

## Clarification Properties of Trash and Stalk Tissues from Sugar Cane

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The effect of the U.S. and worldwide change from burnt to unburnt (green) sugar cane harvesting on processing and the use of sugar cane leaves and tops as a biomass source has not been fully characterized. Sugar cane whole-stalks were harvested from the first ratoon (repeat) crop of five commercial, Louisiana sugar cane varieties (LCP 85-384, HoCP 96-540, L 97-128, L 99-226, and L 99-233). Replicated sample tissues of brown, dry leaves (BL), green leaves (GL), growing point region (GPR), and stalk (S) were separated. Composite juice from each tissue type was clarified following a hot lime clarification process operated by most U.S. factories. Only GPR and GL juices foamed on heating and followed the normal settling behavior of factory sugar cane juice, although GL was markedly slower than GPR. GPR juice aided settling. S juice tended to thin out rather than follow normal settling and exhibited the most unwanted upward motion of flocs. Most varietal variation in settling, mud, and clarified juice (CJ) characteristics occurred for GL. The quality rather than the quantity of impurities in the different tissues mostly affected the volume of mud produced: After 30 min of settling, mud volume per unit tissue juice °Brix (% dissolved solids) varied markedly among the tissues (S 1.09, BL 11.3, GPR 3.0, and GL 3.1 mL/°Brix). Heat transfer properties of tissue juices and CJs are described. Clarification was unable to remove all BL cellulosic particles. GL and BL increased color, turbidity, and suspended particles in CJs with BL worse than GL. This will make the future attainment of very high pol (VHP) raw sugar in the U.S. more difficult. Although optimization of factory unit processes will alleviate extra trash problems, economical strategies to reduce the amount of green and brown leaves processed need to be identified and implemented.

**KEYWORDS:** Sugar cane tissues; green leaves; brown leaves; biomass; clarification; clarified juice; mud

### INTRODUCTION

With an increasing shift in the U.S. and worldwide from the harvesting of burnt to unburnt (green) sugar cane and from whole-stalk to billeted sugar cane, even more trash (brown and green leaves, and tops) impurities are expected to be delivered to factories. Unfortunately, the effect of changing to green harvesting on processing has not been fully characterized, and few solutions to minimize the detrimental processing effects of trash have been implemented. Furthermore, the current trend to investigate sugar cane trash as biomass for the production of bioproducts, i.e., cellulosic ethanol, has made knowledge of the processing quality of trash tissues even more important. Such knowledge would underpin decisions on which bioproducts to manufacture from different trash tissues. Eggleston et al. (1) recently reported that over one-third (up to 43%) of the total dry biomass from Louisiana sugar cane was from the total trash, with green leaves delivering the most biomass of all the trash tissues.

The definition of what constitutes sugar cane trash varies in the literature and is frequently not clearly defined. Trash has been

defined as leaves with or without green tops. Green tops sometimes equate to green leaves (GL) and the growing point region (GPR) or apical internodes (2). Trash has also been defined as tops, leaves, plus soil. However, a complete definition of sugar cane trash should include both brown, dried leaves (BL), GL, and the GPR. Very little is known about the physicochemical and processing properties of the different types of trash and stalk (S) tissues, particularly the processing effects of brown versus green trash. It is known that green leaves and tops contain less sucrose and more ash, reducing sugars, color, organic acids, and starch (1) than stalks. Kestose (fructosyl sucrose) trisaccharides, which can deform the crystal shape during crystallization, are more abundant in the GL and GPR than in the S (1). Higher amounts of impurities in the GL and GPR, including invert sugars, can be attributed to these tissues being more active physiologically and having much more enzyme activity associated with them, particularly invertases ( $\beta$ -fructofuranosidases (3)). BL has been reported (4) to have relatively little effect on the purity of extracted juice but a more pronounced effect on the loss of sucrose to bagasse by absorption, and vice versa for GL.

Juice clarification has a great impact on factory evaporators' heat transfer coefficients, sucrose crystallization, raw sugar yield,

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**Table 1.** Variety Agronomic Characteristics<sup>a</sup>

| characteristic         | LCP 85-384           | HoCP 96-540                    | L 97-128                       | L 99-226          | L 99-233                              |
|------------------------|----------------------|--------------------------------|--------------------------------|-------------------|---------------------------------------|
| lodging <sup>b</sup>   | lodges easily        | lodges with difficulty (erect) | lodges with difficulty (erect) | mid-level lodging | lodges easily                         |
| leaf sheath attachment | leaves cling tightly | mid-leaf clinginess            | loose leaf sheath              | loose leaf sheath | very loose leaf sheath (self-removal) |
| % S <sup>c,d</sup>     | 80.2 bcA             | 83.6 abA                       | 81.4 bA                        | 82.6 aA           | 83.3 cA                               |
| % GPR <sup>c</sup>     | 5.1 cC               | 4.6 bB                         | 6.9 aB                         | 4.6 bC            | 5.8 bcBC                              |
| % GL <sup>c</sup>      | 11.0 bcB             | 8.4 bB                         | 8.2 bcB                        | 10.2 aB           | 9.0 cB                                |
| % BL <sup>c</sup>      | 3.8 bC               | 3.3 aB                         | 3.5 abC                        | 2.7 abD           | 1.9 cC                                |
| maturity               | early maturing       | midmaturing                    | very early maturing            | early maturing    | mid-maturing                          |

<sup>a</sup> Adapted from Tables 1 and 3 in ref 1. <sup>b</sup> Lodging = falling down in the field. <sup>c</sup> Average % tissue weights are given. The same lower case letters represent no statistical differences ( $P < 0.05$ ) among the five different sugar cane varieties for an individual tissue. The same upper case letters represent no statistical differences ( $P < 0.05$ ) among the three different tissue types for an individual variety. <sup>d</sup> S = stalk, GPR = growing point region, GL = green leaves, and BL = brown leaves.

and refining quality (5). The major aim of clarification is the maximum removal of suspended, turbid particles, and nonsucrose soluble impurities, e.g., proteins, polysaccharides, and inorganic materials, from juice to produce clarified juice with low turbidity. Most U.S. raw sugar factories now operate a hot lime clarification process (5). In hot lime clarification, the sugar cane juice is first heated, and natural flocs form by coagulation of colloids, particularly proteins. The heated juice is then flash heated to remove air bubbles and burst bagacillo (very small bagasse (fiber byproduct) particles), and reacted with milk of lime to form even larger flocs through calcium phosphate bridges. (Phosphate is present in the juice, and levels in Louisiana juices do not usually necessitate extra addition in the factory.) The created flocs subsequently precipitate in clarification tanks. Precipitation is aided by polyanionic flocculants which add weight to the flocs (bridge flocculation (6)).

