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Audubon Sugar Institute
Factory Operations Seminar

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MILLING AND BOILER TEST RESULTS - 2018

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INTRODUCTION

As in past years, mill performance tests and boiler efficiency and compliance tests were conducted at the request of individual Louisiana factories. Emphasis continues to be placed on good mill work and boiler efficiency. Results from testing over the 2018 grinding season follow.

MILLING

Three mill performance tests were requested during the 2018 season. Results provided to factories included preparation index, cane pol and fiber, bagasse pol and moisture, actual pol extraction, and reduced pol extraction. Routine factory methods of analyses were used.

Table 1. Prepared cane and bagasse analyses for all mill tests.

	High	Low	Average
Preparation Index	88.85	78.28	82.26
Pol % Cane	13.18	10.67	12.14
Fiber % Cane	13.19	12.62	12.87
Pol % Bagasse	2.87	1.76	2.14
Moisture % Bagasse	52.58	47.33	50.14
Actual Pol Extraction, %	95.43	93.51	94.69
Reduced Pol Extraction, %	95.56	93.90	94.88

Table 1 shows prepared cane and bagasse analyses along with actual and reduced tandem pol extractions. The preparation index averaged 82% with a range of 78 to almost 89%. Pol % prepared cane ranged from 10.67 to 13.18 and averaged at 12.14. True fiber % prepared cane averaged 12.87 with a low of 12.62 and a high of 13.19. Bagasse moisture varied from a low of about 47% to a high of 52.6% and averaged 50%. Pol % bagasse averaged 2.14 and varied from 1.76 to 2.87. The overall tandem pol extraction averaged 94.69%, ranging from 93.5% to 95.4%. Reduced pol extraction varied from 93.90% to 95.56 % and averaged 94.88%.

Table 2 lists the averages for the past five crops in each category shown. Preparation index was shown to increase over the past 2 years and is now above 80. As expected, pol % cane and fiber % cane varied year by year. The pol % bagasse is slowly improving while the moisture % bagasse remains steady around 50%. Actual and reduced pol extractions have remained in the 94-95 range even with increasing grinding rates.

Table 2. Yearly averages of prepared cane and bagasse analyses.

	2018	2017	2016	2015	2014
Preparation Index	82.26	81.43	77.22	75.74	78.03
Pol % Cane	12.14	12.93	13.72	12.20	12.63
Fiber % Cane	12.87	12.94	14.43	12.90	12.82
Pol % Bagasse	2.14	2.18	2.48	2.22	2.26
Moisture % Bagasse	50.14	50.38	49.52	51.73	52.04
Actual Pol Extraction, %	94.69	94.60	94.34	93.80	94.27
Reduced Pol Extraction, %	94.88	94.84	95.16	93.99	94.41

BOILERS

This past crop (2018), assistance was given to five factories requiring compliance testing of specific bagasse boilers as permitted by the Louisiana Department of Environmental Quality. Another factory also requested assistance in determining the efficiency of all five of their bagasse boilers. In all, twenty-two boiler tests were conducted. Operating conditions and results provided included preheated air temperature, flue gas temperature, bagasse moisture, bagasse ash, oxygen % flue gas, excess air %, efficiency % and pounds steam produced per ton of bagasse burned (Table 3). The effective moisture was also calculated and is shown in Table 3.

Table 3 gives the results of all boiler tests conducted during the 2018 crop. Preheated air temperature varied from 265 to 608 °F and averaged at 452 °F. Flue gas temperatures ranged from a low of 334 °F to a high of 514 °F and averaged at 413°F. Bagasse samples were collected during each test run and analyzed for moisture and ash. Moisture % bagasse averaged at 50.53 (ranging from 45.42 to 53.39) while the ash % bagasse averaged 5.17 (and ranged from 3.09 to 10.89). Because bagasse fuel content is so critical to boiler efficiency, it is useful to look at its effective moisture (which is the moisture taking the ash into account). The effective moisture % bagasse averaged 53.29 or about 2.8% higher than the regular moisture. Oxygen levels varied from 5.4% to 11.8% and averaged 8.55%. Excess air levels ranged from 26% to 129% and averaged 71%. Boiler efficiency ranged from 53% to 65% and averaged 61%. Each pound of bagasse burned produced between 1.94 and 2.57 pounds of steam and averaged 2.24 for the crop. This number has increased since the early days of boiler testing when the average pounds of steam produced per pound of bagasse burned hovered around 2.00.

Overall, Mill extraction and boiler efficiency continue to improve even as many factories process more cane.

Table 3. Temperatures and results of bagasse boiler testing.

Factory	Preh. Air, °F	Flue Gas, °F	Moist. % Bag.	Ash % Bag.	Effec. Moist., %	O_2, %	Excess Air, %	Effic., %	Lbs Steam/ Lb Bag.
A	550	498	51.05	4.25	53.32	5.40	25.95	53.13	2.04
A	550	500	51.05	4.25	53.32	5.60	34.85	59.42	2.28
A	528	477	51.05	4.25	53.32	7.30	52.79	59.95	2.30
A	520	514	51.05	4.25	53.32	7.50	53.46	57.08	2.19
A	528	457	51.05	4.25	53.32	7.90	59.52	60.04	2.30
A	504	474	51.05	4.25	53.32	8.55	67.52	58.51	2.25
A	608	439	51.05	4.25	53.32	8.65	69.73	60.45	1.95
F	325	401	49.13	3.70	51.02	10.58	101.21	62.63	2.48
F	325	402	47.98	6.68	51.41	10.31	96.10	62.74	2.39
F	325	398	48.67	3.15	50.25	10.30	95.94	63.64	2.57
H	530	417	52.67	4.38	55.08	8.09	62.33	61.68	2.14
H	537	422	51.55	3.09	53.19	7.91	60.10	62.91	2.31
H	541	429	53.39	4.35	55.82	7.80	58.77	60.83	2.08
K	265	367	48.33	4.48	50.60	11.83	128.71	60.97	2.41
K	290	382	45.42	10.89	50.97	11.77	127.17	60.59	2.22
K	290	384	48.90	4.47	51.19	11.46	119.77	60.91	2.38
N	512	382	51.16	5.52	54.15	8.15	63.08	61.94	2.21
N	514	365	51.37	5.60	54.42	8.14	62.96	62.39	2.21
N	509	361	47.98	6.30	51.21	8.17	63.36	64.51	2.43
S	400	339	52.39	5.09	55.20	7.28	52.76	64.93	2.17
S	393	334	52.33	8.72	57.33	7.90	59.99	63.45	1.94
S	395	337	53.00	7.58	57.35	7.50	55.23	63.57	1.96
Average:	**452**	**413**	**50.53**	**5.17**	**53.29**	**8.55**	**71.42**	**61.19**	**2.24**

MANNITOL KITS AT THE FACTORY – WHAT THEY CAN DO FOR YOU

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INTRODUCTION

The Louisiana sugarcane industry has always been susceptible to the bacterial deterioration of cut or injured cane due to its prevalent humid and warm conditions. Deteriorated sugarcane can lead to processing difficulties, expensive sugar losses, and penalties for high dextran concentrations in raw sugars. Furthermore, in late season, Louisiana is susceptible to freezes and associated bacterial infections.

