2950 Niles Road, St. Joseph, MI 49085-9659, USA
269.429.0300 fax 269.429.3852 hq@asabe.org www.asabe.org

An ASABE Meeting Presentation

Paper Number: 1111089

Application of Buckmaster Electrolyte Ion Leakage Test to Woody Biofuel Feedstocks

Thomas F. Broderick

James H. Dooley

Forest Concepts, LLC

**Written for presentation at the
2011 ASABE Annual International Meeting
Sponsored by ASABE
Gault House
Louisville, Kentucky
August 7 – 10, 2011**

***Abstract*.** In an earlier ASABE paper, Buckmaster reported that ion conductivity of biomass leachate in aqueous solution was directly correlated with activity access to plant nutrients within the biomass materials for subsequent biological or chemical processing. The Buckmaster test involves placing a sample of the particles in a beaker of constant-temperature deionized water and monitoring the change in electrical conductivity over time. We adapted the Buckmaster method to a range of woody biomass and other cellulosic bioenergy feedstocks. Our experimental results suggest differences of electrolyte leakage between differently processed woody biomass particles may be an indicator of their utility for conversion in bioenergy processes. This simple assay appears to be particularly useful to compare different biomass comminution techniques and particle sizes for biochemical preprocessing.

Keywords. *Biomass, forest, bioenergy, quality, standards, assay*

Introduction

Biomass assay development is an important component of the ongoing Precision Feedstocks™ program at Forest Concepts. LLC. Quality control tests for characterizing biomass feedstocks prior to pre-processing should be simple, rapid, and adaptable to the next generation of biomass feedstocks under development.

Dennis Buckmaster at Purdue University has recently evaluated electrolytic ion leakage as a method to assess activity access for subsequent biological or chemical processing of forage or biomass. (Buckmaster, 2008.) He concluded that ion conductivity of biomass leachate in aqueous solution was directly correlated with activity access to plant nutrients within the biomass materials for subsequent biological, chemical, or even combustion processes. Here we report preliminary observations on adapting this method to a new class of sheared biomass feedstock particles (aka Crumbles™) with consistent piece size uniformity and high surface area to volume ratios.

Safety Emphasis

The assay reported herein uses deionized water at ambient temperature rather than harsh chemicals. The technique is inherently safe to apply outside of a laboratory environment and by technicians with reasonable lab skills but without formal chemical safety training.

Ion Conductivity Leachate Assay

Ion conductivity of biomass leachate in aqueous solution was assessed with the following equipment, protocol, and materials:

Equipment

Jenco® Model 3173/3173R Conductivity/Salinity/TDS/Temperature Meter

Corning® Model PC-420 Laboratory Stirrer/Hot Plate

Aculab® Model VI–1200 Balance

Protocol

(1) Measure the initial temperature compensated conductivity (CC, in microSiemens (µS)) of 500 ml of distilled water maintained at ~25˚C in a glass vessel;

(2) Add a 10 g sample of feedstock particles into the water, and stir the pieces at 250 RPM in the water at ~25˚C for 60 minutes;

(3) Briefly stop stirring and measure the CC of the water at 15-minute intervals; and,

(4) Calculate an experimental CC value for comparison purposes by subtracting the initial CC from the CC at 30 minutes.

Figure 1 Simple experimental set up with EC meter, timer, and hot plate stirring device.

Materials

Wood particles were manufactured by rotary cross-grain shear using 3/16" wide cutters from a knot-free sheet of Douglas fir 1/6" thick of peeled veneer (10-15% moisture content). The resulting particles were sorted five minutes by RotoTap using 1/4”, No. 4, No. 8, and No. 10 screens. Then, for the precision desired in this particular experiment, the Pass 1/4" / No Pass No. 4 fraction was hand sorted to select a 10 g <u>experimental sample</u> of particles that in all dimensions passed through the 1/4" screen (nominal sieve opening 6.3 mm) but were retained by the No. 4 screen (nominal sieve opening 4.75 mm). Representative sheared wood feedstock particles from this experimental sample (FS-1) are shown in FIGURE 1B.

Similarly sized cubes indicative of coarse sawdust and chips were cut from the same veneer sheet, using a Vaughn® Mini Bear Saw™ Model BS 150D handsaw. The sheet was cut cross-grain into approximately 3/16" strips. Then each strip was gently flexed by finger pressure to break off roughly cube-shaped particles of random widths. The resulting particles were size screened, and a 10 g <u>control sample</u> was collected of particles that in all dimensions passed through the 1/4" screen but were retained by the No. 4 screen. Representative cubes from this control sample (Cubes-1) are shown in FIGURE 2A.