An early study in 1948 (7) using old techniques reported that juice extracted from BL had a typical moldy odor, was highly turbid and colored, and had low sucrose, glucose, and fructose. A considerable portion of the solids were in a suspended state, and normal clarification aids (heat and lime, which are still used today) could not remove them. In contrast, GL were also very turbid and colored with chlorophyll, but the juices were easily clarified with heat and lime. Tops were observed to have physical characteristics very similar to those of the GL. However, today's ubiquitous use of flocculants for industrial clarification did not occur in 1948, and how they affect different trash components remains unknown.

The sugar cane variety harvested affects the quality and quantity of trash being delivered to the factory (8). Therefore, a study was undertaken to characterize differences in the physicochemical and processing properties of the trash and stalk tissue types from five sugar cane varieties (midseason), commercially grown in Louisiana. In the first part of the study (1) the total trash (GPR + GL + BL) content on a wet weight basis was reported to range from 16.4 to 19.8% and, generally, reflected BL sheath adherence. Before BL fell off the field plants, inorganic ash nutrients were reassimilated into the S. On a percent tissue weight basis, S and GL delivered the most total soluble polysaccharides, including starch, to the factory. In this second part of the study, differences in the clarification properties of the separated tissues are reported for the first time. Fundamental knowledge gained will increase understanding of trash effects on factory processing and use of trash in biomass utilization, as well as underpin solutions to alleviate detrimental effects of trash.

## MATERIALS AND METHODS

**Variety-Trash Trial: Field Experiment.** First ratoon (new stalks that grow from the plant base following the first annual harvest) sugar cane (five commercial varieties) was grown and then harvested on 17 November 2006 at the Ardoyne Farm of the USDA-ARS Sugar Cane

Research Laboratory, Schriever, Louisiana. Planting occurred on 26 August 2004; experimental design was a randomized complete block with four replications. Plots were cultivated and fertilized according to recommended practices; insecticides were applied as required. No chemical ripener was applied. This study included the two most common commercial varieties grown in Louisiana: LCP 85-384 and HoCP 96-540, and three newer commercial varieties with varying trash characteristics: L 97-128, L 99-226, and L 99-233 (Table 1). There were four replicates per variety; each replicate consisted of 25 randomly chosen hand-cut whole-stalks, with green and brown leaves still attached. Each sample was separated as follows: brown, dried leaves (BL); green leaves (GL); the growing point region (GPR that included immature apical internodes above a natural breaking point in the stalk); and the remaining stalk (S) composed of hardened internodes. Each tissue type was pooled by replicate and weighed. The S and GPR tissues from each replicate were separately passed through a prebreaker (Cameco Industries Inc., Thibodaux, Louisiana, U.S.). A shredded subsample (1000 g) was then passed through a core press (Cameco, U.S.) to extract juice and produce filter cake. The BL and GL were shredded by passing through a Jeffco cutter grinder (Jeffress Engineering Pty Ltd., Australia). The shredded GL were processed through the core press and the juice processed as described above. Shredded BL (100 g) was blended with 1 L of deionized water in a heavy duty blender (Waring Commercial, U.S.) for 10 min. Juice was obtained by pressing through a coarse sieve (600  $\mu\text{m}$ ). The remainder of the juice was treated with biocide (Bussan 881, Buckman Laboratories., U.S.), frozen, and subsequently transported to the USDA-ARS laboratory at the Southern Regional Research Center in New Orleans, Louisiana.

**pH.** The pH of the juice was measured immediately after extraction and before biocide was added on a Model SA 720 Orion pH meter at room temperature ( $\sim 25$  °C). The pH of clarified juices was also measured at 25 °C.

**°Brix (Percent Dissolved Solids).** The °Brix of samples was measured using an Index Instruments TCR 15-30 temperature controlled refractometer accurate to  $\pm 0.01$  °Brix. Results are expressed as an average of three measurements.

**Thermal Properties of Juices.** The thermal conductivity ( $\text{Wm}^{-1} \text{C}^{-1}$ ) and resistivity ( $\text{mCW}^{-1}$ ) were measured using a KD2 Thermal Properties Analyzer (Decagon, U.S.); accuracy  $\pm 5\%$ . The needle probe was held by a clamp to minimize vibrations and the needle inserted into the middle of a beaker (100 mL) of tissue or clarified juices. Results are expressed as an average of six measurements.

**Clarification Studies.** *Preparation of Composite Tissue Juices.* Juice (120 mL) from each tissue replicate was combined to form a composite juice.

*Laboratory Hot Lime Clarification.* Composite juice (500 mL) in a covered beaker was heated over a hot plate with constant stirring to 92 °C. Milk of lime (MOL; 10 Baumé) was added with stirring until the pH of the juice reached 6.7. The heated, limed juice was brought to a boil for 1 min to remove interfering bubbles, then flocculant (Stockhausen polyanionic) solution (0.1%) was added at 5 ppm using a pipet. The juice was immediately poured into a settling tube ( $5 \times 34$  cm) in a glass water bath (96 °C) to a volume of 400 mL and stoppered. Mud level readings were taken between 0 and 18 min, and also after 30 min of settling. The tube was then removed and the contents cooled to room temperature ( $\sim 25$  °C). A digital photograph of decanted clarified juice was taken with an Olympus