For well over 100 years the sugar industry had considered dextran, a viscous glucopolysaccharide, as the major deterioration product of the bacterial (mainly *Leuconostoc*) deterioration of sugarcane. In the past 15 years, however, it has increasingly been known that mannitol, a sugar alcohol, is also a major deterioration product of such lactic acid bacterial degradation (Figure 1). This has revolutionized our understanding of deterioration and how it is analyzed, since dextran has always been difficult to measure and control due to methods being either too slow, too complicated, not specific, or too expensive (Eggleston and Huet, 2012).

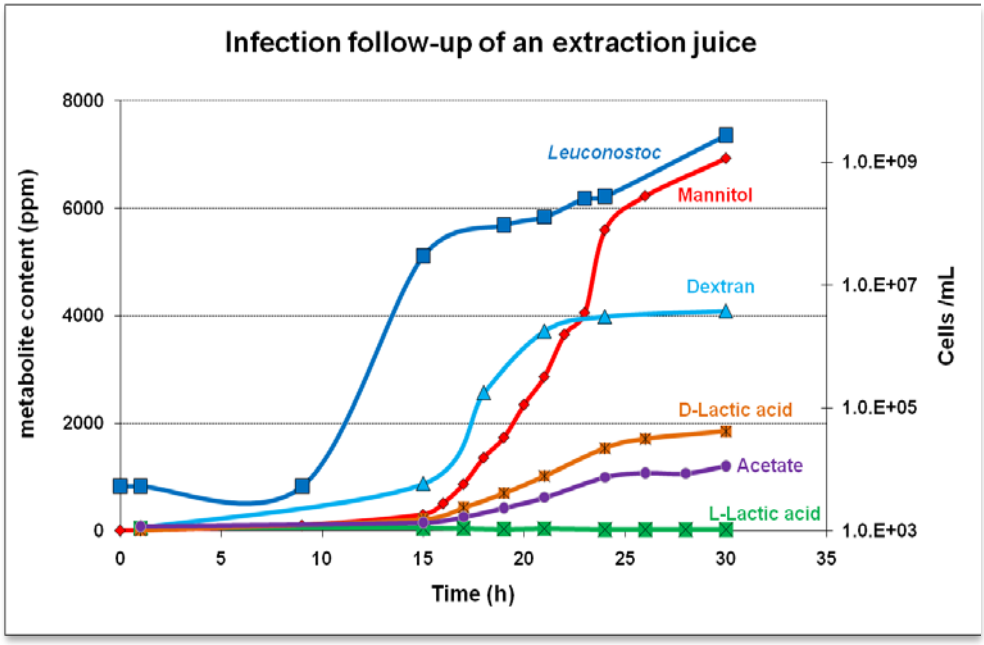


Figure 1. Growth profile of *Leuconostoc* bacteria in sugar crop juice at room temperature with associated metabolite (deterioration products) profiles. From Eggleston and Huet (2012).

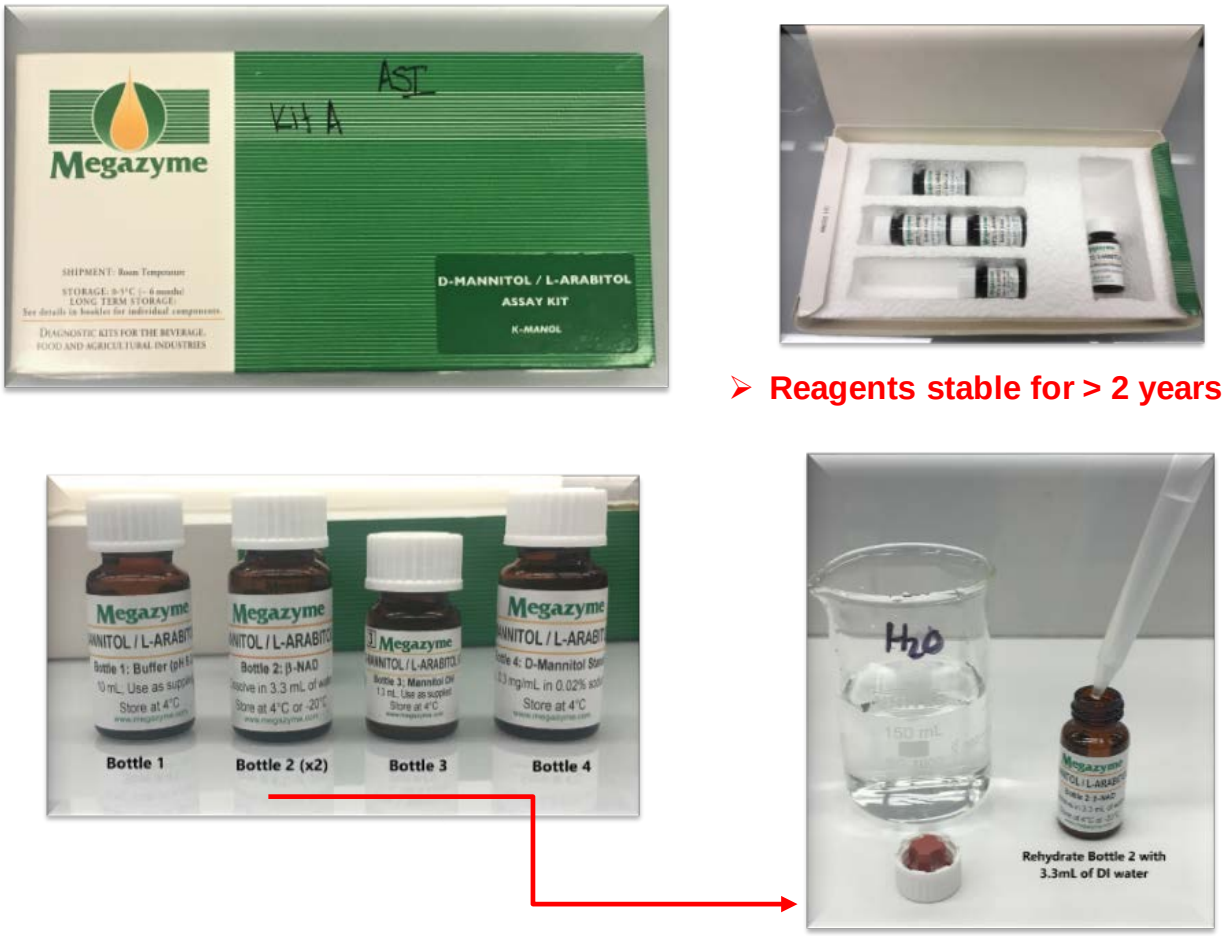
Factory enzymatic mannitol methods, which are available in kit form, can rapidly, simply and inexpensively measure mannitol as a marker for (i) sugarcane, sugar beet, and sweet sorghum deterioration, (ii) the bacterial contamination of fuel ethanol produced from sugarcane products, and (iii) for lactic acid bacterial infections in factories and refineries. Most sugar beet factories in Europe now measure mannitol as well as numerous other factories/distilleries around the world. Breeders also now measure mannitol to identify and develop cold tolerant sugarcane varieties.