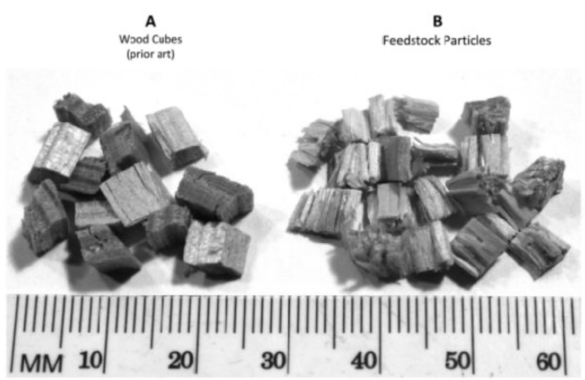


Figure 2. (A) Wood cubes and (B) sheared wood feedstock particles

The extent length, width, and height dimensions of each piece in each sample were individually measured with a digital caliper and documented in table form. Table 1 summarizes the resulting data.

Table 1

Samples (10 g)	Number of pieces	Length (L)	Width (W)	Height (H)
Control cubes (Cubes-1)	n = 189	Mean 5.5 SD 0.48	Mean 5.0 SD 1.17	Mean 3.9 SD 0.55
Experimental particles (FS-1)	n = 292	Mean 5.3 SD 0.74	Mean 5.8 SD 1.23	Mean 3.3 SD 0.82

The Table 1 data indicates that the extent volumes of these size-screened samples were not substantially different. Accordingly, the cubes and particles had roughly similar extent volumes (extent L x W x H). Yet the 10 gram experimental sample contained 54% (292/189) more pieces than the 10 gram control sample, which equates to a mean density of 0.34 g/particle (10/292) as compared to 0.053 g/cube. FIGURE 1 shows that the roughly parallelepiped extent volumes of typical particles (1B) contain noticeably more checks and air spaces than typical cubes (1A). These differences indicate that the sheared wood feedstock particles had significantly greater skeletal surface areas than the wood cubes. One would therefore expect the particles to exhibit more ion leachate than the cubes in aqueous solution.

Individual handling during the caliper measurements tended to damage the Table 1 particles (FS-1), and so a second set of 10 g samples of cubes (Cubes-2) and particles (FS-2) were made as described above from another sheet of veneer for ion conductivity leachate assessments as described below.

Results

The resulting Calibrated Conductivity data from the replicate set of samples is shown in Table 2 and plotted FIGURE 3.

Table 2

Sample	Temperature Calibrated Conductivity (µS)				
	0 min	15 min	30 min	45 min	60 min
Control cubes (Cubes-2)	1.9	6.7	8.6	9.8	10.8
Experimental particles (FS-2)	1.9	12.0	15.0	16.5	17.8