**Table 2.** Variation in the Thermal Conductivity and Resistivity of Juices Extracted from Different Tissues in Five Commercial Louisiana Sugarcane Varieties

| tissue <sup>b</sup>                                      | average values <sup>a</sup> |                 |          |                    |          | Av ± SD         |
|--|-----------------------------|-----------------|----------|--------------------|----------|-----------------|
|  | LCP 85-384                  | HoCP 96-540     | L 97-128 | L 99-226           | L 99-233 |                 |
| Thermal Conductivity (Wm <sup>-1</sup> C <sup>-1</sup> ) |                             |                 |          |                    |          |                 |
| S  | 0.530 <sup>c</sup>          | 0.550           | 0.503    | 0.520              | 0.538    | 0.528 ± 0.018 C |
| GPR  | 0.555                       | 0.555           | 0.550    | 0.540              | 0.543    | 0.549 ± 0.007 B |
| GL   | 0.550 <sup>c</sup>          | nd <sup>d</sup> | 0.558    | 0.660 <sup>c</sup> | nd       | 0.589 ± 0.061 B |
| BL <sup>e</sup>  | 0.597                       | 0.573           | 0.597    | 0.608              | 0.568    | 0.589 ± 0.017 A |
| Thermal Resistivity (mCW <sup>-1</sup> )                 |                             |                 |          |                    |          |                 |
| S  | 1.895                       | 1.993           | 1.995    | 1.928              | 1.868    | 1.936 ± 0.057 A |
| GPR  | 1.808                       | 1.815           | 1.820    | 1.848              | 1.838    | 1.826 ± 0.017 B |
| GL   | 1.850 <sup>c</sup>          | nd              | 1.795    | 1.680 <sup>c</sup> | nd       | 1.775 ± 0.087 B |
| BL <sup>d</sup>  | 1.723                       | 1.753           | 1.677    | 1.655              | 1.768    | 1.715 ± 0.048 C |

<sup>a</sup>  $N = 4$ . <sup>b</sup> S = stalk, GPR = growing point region, GL = green leaves, and BL = brown leaves. <sup>c</sup> The same upper case letters represent no statistical differences ( $P < 0.05$ ) among the four different tissue types for the thermal parameter. <sup>d</sup> nd = not determined. <sup>e</sup> Composite juice analyzed.

MIC-D digital microscope (Center Valley, U.S.). The mud settled at the bottom of the settling tube was also observed under the digital microscope. At least five random subsamples of each mud and clarified juice sample were photographed.

**Settling Rates and Mud Volumes (MV).** Settling rates and mud volume measurements and calculations were based on the methods of Schmidt (9) and Lionnet and Ravno (10) with modifications. Mud height (mL) was plotted against time (min). Break point (min) was the time it took for the juice to settle to half its original volume. The initial settling rate (ISR) in mL/min was determined graphically from the initial linear slope. MV after 18 (MV<sub>18</sub>) and 30 min (MV<sub>30</sub>) were read directly.

**Color and Turbidity of Clarified Juices.** The color and turbidity of clarified juices were measured as the absorbance at 420 nm and calculated according to the official ICUMSA method GS2/3-9 (1994). Samples (5 mL) were diluted in triethanolamine/hydrochloric acid buffer (pH 7) and filtered through a 0.45 μm filter.

**Factory Clarified Juices and Mud.** Factory clarified juices and mud were collected randomly, for comparison purposes, at two Louisiana raw sugar factories (Alma and Leighton) operating hot lime clarification in the 2007 processing season. Approximately 15 mL was collected every 30 s to form a composite clarified juice (250 mL).

**Statistical Analysis.** Following one-way ANOVA, mean comparisons were undertaken using PC-SAS 9.1.2 (SAS Institute, Cary, North Carolina) using Duncan's New Multiple Range Test.

## RESULTS AND DISCUSSION

**Thermal Properties of Juices from Trash and Stalk Sugar Cane Tissues.** The thermal properties of juices and sugar products in the factory are critical because many unit processes, including clarification, involve heat transfer. The thermal conductivity ( $k$ ) and resistivity ( $R$ ) properties of juices extracted from different sugar cane tissues have not been previously reported. Thermal conductivity is the ratio of heat flux density to temperature gradient in a material and a measure of the ability of a substance to conduct heat. Thermal resistivity is a measure of the ability of a substance to prevent heat flowing through it.

In this study, differences for average  $k$  and  $R$  values among the different tissue juices were small but, except for GPR and GL tissues, significant ( $P < 0.05$ ) (Table 2). For both  $k$  and  $R$ , the most variation occurred in the GL juices (Table 2).

There was a polynomial rather than a linear relationship between  $k$  and  $R$  ( $R^2 = -0.985$ ;  $y = 8.05x^2 - 12.09x + 6.04$ ) even though the instrument computed  $R$  as the reciprocal of  $k$ . However, the thickness and nature of the sample as well as the

**Table 3.** Variation in the pH of Juices Extracted from Different Tissues in Five Commercial Louisiana Sugarcane Varieties

| tissue <sup>b</sup> | average juice pH <sup>a</sup> |             |          |          |          |
|---------------------|-------------------------------|-------------|----------|----------|----------|
|                     | LCP 85-384                    | HoCP 96-540 | L 97-128 | L 99-226 | L 99-233 |
| S                   | 5.22 abcB <sup>c</sup>        | 5.18 cB     | 5.26 abB | 5.27 aB  | 5.21 bcB |
| GPR                 | 4.94 bC                       | 4.93 bC     | 5.04 aC  | 5.01 aC  | 4.95 bC  |
| GL                  | 5.27 bcB                      | 5.25 cB     | 5.33 aB  | 5.31 abB | 5.28 bcB |
| BL <sup>d</sup>     | 6.44 cdA                      | 6.91 abA    | 6.73 bcA | 7.08 aA  | 6.32 dA  |

<sup>a</sup>  $N = 4$ . <sup>b</sup> S = stalk, GPR = growing point region, GL = green leaves, and BL = brown leaves. <sup>c</sup> The same lower case letters represent no statistical differences ( $P < 0.05$ ) among the five different sugar cane varieties for an individual tissue. The same upper case letters represent no statistical differences ( $P < 0.05$ ) among the four different tissue types for an individual variety. <sup>d</sup> The pH of the brown, dried leaves is higher than those of the other tissues because it was the only tissue extracted with deionized water.

reciprocal conductivity can also contribute to thermal resistivity. As the °Brix and, therefore, the water content, varied in the different tissue types, i.e., for all tissues there was a negative, linear correlation between °Brix and  $k$  ( $R^2 = -0.733$ ) and °Brix and  $R$  ( $R^2 = 0.849$ ), this may have affected the resistivity. Water content, density, temperature, and composition of a material affect thermal conductance and resistivity ( $1/I$ ). Water has much higher thermal conductivity (0.57) than air (0.025) and organic matter (0.25), which is why the thermal properties of foods are often manipulated by changing the water or air content ( $1/I$ ). Another, more simple explanation for the lack of true reciprocity between  $k$  and  $R$  for some samples (Table 2) may be the experimental error associated with the instrument ( $\pm 5\%$  accuracy).