Mannitol is a small sugar alcohol that does not evaporate during sugar processing and is very stable. Mannitol does incorporate into sugar crystals but not to the degree of dextran. As the concentration of mannitol increases in syrup, however, then the more mannitol occurrence increases in sugar (Eggleston et al. 2012). At high concentrations (<20,000 ppm/Brix in refined sugar liquors) mannitol can cause conglomeration of crystals and co-crystallize with sucrose forming needle crystals that can look like dextran elongated crystals (Eggleston et al. 2012).

The ability of factory personnel to rapidly screen individual consignments of sugarcane for deterioration as well as sugarcane products across the factory would be beneficial for improving overall manufacturing efficiency. An inexpensive, reliable, and rapid enzymatic method to measure mannitol levels in sugarcane juices was developed by Eggleston and Harper in 2006 and modified by Eggleston in 2009. The method utilizes mannitol dehydrogenase (MDH) to convert mannitol to fructose in the presence of the co-enzyme nictonamide-adenine-dinucleotide (NAD). The NADH formed is directly related to the amount of mannitol in the cane product and is easily measured at 340 nm using a factory spectrophotometer. In 2010, this method became ICUMSA GS8-12 method (Eggleston and Huet, 2012) and is the basis of a Biosentec® kit that is now being ubiquitously used in the European sugar industry (approximately US$4.65 per analysis). The intent of the original method, however, was for users to purchase their own chemicals to keep the cost down to ~US$0.65 per analysis. Unfortunately the source of MDH enzyme was from abroad and not readily available in Louisiana. Now, another new kit, based on the enzyme method, is being manufactured by Megazyme® (used to measure mannitol in urine; Anon. 2018) and used in the Colombian sugarcane industry. This kit cost is ~US$4.00 per analysis but has the advantage of being easily purchased and delivered to Louisiana within 2 to 3 days. This kit was evaluated at the Audubon Sugar Institute in 2018 and used at Louisiana factories during the 2018 grinding season, which is described herein.

EXPERIMENTAL

The Megazyme® kit arrives in a small box containing five bottles. All contents of the bottles are used *as is* except for bottle 2 (NAD solution) which requires the addition of 3.3 mL of distilled water (Figure 2).



➢ **Reagents stable for > 2 years**

Figure 2. The Megazyme® enzymatic mannitol kit contents (see APPENDIX I for full method).

Some adjustments to the Megazyme® method were necessary since Eggleston and Harper (2006) had previously shown that cane sample particles interfere with the enzymatic assay. Fortunately, it was found that centrifuged juice from the factory core laboratory could be used. The whole kit instructions were written up as a method for the factories (see APPENDIX I), and laminated copies were given to ten Louisiana factories along with a "start-up" Megazyme® kit.

The linearity of the method (Figure 3) was shown to be excellent and the precision was very acceptable at less than 3.5% (Figure 4). Precision was worse at lower concentrations of mannitol which has also been previously reported (Eggleston and Harper, 2006; Eggleston and Huet, 2012). Another real advantage of the method is that no standard curve or check standards are necessary. Results in Table 1 show that the calculated mannitol concentrations were as accurate as the ones obtained using a standard curve, and were slightly higher than those obtained from the standard curve. Overall, it was found to be a very easy method and accurate to within ± 3.9%.

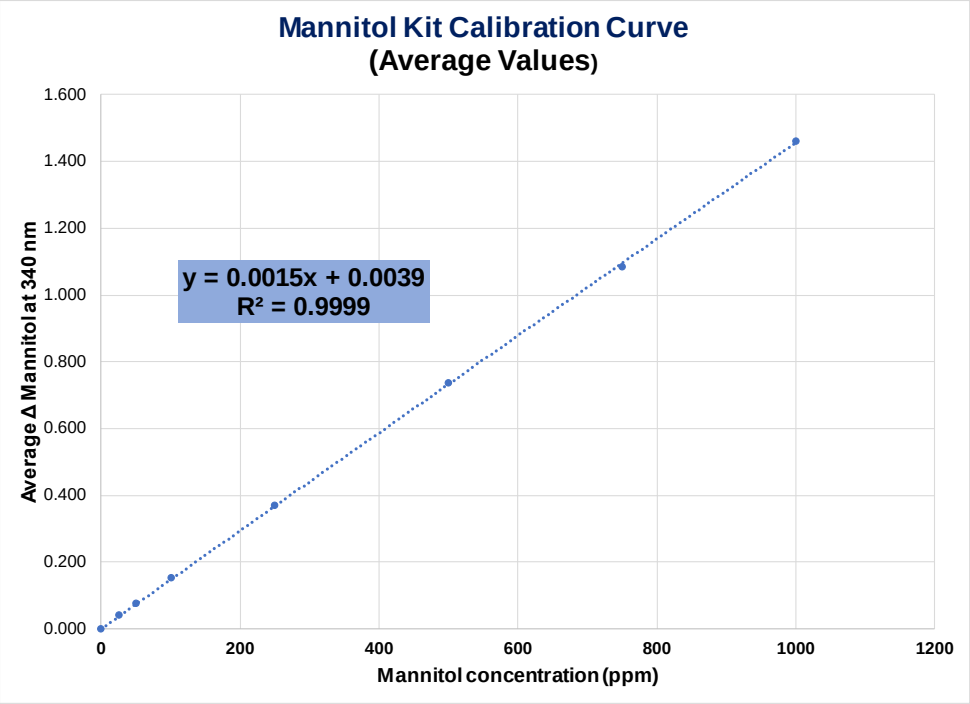


Figure 3. Excellent linearity of the mannitol kit following the method described in APPENDIX I.

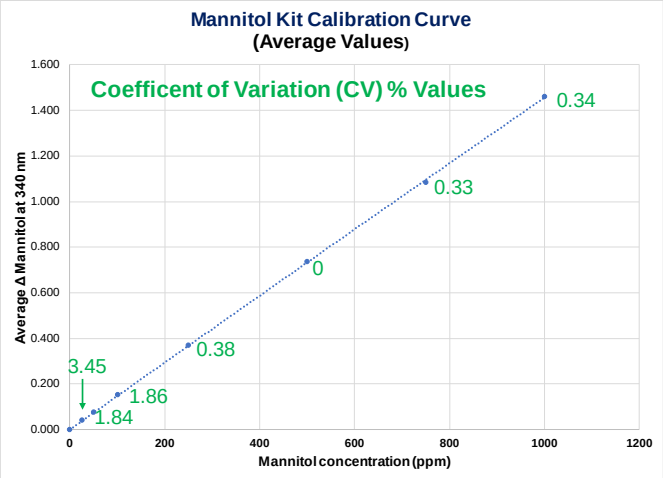


Figure 4. Precision values of the mannitol kit method at various mannitol concentrations.