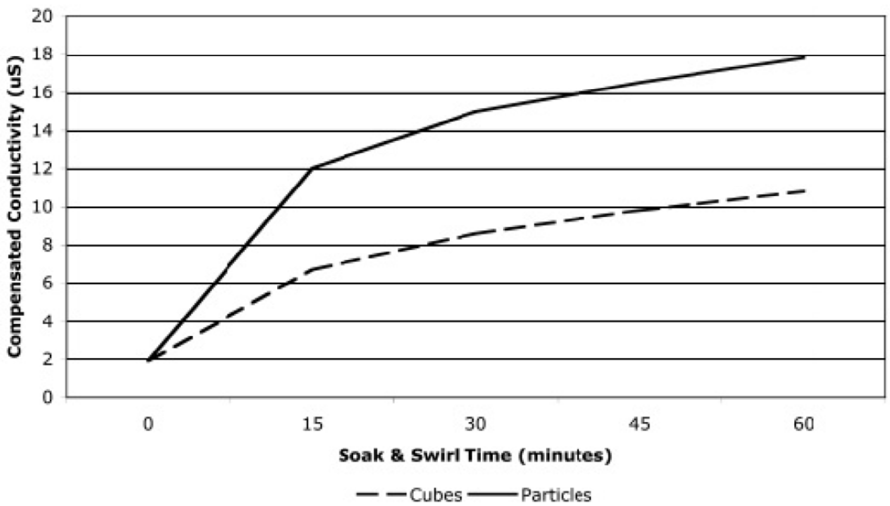


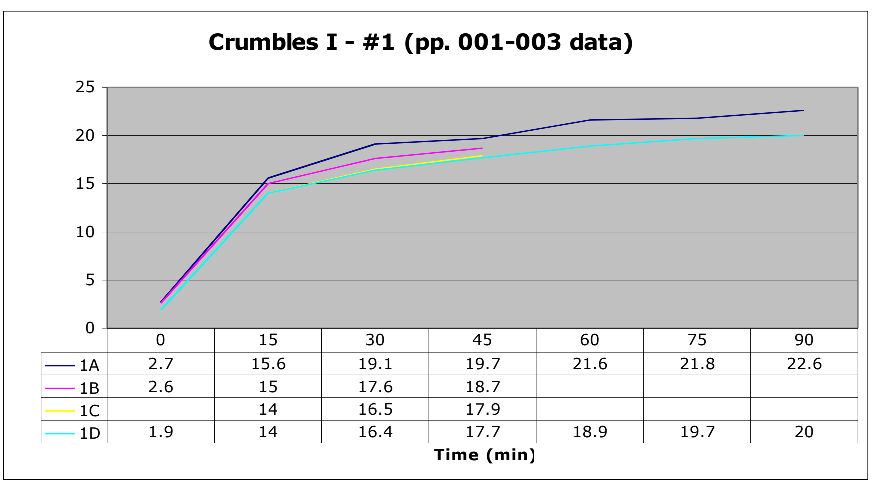
Figure 3. Calibrated conductivity vs time for wood cubes and wood particles

These results indicate that the particles exhibited nearly twice the activity index of similarly sized wood cubes that generally lacked the cross-grain end checking that characterizes the sheared wood feedstock particles.

Discussion

The skirmish experiment reported above is suggestive that this simple ion leachate assay may serve as a rough indicator of particle density, and perhaps even surface area.

We have observed that the assay is fairly robust and replicable when run with clean white wood feedstocks.



	0	15	30	45	60	75	90
1A	2.7	15.6	19.1	19.7	21.6	21.8	22.6
1B	2.6	15	17.6	18.7			
1C		14	16.5	17.9			
1D	1.9	14	16.4	17.7	18.9	19.7	20

Figure 4 Ion leachate data from four samples of unscreened Crumbles™ particles made from 1/10th inch Douglas fir veneer with 3/16" cutters.