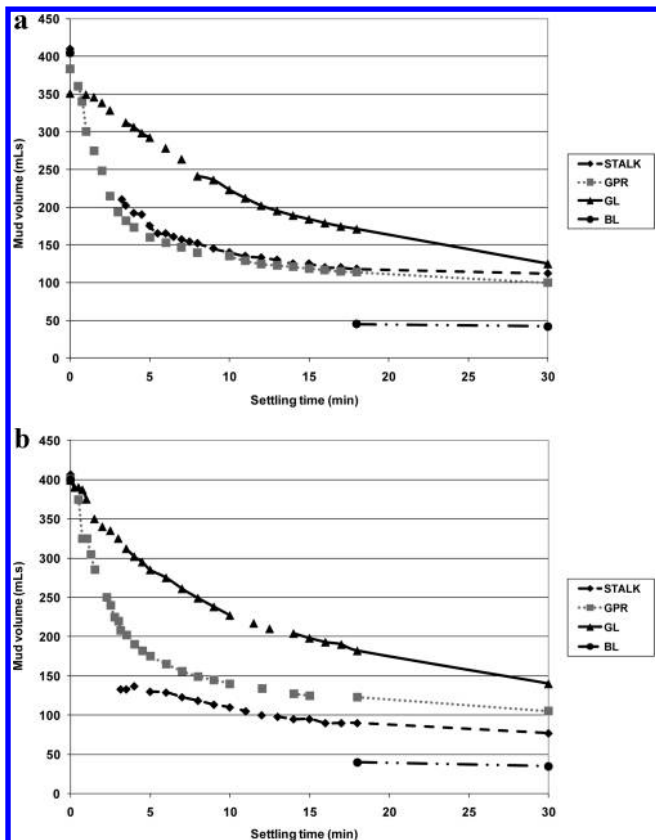
The thermal properties of the tissue juices were also compared to other juice quality parameters reported in the first part of this study (1). For all tissue types, there was no significant relationship between soluble ash and  $k$ , although ash affected the BL ( $R^2 = -0.426$ ) and particularly GL ( $R^2 = -0.913$ ) tissues (1). Total polysaccharides and starch (1) had no significant effect on  $k$  of the juice tissues.

**Juice pH.** The pH of sugar cane juice entering the factory affects processing and varies with time of season, variety, and environmental conditions. The pH of typical fresh sugar cane juice in Louisiana is ~5.4–5.5. The more acidic the juice, the more lime is required to neutralize and raise the juice pH before floc formation during the clarification process. Lime can also contribute to scale formation in the evaporator bodies. Even though differences in juice pH values among the five varieties for each type of tissue were small, some significant ( $P < 0.05$ ) differences still existed (Table 3). GPR juice had the lowest ( $P < 0.05$ ) pH values, but no significant differences existed between the juice pH values of S and GL (Table 3). Eggleston et al. (12) similarly observed that the lowest pH values occurred in the GPR, the most physiologically active tissue. The significantly ( $P < 0.05$ ) higher pH values of L 97-128 and L 99-226 GPR juices may reflect the very early and early maturing characteristics (Table 1) of these varieties, respectively.

As expected, the pH of BL juice was significantly ( $P < 0.05$ ) higher (Table 3) than that of all other tissues because of the pH contribution from the extraction water (the other tissues were obtained after hydraulic pressing). However, it has been reported (13) that sugar cane juice pH values were generally higher with added BL than GL (the leaves were physically combined with billeted sugar cane stalks and juice pressed from the mix). Higher BL pH values may be because of decreased acids, particularly aconitic acid that occurs in leaves and tops (7, 14), or the buffering capacity of BL.

**Clarification Settling Performance of Juice from Different Sugar Cane Tissues.** In this study, all tissue juices were clarified by a





**Figure 1.** Settling performances of juice extracted from separated tissues of sugar cane varieties (a) HoCP 96-540 and (b) L 99-233.

laboratory, hot lime clarification process. In general, because of their lower pH values (Table 3), slightly more lime had to be added to GPR juices than that for the other tissue juices to obtain a heated, limed juice pH of 6.7 at 92 °C.

Typical settling profiles of two varieties (HoCP 96-540 an established commercial variety and L 99-233 a new variety) are illustrated in Figure 1a,b. Significant ( $P < 0.05$ ) differences in the average settling performance of the different tissues were observed (Table 4). In contrast, because composite tissue juices were studied for clarification characteristics, differences among varieties were less significant than those among tissues. Only GPR and GL foamed on heating and followed settling behavior similar to that of the factory sugar cane juice, although GL was markedly slower than GPR (Table 4 and Figure 1). Greater than 25% mud volume usually means a drastic decrease in settling rate (15), which explains the slower initial settling rate (ISR) and break point (BP) for GL than GPR. This was confirmed by the strong correlation between  $MV_{30}$  and ISR ( $R^2 = 0.840$ ) for GL compared to no significant correlation for GPR. The larger flocs for GPR than GL would also have contributed to the faster settling rate (Table 4). GL and particularly GPR are the most physiologically active sugar cane tissues and contain the most enzymes. The enzymes and other proteins would coagulate on heating and markedly contribute to natural floc formation, which most likely explains why both of these tissues followed normal settling. The slower but not significantly different (Table 4) settling of GL compared to that of GPR may also be explained by the much higher amount of polysaccharides occurring in the GL than in the GPR tissues (1). Scott et al. (16) observed that the addition of 1% tops or trash to stalk juice did not influence the ISR of juice. Unfortunately, Scott et al. (16) did not define trash fully; therefore, it is unknown if the trash was mostly GL or BL.