Table 1. A comparison of the accuracy of the results calculated from the (a) method equation in APPENDIX I or (b) from a standard curve.

Mannitol Concentration (ppm/Brix)

Theoretical	Mannitol Kit Equation	Mannitol Kit Standard Curve
25	27.5	24.7
50	51.7	48.7
100	102.0	98.7
250	247.5	243.4
500	495.1	489.4
750	726.8	719.7
1000	980.4	971.7

RESULTS AND DISCUSSION

Audubon staff demonstrated the use of the kit to factory personnel and discussed how to interpret the results with them. Some factories used it during the late freeze that occurred in the 2018 grinding season. It is already known that values of mannitol greater than 300-500 ppm/Brix in sugarcane juice predict processing problems in the factory (equivalent to 1500 ppm/Brix Haze dextran) (Eggleston, 2009).

Figure 5 shows the mannitol concentrations in core laboratory juices measured using the kit method by personnel at a Louisiana factory on crop days 76 to 96. It was clearly shown that there were numerous days when dextranase should have been added (≥ 500 ppm/Brix mannitol) to control dextran. It must be noted, however, that mannitol measurement cannot be used to assess how well dextranase breaks dextran down at the factory. The results also indicate the *Leuconostoc* deteriorated cane was consistenly being delivered to and processed at this factory, suggesting better cane management strategies were needed such as reducing cut-to-crush times. The factory also daily measures dextran (measured using a Haze dextran method) in composite raw sugars and these results are also illustrated in Figure 5. Results do not correlate as the random samples were taken on different dates and are not expected to directly linked. The relationship of between dextran and mannitol in raw sugars has not yet been established which also makes these results difficult to interpret. It is known, however, that considerably less mannitol (~0.4%) partitions into the raw sugar than dextran (~9-10%) (Promraska et al. 2009; Eggleston et al. 2012).

Some example values of mannitol in various factory products are listed in Table 2. In the four juices measured at two factories, all had values that suggested dextranase should be added to control dextran. One factory who stored billeted cane in the factory yard during foggy conditions had extraordinary high values of mannitol, e.g., 23,125 ppm/Brix. Such levels would be expected to contribute to crystallization problems including conglomerations, and the possibility of mannitol needle crystals (Eggleston et al. 2012). The factory had noticed severe difficulties in vacuum pan crystallizations.

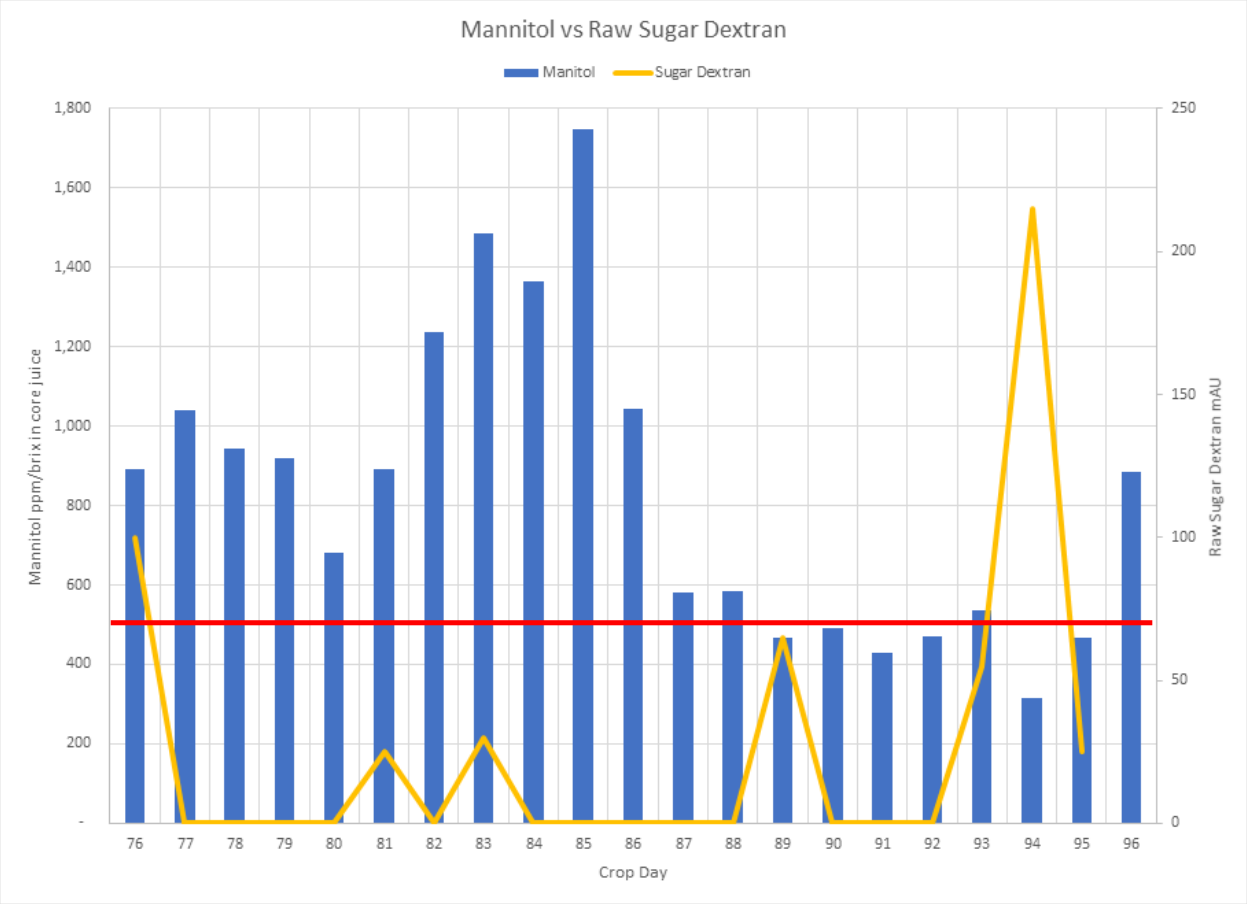


Figure 5. Mannitol concentrations measured in core (pressed) juice at one Louisiana Factory in 2018 using the kit method. The red line represents the maximum limit of mannitol in juice before dextranase should be added to control dextran in the factory.

Table 2. Mannitol measured in various factory product at Louisiana factories during the 2018 grinding season using the kit method.