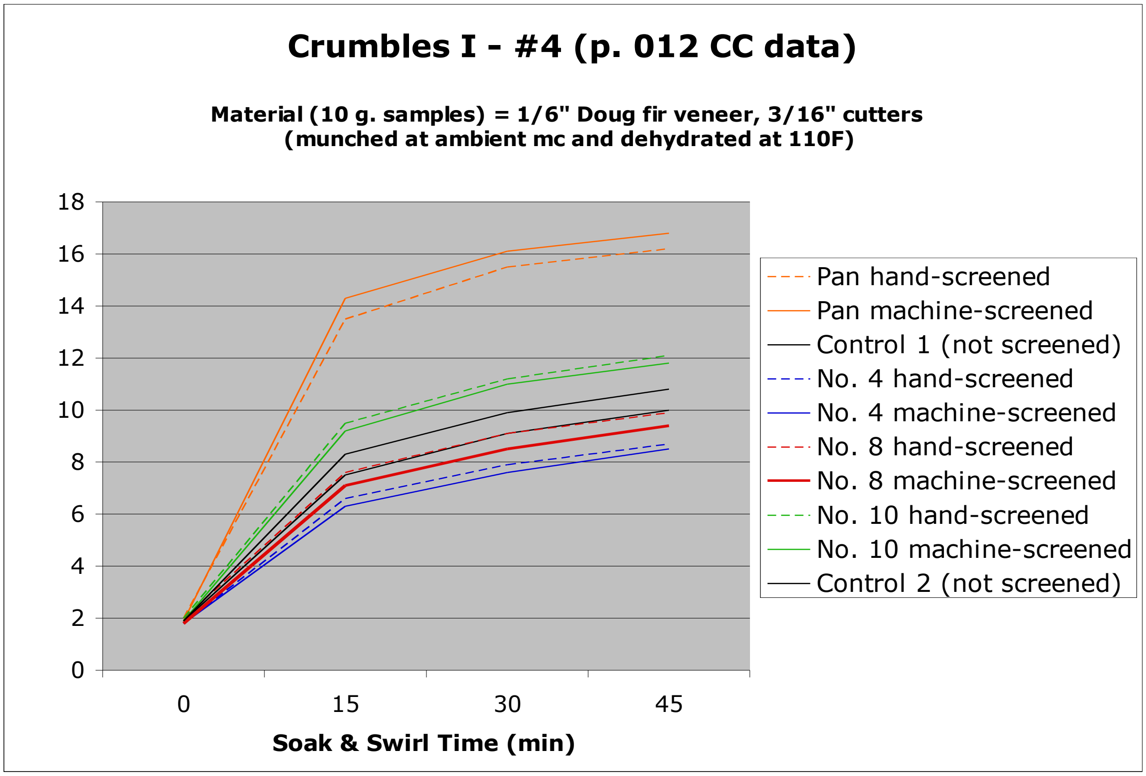


Figure 5 Ion leachate data for size sorted fractions of sheared feedstock particles made from 1/16^{h} inch Douglas fir veneer with 3/16" cutters.

Figure 5 is again suggestive that the assay may serve as a rough indicator of particle surface area.

Here we should emphasize that all the data reported above resulted from white wood particles of substantially uniform shape and dimensions, made from peeled veneer and consequently lacking bark and forest dirt contaminants. The skirmish experiments below indicate that more heterogeneous materials produce less consistent ion leachate results.

Notably, dirt and bark contaminants tend to elevate the assay results. For example, Figure 6 shows a Pass No. 4 / No pass No. 8 fraction of relatively clean microchips (produced from debarked logs of Oregon source fir) that provided the ion absorbance data shown in Table 3.

Figure 6. Sample of conifer microchips produced by commercial scale drum chipper.

Table 3

Sample	Temperature Calibrated Conductivity (µS)				
	0 min	15 min	30 min	45 min	60 min
Micro chips	2.1	12.5	15.0	16.5	17.9

The Table 3 leachate results are noticeably higher than for the comparably sized (No. 8) white wood particles in Figure 6. This result was somewhat unexpected, as preliminary stereomicroscopic examination had indicated that these chips had characteristically solid shear faces without prominent checking. However, the soak & swirl water of the Figure 6 microchips became noticeably cloudy and dirty, as shown in the Figure 7 photograph taken at ten minutes into the assay.

Figure 7. Cloudy and dirty solution after 10 minutes of microchip agitation.

Salt is an additional contaminant that will ("by definition") skew this assay's results. Forest Concepts recently notes some odd-looking veneer sheets (see Figure 8 below) in a pallet we received for WoodStraw® much production.

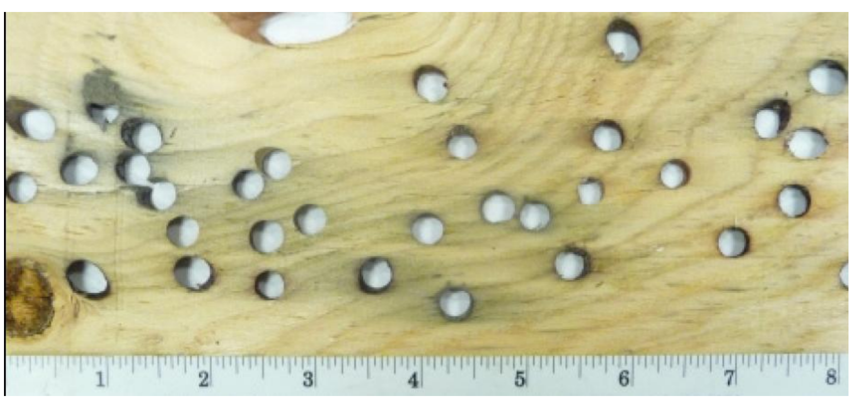


Figure 8. Wood veneer containing saltwater worm holes.

Our initial thinking was that the ~1/2 inch diameter boreholes were formed by woodpeckers, but forestry colleagues at Oregon State University suggested shipworms (a marine bivalve mollusk, genus *Teredo*). Evidently at least some of the veneer sheets in this pallet were made from logs that had spent some considerable time in seawater. This possibility raised a potential quality control concern, as salt water-contaminated biomass might disrupt certain biofuel preprocessing techniques. Our hypothesis that shipworm boreholes provide pathways for salt water to penetrate deep within logs was supported by the following skirmish experiment.

