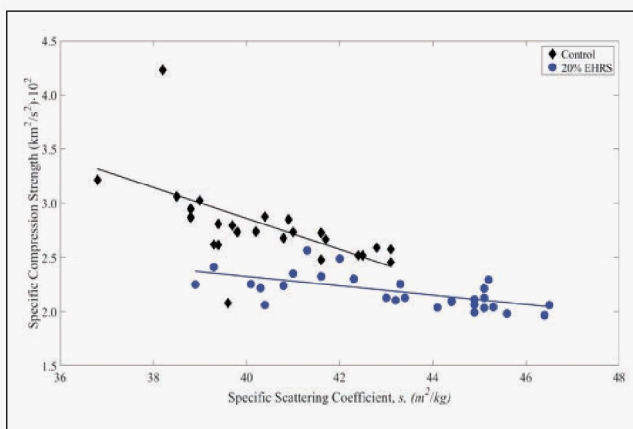
Strength is controlled by weak regions [19], which increase in number with added material and was similar in both tension, Fig. 4, and compression, Fig. 7. Bulking increased the probability density of weak regions. In some instances, fines act as small fibers, which assist inter-fiber bond formation



8. Light scattering and tensile index. Lines are smoothed cubic splines intended to guide the reader's eye.



10. Light scattering and ultrasonic stiffness. Lines are smoothed cubic splines intended to guide the reader's eye.



9. Light scattering and compression index. Lines are smoothed cubic splines intended to guide the reader's eye.

[21]. However, EHRS behaved more like inorganic fillers which interfere with fiber bonds and reduce tensile index and Scott Bond [22].

The mechanisms that influence stiffness and strength are not the same [23]. Stiffness is relatively insensitive to material heterogeneities, whereas strength is greatly influenced. Fiber bonding has a significant influence on both. Loss of ultrasonic stiffness, as seen in Fig. 6, was almost independent of EHRS loading up to 100 g/m². Above that grammage, the ultrasonic stiffness was not independent of EHRS loading, likely caused by increased bond disruption, which affects the ability of the bulk wave to travel through the material.

Specific tensile stiffness, which is not shown, is not a relevant property to compare EHRS addition among the various grammages. Because ultrasonic stiffness is a bulk material property, the differences between EHRS loading amounts better represent network behavior. Coffin et al. [24] attributes the differences between these measurements to time dependent behavior, experimental errors, and difficulty in slope determination for the tensile stiffness. Machine compliance of the tensile tester also increases with greater material

stiffness, i.e., the tensile stiffness will be reduced more at higher grammages than lower grammages as the machine compliance effects become more dominant.

At 20% EHRS loading, the EHRS decreased scattering, Fig. 8, at the similar tensile indices. This would occur if EHRS filled void spaces and reduced surface roughness [25]. Here, EHRS material was not refined, but purposeful fines refining can improve scattering and tensile index [26].

Figure 8 also indicates the significant effect of EHRS addition; this effect is similar in compression strength, as shown in Fig. 9. Changes in relationship between scattering and strength are less sensitive with addition of EHRS than that of the control material. These data indicate that for these materials, strength can be predicted by amount of kraft fibers and a bonding factor, where the bonding factor is given by s . Knowledge of the relationship between strong fibers, scattering (i.e., bonding), and strength is important in process control systems.

EHRS addition creates extensive bond disruption and reduces ultrasonic stiffness. Figure 10 suggests that the bond disruption is so prevalent that increased scattering (more bond disruption) causes less network disturbance because the additional EHRS has less probability of disrupting critical bonding regions. Increased scattering for the control material proportionally decreased ultrasonic stiffness more; the increased scattering was directly disrupting critical bonding regions.

CONCLUSION

This study suggested a framework for the investigation of incorporation of processed biomass in a paper sheet in order to provide important information regarding mechanical, physical, and optical property changes. Potential users will be able to make decisions on this biomass as an additive for their products. Furthermore, this framework provides a basis for investigation of the use of other processed biomass in cellulose-fiber materials.

Addition of EHRS increased bulk and reduced tensile strength, ultrasonic stiffness, and specific compression strength in proportion to the amount of EHRS addition.

Stretch was unaffected by EHRS addition. Specific tensile energy absorption had significant reduction across all grammages, regardless of the amount of EHRS added.

The effect of EHRS was similar to that of other bulking materials where bulk is increased at the expense of strength properties. When increased bulk is desired in paper materials, use of EHRS would improve the economic viability of cellulosic ethanol production and provide an additional revenue stream for papermakers.

Light scattering was related to specific tensile strength, specific compression strength, and ultrasonic stiffness, but the relationship was different with EHRS addition. EHRS decreased the sensitivity of these properties to scattering differences.

Optimizing ethanol production and paperboard properties by feedstock source, byproduct refining, and end product requirements is a difficult task but also promises significant economic reward. **TJ**

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ABOUT THE AUTHORS

Bulking agents can reduce fiber costs. We decided to examine the byproducts of cellulosic ethanol production as bulking agents to improve cellulosic ethanol economics. This work supports earlier work where identification of material properties was critical.

We did encounter difficulty in directly determining the amount of EHRS in the handsheets, but use of light scattering provided a good surrogate for direct determination. We also discovered that much work remains to be done. EHRS, and other bulking agents, may be viable, but require optimization for particular applications. Our most interesting finding was that the mechanical properties varied directly with EHRS addition.

Bulking agents are in great demand. Production of bulking agents from low value wood may add a revenue stream for mills. Our next step is to examine processing the bulking agent to reduce mechanical property loss.

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