Factory	Sample Type	Mannitol (ppm/Brix)
B	Crusher Juice	1,336
B	Mixed Juice	467
B	Clarified Juice	451
C	Crusher Juice	23,125
D	Raw Sugar	32
D	Raw Sugar	37
D	Raw Sugar	43
D	Raw Sugar	99
D	Raw Sugar	107
D	Raw Sugar	98

CONCLUSIONS

The Megazyme® enzymatic mannitol kit was further developed for use at sugarcane factories. The kit is readily available, uses equipment already available at the factory, and the method is simple, accurate, and precise. Other urgent questions now need to be answered to expand the use of mannitol measurement at the factory:

1. What is the relationship between dextran and mannitol concentrations in raw sugar, molasses, massecuites, syrup, etc. at the factory?
2. Is there a threshold concentration value for mannitol in various factory products that can cause and predict processing problems?

ACKNOWLEDGEMENTS

- Management and laboratory personnel of St. Mary's factory

REFERENCES

Anon. (2018). Megazyme D-mannitol/D-arabitol assay procedure booklet. www.megazyme.com.

Eggleston G. (2009). Analysis of mannitol, as tracer of bacterial infections in cane and beet sugar factories. Proc. XVI International Andrew Van Hook Symposium, 24-34.

Eggleston G., Harper W. (2006). Determination of sugarcane deterioration at the factory: Development of a rapid, easy and inexpensive enzymatic method to determine mannitol. Food Chemistry, 98(2), 366-372.

Eggleston G., Huet J. M. (2012). The measurement of mannitol in sugar beet factories to monitor deterioration and processing problems. Zuckerindustrie, 137, 33-39.

Eggleston G., Wu Tiu Yen J., Alexander C., Gober J. (2012). Measurement and analysis of the mannitol partition coefficient in sucrose crystallization under simulated industrial conditions. Carbohydrate Research, 355, 69-78.

Promraska A., Flood A.E., Schneider P.A.J. (2009). Measurement and analysis of the dextran partition coefficient in sucrose crystallization. Crystal Growth, 311, 3667-3673.

APPENDIX I
Factory Enzymatic Method to Determine Mannitol in Sugarcane Juices Using the Megazyme® Kit

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Scope:
This enzymatic method specifically measures the amount of mannitol in sugarcane juices. The concentration of mannitol indicates the extent of sugarcane deterioration by *Leuconostoc* bacteria. The method is intended for assessing the extent of deterioration in cane at the factory (Eggleston and Harper, 2006)

Principle:
Mannitol dehydrogenase (MDH) converts mannitol to fructose in the presence of the co-enzyme nictonamide-adenine-dinucleotide (NAD):

$$\text{Mannitol} + \text{NAD} \xrightarrow{\textit{Mannitol dehydrogenase}} \text{Fructose} + \text{NADH} + \text{H}^+$$

The NADH formed is directly related to the amount of mannitol in the juice and is easily measured spectrophotometrically because it absorbs light in the ultraviolet region at 340 nm.

Reaction Conditions:

Temperature : Room temperature (~25 ºC)
Absorption : 340 nm
pH : 9.0
Reaction time : 7 min

Megazyme® Kit:
The kit is suitable for performing 60 assays. It contains all the needed reactants:

Bottle 1: Buffer (10 m, pH 9.0) plus preservative. *Stable for >2 years at refrigeration temperature (4 °C)*

Bottle 2: (x 2) NAD. *Stable for > 2 years stored in the freezer (less than -10 °C)*

Bottle 3: Mannitol dehydrogenase (enzyme) suspension (1.3 mL). *Stable for > 2 years at refrigeration temperature (4 °C)*

Bottle 4: Mannitol standard solution (5 mL; 0.30 mg/mL). *Stable for > 2 years at refrigeration temperature (4 °C)*

Simple Equipment Required:

Spectrophotometer set at 340 nm	Timer
Disposable cuvettes (1 cm)	Pipettes (100 µL and 5 mL)

Juices before clarified juice will need filtering first. This will depend on the instrumentation at the factory. Centrifuged juice is acceptable. Filtering through 0.45 then 0.22 µm PVDF syringe filters also works but is more expensive. Glass filters (25 mm) in a syringe holder with Celite will also work. As a last resort, the juice can be allowed to settle in a refrigerator (4 ^{o}C) and the supernatant used.

Preparation of Reagent Solutions/Suspensions:

1. Use the contents of bottle 1 as supplied.

2. Dissolve the contents of one bottle 2 in 3.3 mL distilled water.

3. Use the contents of bottle 3 as supplied. NOTE: Before opening for the first time, shake the bottle to remove any protein that may have settled on the rubber stopper. Subsequently store the bottle in an upright position.

4. Use the contents of bottle 4 as supplied.

Preparation of Cane Juices and Other Cane Product

The amount of mannitol present in the juice should be in the range of 0.05 to 0.75 g/L (equivalent to 50 to 750 ppm) and the juice should be free of interfering particles. Juices before clarified juice, e.g., pressed juice, crusher juice, and mixed juice, and heated limed juice must be filtered first (see above in green). All samples must first be diluted:

First dilute the juice 1:1 (i.e., 2-fold dilution) with distilled water by pipetting 5 mL juice into a small beaker and adding 5 mL of distilled water. Mix or swirl gently. Filter this diluted juice if the juice contains particles (see above).

Procedure

1. First blank the spectrophotometer against distilled water.

2. To two cuvettes (labelled blank and sample) at room temperature add the following (Table 1):

Table 1.

Pipette into Cuvettes	Blank	Sample
Distilled water	2.10 mL	2.00 mL
Bottle 1 solution (buffer)	0.10 mL	0.10 mL
Bottle 2 solution (NAD)	0.10 mL	0.10 mL
Prepared sample juice	-	0.10 mL

3. Mix the contents of each cuvette by covering the cuvettes with separate pieces of Parafilm® or aluminum foil and gently invert two times. As soon as mixing is finished start the timer and after **2** min read the absorbance (A_1).

4. Add 0.02 mL of bottle 3 contents (Table 2) to each cuvette, cover the cuvettes with separate pieces of Parafilm® or aluminum foil and gently invert two times. Start the timer and read the absorbance after **5** min (A_2).

Table 2.