More variation occurred for the settling and mud characteristics of the GL juice compared to the other tissue juices (Table 4). This may reflect the highest variation in thermal properties occurring for juice from GL (Table 2) and the markedly higher polysaccharide content of the GL juice (1). This suggests that there is a stronger varietal effect for GL on clarification characteristics than the other tissues.

Of all the four tissues, the GPR followed the typical settling of factory sugar cane juice (not shown) (9) the most (Figure 1). For GPR, there was a strong, linear relationship ( $R^2 = 0.994$ ) between ISR and BP, which was slightly higher than that for GL ( $R^2 = 0.847$ ). The average ISR for GPR ( $93.5 \pm 31.6$  mL/min) fell within the range expected for factory sugar cane juice (91–105 mL/min (5)). This strongly indicates that GPR juice aids the industrial clarification of sugar cane juice, although the average GPR BP was ~3-fold slower than that for factory juice, which suggests that GPR juice does not fully govern the settling behavior of factory juice. These results most likely explain why sugar cane juice is difficult to industrially clarify when the GPR is damaged after (i) the application of chemical glyphosate ripener (17) or (ii) a freeze. This warrants further investigation.

Even though S juice produced the largest flocs (Table 4), it tended to thin out rather than follow normal settling (Figure 1). The most unwanted upward motion of flocs was also observed for S. Therefore, although the flocs were larger, they were either too low in density or did not have optimum charge distribution (6), or contained too many thermal gradient distributions to follow normal settling. Because of the low °Brix of the BL juice, settling behavior could not be determined, although final mud volumes could (Table 4).

Tissue juice °Brix had no relationship with the mud volume produced (Table 4). After 30 min of settling, mud volume per unit tissue juice °Brix varied significantly ( $P < 0.05$ ) among the tissues: S produced the lowest value (1.09 mL/°Brix) and BL the highest (11.3 mL/°Brix). GPR (3.0 mL/°Brix) and GL (3.1 mL/°Brix) values were similar. Scott et al. (16) observed that the addition of 1% tops or trash (GL and BL were not differentiated) to stalk juice increased mud density slightly, but significantly. Overall, these results strongly indicate that it is the quality rather than the quantity of impurities in the different tissue that mostly affects the volume of mud produced. Too much mud is detrimental to factory operations because if it is allowed to cool, then it can become easily infected by microbes such as *Leuconostoc* bacteria, which, in turn, can cause losses of sucrose and the production of CO<sub>2</sub>. Moreover, the mud filter station can become overloaded.

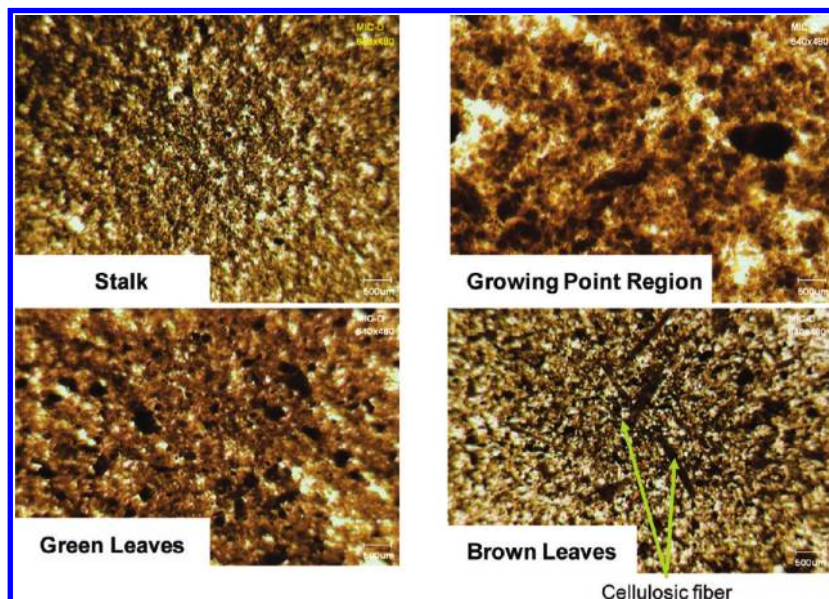
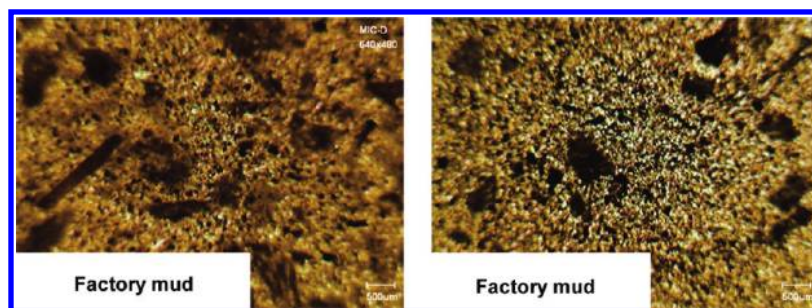
Typical micrographs of mud produced from the different tissues are illustrated in Figure 2 and can be compared with composite factory muds in Figure 3. Mud is a suspension of soil, scum, flocs, bagacillo, salts, and juice, and contains wax and large quantities of polysaccharides and proteins (18). Mud produced from S was visually light brown in color and clean of all dark impurities and cellulosic material. BL produced very dark brown mud with numerous cellulosic fibers visible. Both GL and GPR were dark brown in color and contained dark impurities (Figure 2). Little fibrous (mostly cellulosic) material was apparent in the GL or GPR mud, with GPR containing slightly more than GL (Figure 2). As expected, the typical digital micrographs of factory muds (Figure 3) contained particles seen in the micrographs of all four tissue types (Figure 2).