Crumbles™ sheared wood feedstock particle were prepared from the Figure 8 veneer sample by running the 1/10” conifer sheet cross-grain through 3/16” rotary cutters. The resulting particles were sorted 5 minutes by RotoTap using 1/4”, No. 4, No. 8, and No. 10 screens. Ten grams of the No. 4 Pass / No. 8 No Pass particles were assayed as described above. For comparison, reference was made to several prior runs of CC data from similarly sized particles made from peeled conifer veneer, which “control” runs were presumed to be direct-from-forest wood because they all exhibited similar and consistent ion leachate profiles.

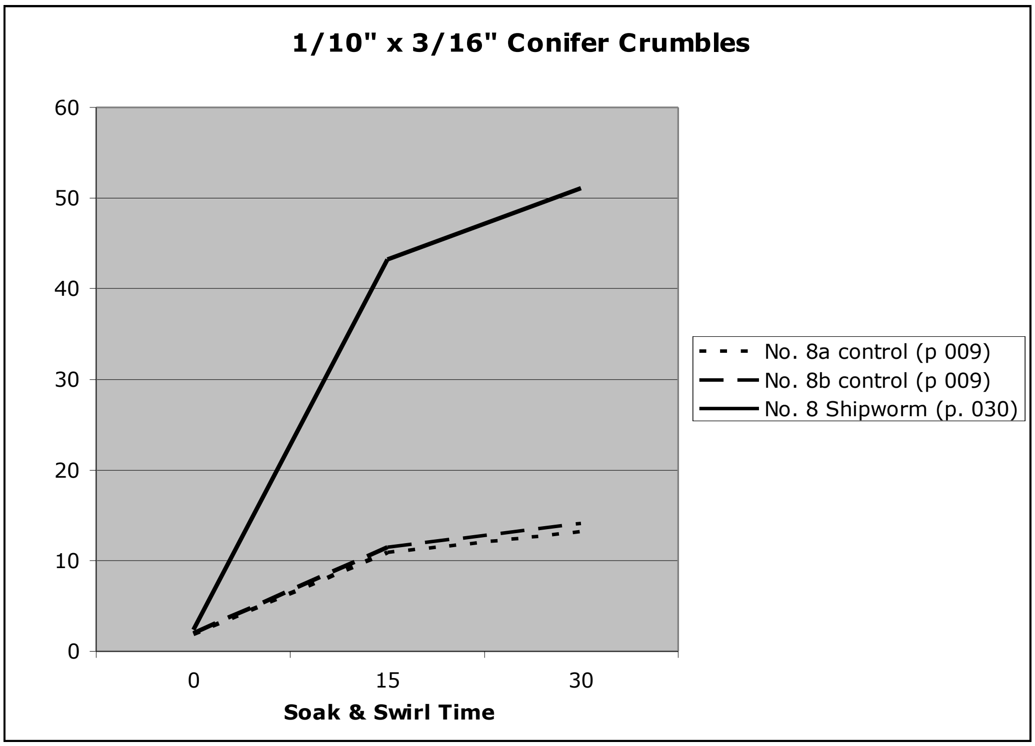


Figure 9. Comparison of saltwater rafted wood (shipworm affected) to similarly sized (No. 8) control particles.

This skirmish result suggests that seawater-exposed biomass may contain elevated salt concentrations that could interfere with cellulase preprocessing techniques, e.g., microbial digestion.

Moreover, the skirmish experiments reported above suggest that such the subject assay may be sufficiently qualitative (yes / no) for field or plant receiving station use, as the CC of the forest-chip and ocean-log samples greatly exceeded the established baseline range for sheared wood feedstock particles produced direct-from-forest veneer. Tap water and a hand-held conductivity meter might suffice for a quick, initial screen at a biomass receiving station.

Conclusion

Our experimental results suggest differences of electrolyte leakage between differently processed woody biomass particles may be an indicator of their utility for conversion in bioenergy processes. This simple assay appears to be particularly useful to compare different biomass comminution techniques and particle sizes for biochemical preprocessing.

Acknowledgements

This research was supported by the Small Business Innovation Research program of the U.S. Department of Energy, Contract SC0002291, which support is gratefully acknowledged.

References

Buckmaster, D.R., "Assessing activity access of forage or biomass," *Transactions of the ASABE 51*(6):1879-1884, 2008.