Pipette into Cuvettes	**Blank**	**Sample**
Bottle 3 solution (MDH)	0.02 mL	0.02 mL

5. Determine the absorbance difference $\Delta A = A_2 - A1$ for both the blank (ΔA_{blank}) and sample (ΔA_{sample}).

Calculations:

1. Determine the absorbance difference $\Delta A = A_2 - A_1$ for both the blank and sample (see above).

2. Subtract the absorbance difference for both blank and sample:

$$\mathbf{\Delta A_{mannitol} = \Delta A_{sample} - \Delta A_{blank}}$$

3. The concentration of mannitol is then calculated using the following equation:

$$\mathbf{C} = \frac{\mathbf{V \times MW}}{\mathbf{\varepsilon \times d \times v}} \times \Delta A \times \text{dilution factor} \quad \text{(g/L)}$$

where:

C = concentration of mannitol in cane juice
V = final volume (2.32 mL)
MW = molecular weight of mannitol (182.17 g/mol)
ε = extinction coefficient of NADH at 340 nm (6300)
d = light path length (1 cm)
v = sample volume (0.1 mL)

Therefore, it follows for mannitol in juice:

$$\mathbf{C} = \frac{\mathbf{2.32 \times 182.17}}{\mathbf{6300 \times 1.0 \times 0.10}} \times \Delta A_{mannitol} \times \text{dilution factor} \quad \text{(g/L)}$$

$$\mathbf{C = 0.6708} \times \Delta A_{mannitol} \times \text{dilution factor} \quad \text{(g/L)}$$

To convert to ppm on a Brix basis:

$$\mathbf{ppm\ mannitol\ on\ a\ Brix\ basis} = \frac{\mathbf{C \times 100{,}000}}{\mathbf{B}}$$

where:

B = original Brix of the juice

Sample Dilution:

If the value of ΔA_{sample} is too low (e.g., <0.100) then dilute less strongly.
If the value of ΔA_{sample} is too high (e.g., >0.75) then further dilution is required.

Interpretation of Mannitol Results at the Louisiana Sugar Factory:
Typically in Louisiana sugarcane factories, values of greater than 300 to 500 ppm/Brix mannitol in juices indicate processing problems in the factory and the need to add dextranase to control dextran levels in raw sugar as well as improve processing.

Vendors are recommendations only

Megazyme® Mannitol Kits can be purchased at www.megazyme.com. It typically takes 3 days to arrive so please plan accordingly.

Product Name: D-Mannitol/L-Arabitol Assay Kit
Product Code: K-MANOL
Price: $270.00 (Does not include shipping)

HOW TO MONITOR MUD CONSISTENCY AT SUGARCANE FACTORIES TO IMPROVE MUD FILTRATION

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INTRODUCTION

One of the sources of expensive sucrose losses in the process of sugar manufacturing is clarification mud. Although the amount of the end by-product filter cake only accounts for around 7.6% of the cane ground in Louisiana, during rainy days this figure can rise up to 10% or more. The purpose of juice clarification is the removal of as many turbid non-sugars as possible, including, organic and inorganic compounds, while preserving sucrose and reducing sugars in the clarified juice. This delicate balance is necessary in order to produce a good quality raw sugar.

The basic goals of the Mud Filtration Station (Rainey et al. 2014) are:

- Recovery of sugar from mud, returning it as filtrate juice to the process, but minimizing clarified (clear) juice recirculation to avoid overloading the capacity for the grinding rate of the factory
- Maximize mud solids retention in the filters (Rotary or Belt Filters), but minimize the pol in filter cake

To make sure these goas are met, the mud underflow fed to the filters must undergo a conditioning treatment to assure:

- Maximum solids retention
- Enough porosity of filter cake to allow water to get through
- Sufficient resistance of filter cake to prevent it breaking apart.

The mud filter station is one of the last remaining sections of a factory that has not yet been automated. Nowadays the control of mud conditioning in Louisiana factories relies mostly on visual and sensory tests, with a few attempts to automate the process: Ziegler Consistency Monitor & Probe Model 970-C by one factory and flowmeters and dP Cells in another factory.

One of the first parameters to determine is the nature of the material in mud underflow. A comprehensive characterization of the physical and chemical properties of the mud underflow, beyond the normal routine analysis performed in the factory processing laboratory, is needed. Thus, the objectives of this study were to (i) find a simple, inexpensive instrument that could be used at the factory to measure mud “consistency” and (ii) correlate the physical and chemical characteristics of mud with pol in filter cake to further automate the handling of mud at the factory and positively affect the bottom line of the factory.

EXPERIMENTAL

Methods and Instruments

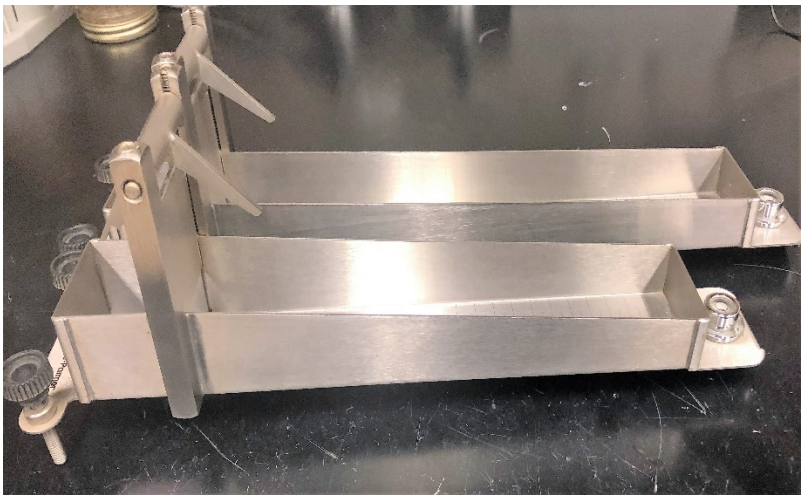
Preliminary tests were undertaken to assess the use of various portable instruments to measure mud consistency in a simple and practical way at the factory site, without the need to bring those samples to a laboratory.

Portable Instruments Initially Tested:

- **A Falling Ball Viscometer (model GV-2300, size # 3 Gilmont Instruments).** This did not work as it was difficult to view the falling ball due to the cloudiness of the sample, plus the instant fall made it impossible to determine any reading.

- **A Falling Needle Viscometer (PDVdi-120 Stony Brook Scientific Portable Field Viscometer, Norristown, PA).** This did not work as the gap between the needle and the outer cylinder was too narrow for the mud particles to pass, causing the needle assembly to stick at the top of the cylinder. The amount of suspended mud particles were also so high that no reading could be obtained.

- **Two Bostwick-type Consistometers (Cole Palmer; 240 mm Trough Length, model ZXCON-CON 2, serial number 0315 and 300 mm Trough Length, model ZXCON-CON4, serial number 0493)**, originally manufactured by Endecotts, were tested. The reason behind testing the Consistometer at two different lengths was to accommodate for reading lighter mud that can run the whole length of the trough. The longer consistomer was found to be of simple and practical use in measuring the consistency of mud at the factory.



Final Bostwick-type Consistometer Analyses

The readings of the Bostwick-type Consistometer in cm/30 sec, were an indication of the flowability of the mud. All consistometers had a spirit level that was centered before use. A mud sample from the underflow of a clarifier was immediately added to fill the 75 mL reservoir of the instrument to ensure the sample was as close to the processing temperature as possible. Once the reservoir was full then the spring-loaded reservoir trap door was opened and the length of flow of the mud in cm was read after 30 secs using the engraved rule track of the device. Reproduciblity of the consistometer was ± 0.5 cm with the same operator.