**Clarified Juice pH, Turbidity, and Color.** Average clarified juice (CJ) pH values at 25 °C with standard deviations were as follows: S,  $7.04 \pm 0.17$ ; GPR,  $6.70 \pm 0.03$ ; GL,  $6.67 \pm 0.24$ ; and BL,  $6.93 \pm 0.14$ . The lower CJ pH values for GPR and GL most likely reflect their better settling behavior as precipitation of basic

**Table 4.** Average Values with Standard Deviation of Settling Characteristics of Juices Extracted from Different Tissues of Five Commercial Sugarcane Varieties<sup>a</sup>

| tissue <sup>b</sup> | initial juice °Brix       | BP <sup>c</sup> min | MV <sub>18</sub> <sup>c</sup> % | MV <sub>30</sub> <sup>c</sup> % | ISR <sup>c</sup> mL/min | floc size and foam formation       |
|---------------------|---------------------------|---------------------|---------------------------------|---------------------------------|-------------------------|------------------------------------|
| S                   | 18.9 ± 1.8 A <sup>d</sup> | na <sup>e</sup>     | 25.6 ± 5.9 B                    | 20.6 ± 3.7 B                    | na                      | small to large flocs; no foam      |
| GPR                 | 8.8 ± 0.2 B               | 3.4 ± 0.4 A         | 30.6 ± 0.6 AB                   | 26.6 ± 0.5 AB                   | 93.5 ± 31.6 A           | small to medium flocs; foamed      |
| GL                  | 9.9 ± 1.2 B               | 10.8 ± 7.2 A        | 38.7 ± 12.7 A                   | 30.6 ± 9.0 A                    | 46.5 ± 43.9 A           | very small to medium flocs; foamed |
| BL                  | 0.7 ± 0.1 C               | na                  | 8.9 ± 2.5 C                     | 7.9 ± 2.6 C                     | na                      | very small; no foam                |

<sup>a</sup> N = 5. <sup>b</sup> S = stalk, GPR = growing point region, GL = green leaves, and BL = brown leaves. <sup>c</sup> BP = break point, the time it took for the juice to settle to half of its original volume; MV<sub>18</sub> = mud volume after 18 min of settling; MV<sub>30</sub> = mud volume after 30 min of settling; and ISR = initial settling rate. <sup>d</sup> The same upper case letters represent no statistical differences ( $P < 0.05$ ) among the four different tissue types for a clarification parameter. <sup>e</sup> na = not applicable.

**Figure 2.** Digital micrographs of settled mud after clarification of separated tissues from sugar cane variety HoCP 96-540.**Figure 3.** Typical digital micrographs of settled mud from a Louisiana sugar cane factory operating hot lime clarification (November 2007). The micrograph on the left illustrates a sample thicker than the one on the right.

salts into the mud is a major contributor to the drop in pH across the clarification tank. Inversion of sucrose with associated production of acids could also have contributed to this. The standard deviation values for CJ pH values were highest in GL, which reflects their highest variation in settling characteristics (Table 4).

The major aim of clarification in raw sugar manufacture is the maximum removal of suspended and turbid particles from juice to produce clarified juice of low turbidity. The turbidities of CJ's from different tissues varied markedly with variety and are illustrated in Figure 4a. On average, CJ turbidity was the lowest from GL (av.  $378 \pm 399$  ICU; ICU = ICUMSA units) and S ( $1052 \pm 500$  ICU), with CJ from GPR being more turbid ( $3008 \pm 1443$  ICU). The high standard deviation for GPR CJ av. turbidity was because of the much higher value for L 99-233. Without the L 99-233 outlier, the av. GPR CJ turbidity would have been  $2180 \pm 218$  ICU. BL tissue from all five varieties consistently produced a

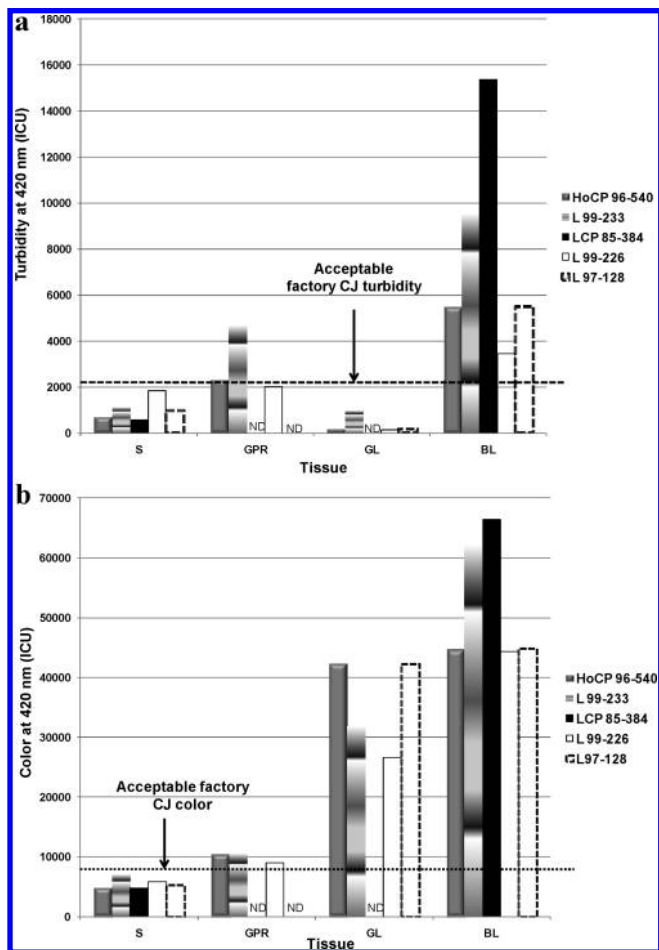
highly turbid CJ ( $7866 \pm 4751$  ICU). This strongly suggests that hot lime clarification is unable to remove all suspended fibrous particles from BL tissue. A very acceptable turbidity for a factory CJ after hot lime clarification is  $\sim 2100$  ICU (5). Scott et al. (16) observed that the addition of 1% tops to stalk juice increased the turbidity of clarified juice by 2.8%. The effect of adding 1% trash (GL and BL were not differentiated) was worse with turbidity increasing by 4.2%.

Green sugar cane harvesting has been reported to increase the color of raw sugar and decrease the recovery of sucrose across the factory boiling house (19–21). The color of clarified juices from GL and BL were markedly higher than that from GPR and S, following the decreasing order: BL > GL  $\gg$  GPR  $\gg$  S (Figure 4b). It was surprising that BL contributed markedly to color, on average even more than GL, although the color of CJ's from the GL and BL of L 97-128 and 384 were very similar (Figure 4). Ivin and Doyle (22) observed that compared to green



tops, brown leafy trash contributed more to CJ color but that there was a much greater influence from the variety and farm

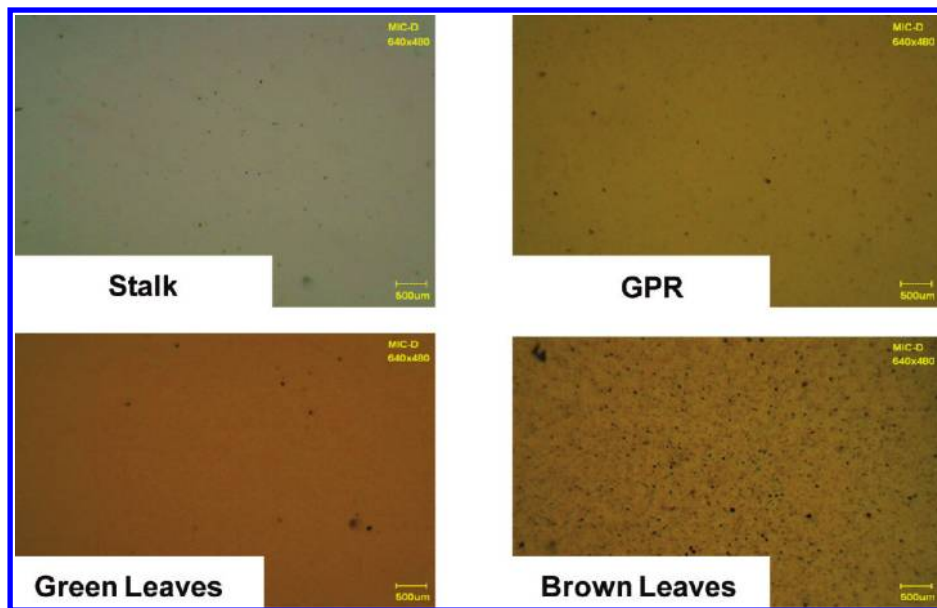
location of the sugar cane. Therefore, leaf components of sugar cane contribute the most to CJ color. Similarly, Scott et al. (16) observed that although both tops and trash (GL and BL were not differentiated) increased CJ color, leaf trash was worse than tops. The addition of 1% tops to stalk juice increased the color of clarified juice by 1.3%; in contrast, the addition of 1% trash increased color by 3.6%.



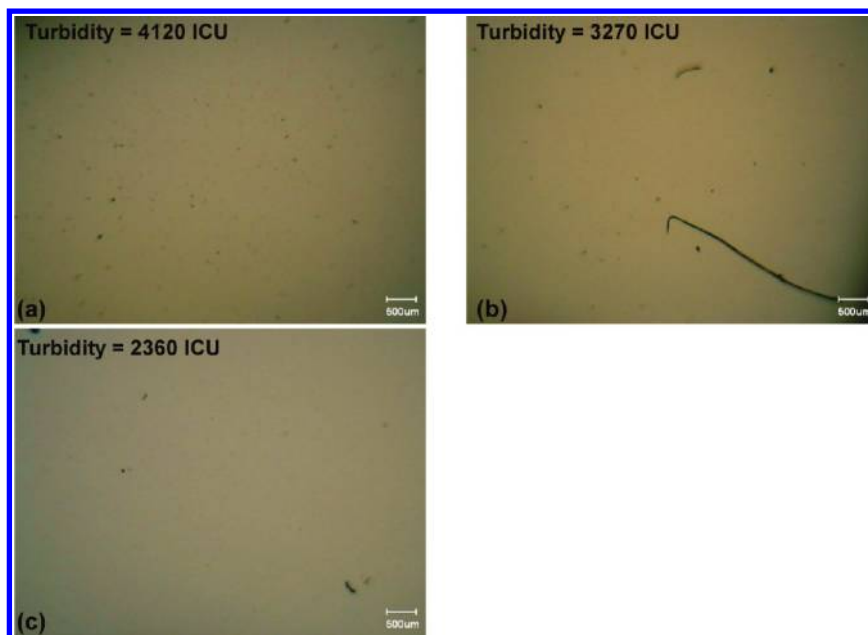
**Figure 4.** (a) Turbidity values of clarified juices produced from the separated tissues of five Louisiana sugar cane varieties. The acceptable turbidity value for Louisiana factory clarified juices is from ref 5. (b) Color values of clarified juices produced from the separated tissues of five Louisiana sugar cane varieties. The acceptable color value for Louisiana factory clarified juices is from ref 5. ND = not determined.

**Digital Micrographs of Clarified Juices.** Typical digital micrographs of CJ's produced from the different tissues are illustrated in **Figure 5** and can be compared to composite factory CJ's in **Figure 6**. A large range in CJ microparticle contents was observed among the various tissues (**Figure 5**). Visually, the CJ's with the highest color occurred from GL and BL, which agrees with the ICU color results (**Figure 4b**). CJ produced from S contained the lowest color and the least amount of dark particles. Nevertheless, microparticles were still present, and the amount varied with sugar cane variety (**Figure 5**). CJ's from GL varied markedly with variety, with some (e.g., from L 97-128) relatively clean of particles, whereas others contained small to large microparticles. This reflects the highest variation in settling characteristics for the GL juice (**Table 4**) and further suggests a stronger GL varietal effect for clarification characteristics than the other tissues. However, further studies are now required for confirmation. CJ's from GPR tissue also varied markedly with variety (**Figure 5**).