Other Analyses of Mud

The temperature of the underflow mud on site was taken immediately using a Digital Thermometer (Hanna Checktemp). Per cent mud volume in the underflow mud was determined using a benchtop Centrifuge Thermo Centra CL2 and a Mini Centrifuge Myfuge C1012. Density of the mud was measured at room temperature (22-25 °C) by accurately weighing 0.5 L of underflow mud. Units were converted lb/gal, to be consistent with factory units. The moisture of mud was measured by using a VWR Oven and a NAPCO model 5861 Vacuum Oven set at 100 °C. The sample was heated in the oven for 24 h and the moisture was calculated as the weight difference. The pH of the mud was measured at room temperature using an Oakton PC700 pH Meter. Brix of the filter cake was measured using a digital Bellingham & Stanley, Code: 22-340, Serial Number BU15151 Refractometer. The pol of filtercake (26 g/100 mL) was measured using a Rudolph Research Analytical, Autopol 880, Serial: 3164 Polarimeter.

Factory Trials:

This study was undertaken at four Louisiana sugarcane factories (named A, B, C & D) during the 2018 grinding season:

1. **Mill A:** Grinding rate: **18,000 TCD**
   3 ea. Tray Clarifiers (Graver & Dorr) – **Total 261,362 gal**
   5 ea. Rotary Vacuum Filters (RVFs) – **Total 2,778 ft²**

2. Mill B: Grinding rate: **13,000 TCD**

3 ea. Tray Clarifiers (Dorr) – **Total 195,000 gal**

3 ea. Rotary Vacuum Filters (RVFs) – **Total 1,369 ft²**

3. Mill C: Grinding rate: **13,000 TCD**

2 ea. Tray Clarifiers (Dorr) – **Total 195,000 gal**

6 ea. Rotary Vacuum Filters (RVFs) – **Total 3,179 ft²**

4. Mill D: Grinding rate: **13,000 TCD**

3 ea. SRT Clarifiers (SRI & LLT) – **Total 153,000 gal**

4 ea. Vacuum Belt Filters (VBFs) – **Total 3,060 ft²**

Factory Sampling

Preliminary factory trials were first undertaken to ensure that accurate sampling and measurements were obtained. As the consistency of mud is dependent on temperature, and the temperature of the sample mud decreased rapidly on sampling, the mud (~0.5 gal) was collected in an insulated sample bottle and the mud consistency measured immediately on the factory floor, using both the short and long length Bostwick-type Consistometers. This way, if the short one could not produce any reading because the mud was too light, then the same sample could produce a number in the longer one. The rest of the sample was split into two containers, with one stored on ice and the other frozen in dry ice. Both samples were then transported to the Audubon Sugar Institute to be analyzed for density, pH, Brix, mud volume, and mud moisture. Analyses were undertaken the next day on the sample stored on ice (refrigerated temperature 4 °C), when possible. If that was not possible, the samples were placed in a freezer (-40 °C), together with the samples stored in dry ice at the mill until later analyses of pol, sediment, and particle size distribution.

Visits to the factories were scheduled to accommodate two collection visits per month during the 2018 grinding season. Four samples per visit were collected, with samples collected every 1 h. These samples were collected from all operating mud pumps in each clarifier to make a composite sample to analyze for the whole clarifier. Two composite filter cake samples of all operating filters (RVFs and BVFs) were also collected, one at the beginning of the day's study and one at the end. One sample of filtrate juice was collected during each visit for the same purpose.

Overall, three out of the four factories received six visits during the grinding season. Only one received five visits, since it closed early January 2019 before we were able to visit the factory

RESULTS AND DISCUSSION

The physical and chemical characteristics of the collected muds from the four factories in this study are listed in Tables 1 to 4.

Table 1. Factory A mud results.

MILL A							
MUD UNDERFLOW CHARACTERISTICS							
VISITS DATE	10/19/18	11/02/18	11/20/18	11/30/18	12/14/18	01/07/19	**AVG**
SAMPLES	18	12	12	12	12	12	
TEMPERATURE MUD (ºF)	190.0	189.1	194.0	196.0	192.6	189.3	**191.8**
pH	6.74	7.36	7.39	7.29	7.32	7.37	**7.24**
TEMPERATURE pH (ºF)	146.8	77.0	73.0	73.0	73.0	73.2	**86.0**
DENSITY (Lb/gln)	9.041	9.124	9.092	9.226	9.186	9.078	**9.124**
CONSISTOMETER (short)(cm/30 sec)	18.81	16.97	16.04	17.08	16.63	22.58	**18.02**
CONSISTOMETER (long)(cm/30 sec)	na	14.37	16.79	16.42	14.46	21.83	**16.77**
POL IN FILTER CAKE (MILL)	2.72	2.21	2.08	2.80	2.04	2.19	**2.34**
POL IN FILTER CAKE (ASI)	2.78	2.40	1.93	2.03	2.15	2.52	**2.30**
% MUD VOLUME	na	38.25%	33.83%	37.33%	38.66%	35.95%	**36.81%**
MUD % MOISTURE	76.94%	77.57%	78.78%	77.30%	78.10%	79.64%	**78.05%**
ZIEGLER READINGS	39.40	42.99	36.92	37.20	36.05	27.75	**36.72**

Table 2. Factory B mud results.

MILL B							
MUD UNDERFLOW CHARACTERISTICS							
VISITS DATE	10/17/18	10/31/18	11/13/18	11/29/18	12/12/18	01/04/19	
SAMPLES	12	12	12	12	12	6	**AVG**
TEMPERATURE MUD (ºF)	193.6	197.6	194.2	196.7	196.7	195.8	**195.8**
pH	6.64	7.35	7.32	7.25	7.18	7.21	**7.16**
TEMPERATURE pH (ºF)	149.0	71.6	73.4	73.4	73.0	73.2	**85.6**
DENSITY (Lb/gln)	9.069	9.191	9.338	9.169	9.236	9.283	**9.214**
CONSISTOMETER (short)(cm/30 sec)	18.17	21.85	20.69	24.00	21.85	21.75	**21.39**
CONSISTOMETER (long)(cm/30 sec)	na	na	16.88	23.71	19.96	18.83	**19.85**
POL IN FILTER CAKE (MILL)	2.00	4.01	4.16	3.70	2.80	3.18	**3.31**
POL IN FILTER CAKE (ASI)	2.38	5.25	4.40	3.87	3.77	4.15	**3.97**
% MUD VOLUME	na	32.33%	37.25%	35.17%	41.14%	39.83%	**37.14%**
MUD % MOISTURE	77.14%	78.00%	75.19%	78.27%	77.37%	77.10%	**77.18%**

Table 3. Factory C mud results.