Of all the tissues, CJ micrographs from BL were the most consistent among varieties. These always contained numerous suspended particles with some containing medium to large microparticles and/or cellulosic particles (**Figure 5**). This confirms that hot lime clarification is unable to remove all BL fibrous/cellulosic material. Balch and Broeg (7) observed that heat and lime could not remove BL suspended particles. At the factory, less BL tissue will be processed than in these separated tissues (the sugar cane varieties in this study contained 1.9–3.8% BL on a tissue weight basis (1)), but indications are that even a small amount cannot be removed. This was further evidenced in composite factory CJ's (**Figure 6**), where small microparticles still exist even in very acceptable CJ's, i.e., turbidity < 2400 ICU (**Figure 6c**), and cellulosic particles are sometimes present (**Figure 6c**). Turbo filtering (23) of the CJ, rather than the typical filtering with static, mesh screens in sugar cane factories, would aid the removal of cellulosic and colloidal particles that con-



**Figure 5.** Digital micrographs of clarified juices from separated tissues of sugar cane variety HoCP 96-540.



**Figure 6.** Digital micrographs of clarified juices (hot lime clarification) from (a) Alma factory, 14 November 2007, (b) Alma factory, 14 November 2007, and (c) Leighton factory, 24 October 2007.

tribute to unwanted high CJ turbidities. Turchetti (23) reported that turbo filters remove much smaller particles (up to 1  $\mu\text{m}$ ) than mesh screens, and 150 turbo filters have now been installed in sugar cane factories and distilleries in Brazil.

Overall, the results suggest that hot lime clarification was unable to remove all BL cellulosic particulate material. Increased color, turbidity, and suspended particles in clarified juices obtained from GL and BL are delivered to the factory. This will cause difficulty downstream in the factory boiling house, particularly in the low grade raw sugar strikes, and make the future attainment of very high pol (VHP) raw sugar more difficult (there is a current trend in the U.S. to produce such higher grade raw sugars for the new refineries). Consequently, strategies to reduce the processing of green and, especially, brown leaves at the factory need to be urgently identified and implemented. The optimization of factory unit processes (i.e., turbo filtering of clarified juice) is not only needed to alleviate problems associated with extra trash but also to identify and implement economical strategies to reduce the amount of green and brown leaves processed at the factory. Trash removal can occur in the field or at the factory before processing. The ground and extraction fan speed of the combine harvester governs the amount of trash blown off in the field (24) as well as the setting of the top cutter. Trash separation technologies at the factory are available (25), including dry cleaning before the sugar cane is shredded. However, questions still remain on how efficient trash separation technologies perform, while *not* removing valuable sucrose from the stalks when trash is removed (24). Furthermore, excessively large piles of trash will be created at the factory that will have to be utilized (e.g., cogeneration of electricity), marketed, or disposed of quickly. Such trash piles would be an excellent source of biomass (1).

**Thermal Properties of Clarified Juices from Different Sugar Cane Tissues.** Thermal conductivity ( $k$ ) and resistivity ( $R$ ) of the CJ's, listed in Table 5, would be expected to affect the heat transfer properties of downstream sugar products such as syrups, massecuites, and molasses (26). Except for CJ's from S, for all other tissue CJ's  $k$  and  $R$  values increased and decreased, respectively, when compared to those of their tissue juices (Table 2), although these changes were small. Moreover, there was more variation in the thermal properties of CJ's compared to

**Table 5.** Variation in the Thermal Conductivity and Resistivity of Clarified Juices Obtained after Hot Lime Clarification of Tissue Juices Extracted from Five Commercial Louisiana Sugarcane Varieties

| tissue <sup>a</sup>                                     | composite values |             |                 |          |          | Av $\pm$ SD       |
|---|------------------|-------------|-----------------|----------|----------|-------------------|
|   | LCP 85-384       | HoCP 96-540 | L 97-128        | L 99-226 | L 99-233 |                   |
| Thermal Conductivity ( $\text{Wm}^{-1} \text{C}^{-1}$ ) |                  |             |                 |          |          |                   |
| S   | 0.507            | 0.518       | 0.402           | 0.503    | 0.512    | $0.488 \pm 0.049$ |
| GPR   | 0.525            | 0.758       | nd <sup>b</sup> | 0.688    | 0.793    | $0.691 \pm 0.119$ |
| GL  | 0.530            | 0.610       | 0.572           | 0.590    | 0.649    | $0.590 \pm 0.044$ |
| BL  | 0.613            | 0.868       | 0.753           | 0.580    | 0.443    | $0.651 \pm 0.163$ |
| Thermal Resistivity ( $\text{mCW}^{-1}$ )               |                  |             |                 |          |          |                   |
| S   | 1.977            | 1.935       | 1.983           | 2.02     | 1.972    | $1.977 \pm 0.030$ |
| GPR   | 1.913            | 1.325       | nd              | 1.453    | 1.260    | $1.488 \pm 0.294$ |
| GL  | 1.883            | 1.653       | 1.754           | 1.700    | 1.473    | $1.693 \pm 0.150$ |
| BL  | 1.635            | 1.153       | 1.327           | 1.733    | 2.253    | $1.620 \pm 0.424$ |

<sup>a</sup> S = stalk, GPR = growing point region, GL = green leaves, and BL = brown leaves. <sup>b</sup> nd = not determined.

that of the tissue juices (compare Tables 2 and 5). These differences most likely reflect the added inorganic lime and polyanionic flocculant during clarification.

The lowest  $k$  and  $R$  values occurred in the CJ's produced from S juice (Table 5) because of the higher °Brix contents. The most variation for both thermal parameters occurred in CJ's from BL, with considerable variation also in CJ's from GPR (Table 5). This is in contrast to the tissue juices, where the most variation occurred in the GL (Table 2). At this present time, we have no explanation for this, but it may in some way be related to the wide differences in settling performance of the various tissues.

#### ABBREVIATIONS USED

BL, brown, dried leaves; GL, green leaves; GPR, growing point region; S, stalk; CJ, clarified juice; VHP, very high pol sugar;  $k$ , thermal conductivity;  $R$ , thermal resistivity; HMW, high molecular weight; MOL, milk of lime; MV<sub>18</sub>, mud volume after 18 min of settling; MV<sub>30</sub>, mud volume after 30 min of settling; BP, break point; ISR, initial settling rate; ANOVA,

analysis of variance; ICUMSA, International Commission for Uniform Methods in Sugar Analysis; ICU, ICUMSA units.

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