MILL C						
MUD UNDERFLOW CHARACTERISTICS						
VISITS DATE	10/12/18	10/26/18	11/09/18	11/26/18	12/07/18	
SAMPLES	8	8	8	8	6	**AVG**
TEMPERATURE MUD (ºF)	193.8	198.1	201.2	197.8	201.6	**198.5**
pH	6.87	7.28	6.89	7.34	7.27	**7.13**
TEMPERATURE pH (ºF)	142.7	71.6	71.6	73.4	72.3	**86.3**
DENSITY (Lb/gln)	9.238	9.144	9.276	8.996	9.152	**9.164**
CONSISTOMETER (short)	4.95	8.69	3.44	13.00	13.50	**8.72**
CONSISTOMETER (long)	na	na	2.81	14.75	13.25	**10.27**
% MUD IN FILTRATE JUICE (MILL)	7.30%	33.00%	4.70%	5.30%	6.70%	**11.40%**
POL IN FILTER CAKE (MILL)	3.04	1.70	0.80	0.90	0.53	**1.39**
POL IN FILTER CAKE (ASI)	3.82	1.61	2.10	2.06	2.77	**2.47**
% MUD VOLUME	na	44.00%	51.13%	34.63%	38.65%	**42.10%**
MUD % MOISTURE	74.42%	77.14%	75.35%	80.57%	77.59%	**77.02%**

Table 4. Factory D mud results.

MILL D							
MUD UNDERFLOW CHARACTERISTICS							
VISITS DATE	10/15/18	10/29/18	11/12/18	11/28/18	12/10/18	01/09/19	
SAMPLES	2	9	12	12	12	12	**AVG**
TEMPERATURE MUD (ºF)	199.0	201.7	202.6	201.9	201.9	202.6	**201.6**
pH	6.22	7.29	7.39	7.41	7.46	7.26	**7.17**
TEMPERATURE pH (ºF)	151.7	71.6	73.4	73.0	73.0	73.2	**86.0**
DENSITY (Lb/gln)	8.871	9.087	9.261	9.188	9.180	9.362	**9.158**
CONSISTOMETER (short)	5.25	8.37	10.39	8.67	10.58	8.00	**8.54**
CONSISTOMETER (long)	na	na	9.42	8.50	10.21	7.58	**8.93**
POL IN FILTER CAKE (MILL)	0.64	0.60	1.04	0.52	0.68	1.69	**0.86**
POL IN FILTER CAKE (ASI)	0.56	0.49	0.82	0.49	0.56	1.58	**0.75**
% MUD VOLUME	na	47.50%	43.33%	44.55%	43.08%	48.91%	**45.47%**
MUD % MOISTURE	79.76%	77.84%	75.68%	75.99%	77.54%	77.54%	**77.39%**

The average mud results of all four factories are also listed in Table 5 for direct comparison.

Table 5. Average mud results for all four factories.

AVERAGE TEST RESULTS ON MUD UNDER FLOW				
PARTICIPATING FACTORIES	**MILL A**	**MILL B**	**MILL C**	**MILL D**
TEMPERATURE (°F)	191.8	195.8	198.5	201.6
pH	7.24	7.16	7.13	7.17
pH TEMPERATURE (°F)	86.0	85.6	86.3	86.0
DENSITY (Lb/gal)	9.124	9.214	9.164	9.158
CONSISTENCY (cm/30 sec) Flowabiliy	17.40	20.62	9.50	8.74
POL IN FILTER CAKE	2.34	3.31	1.39	0.86
% MUD VOLUME	36.81%	37.14%	42.10%	45.47%
MUD % MOISTURE	77.78%	77.19%	76.87%	77.36%
FILTER FEED TEMPERATURE (°F)	167.00	183.20		187.90
TEMPERATURE DROP (°F)	24.80	12.60		13.70
CONSISTENCY (cm/30 sec) Flowabiliy	30.00	15.80		0.50
CONSISTENCY CHANGE	+	–		–
CONSISTENCY NUMERIC CHANGE	12.60	-4.82		-8.24

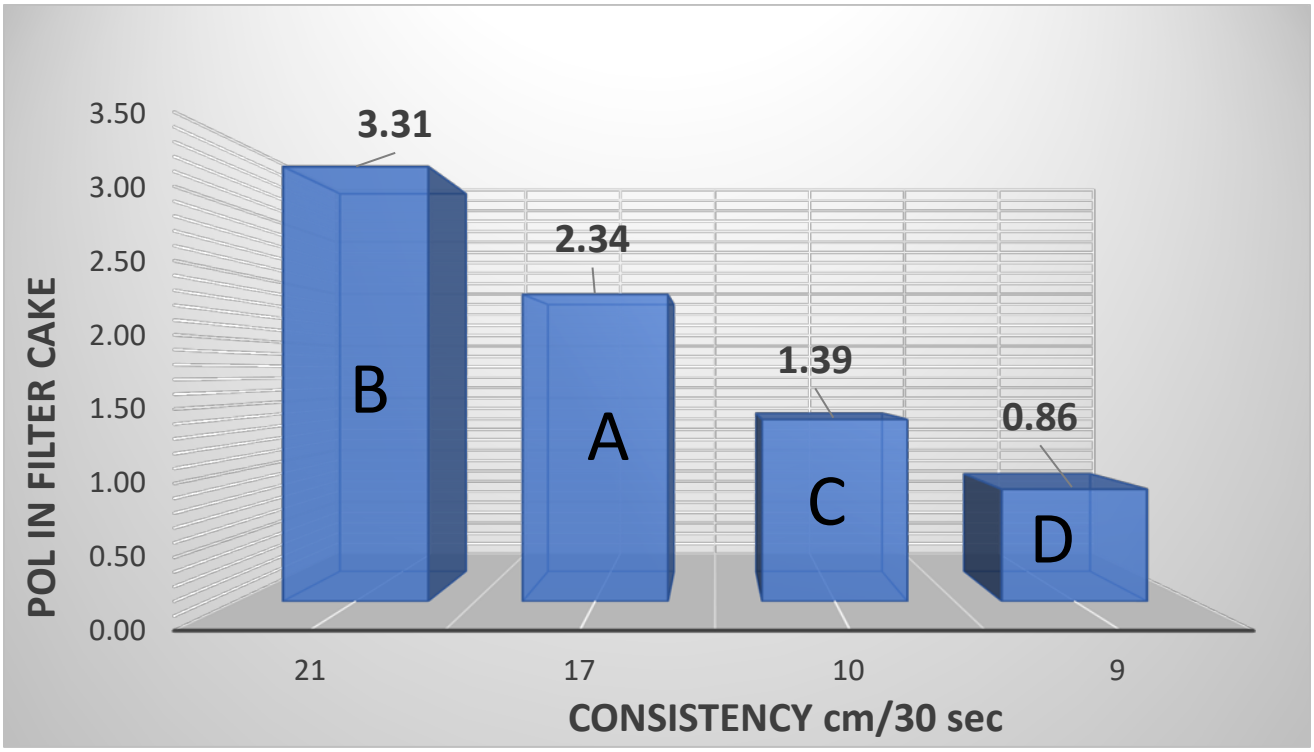


Figure 1. Bostwick-type consistency results versus pol in filtercake results.

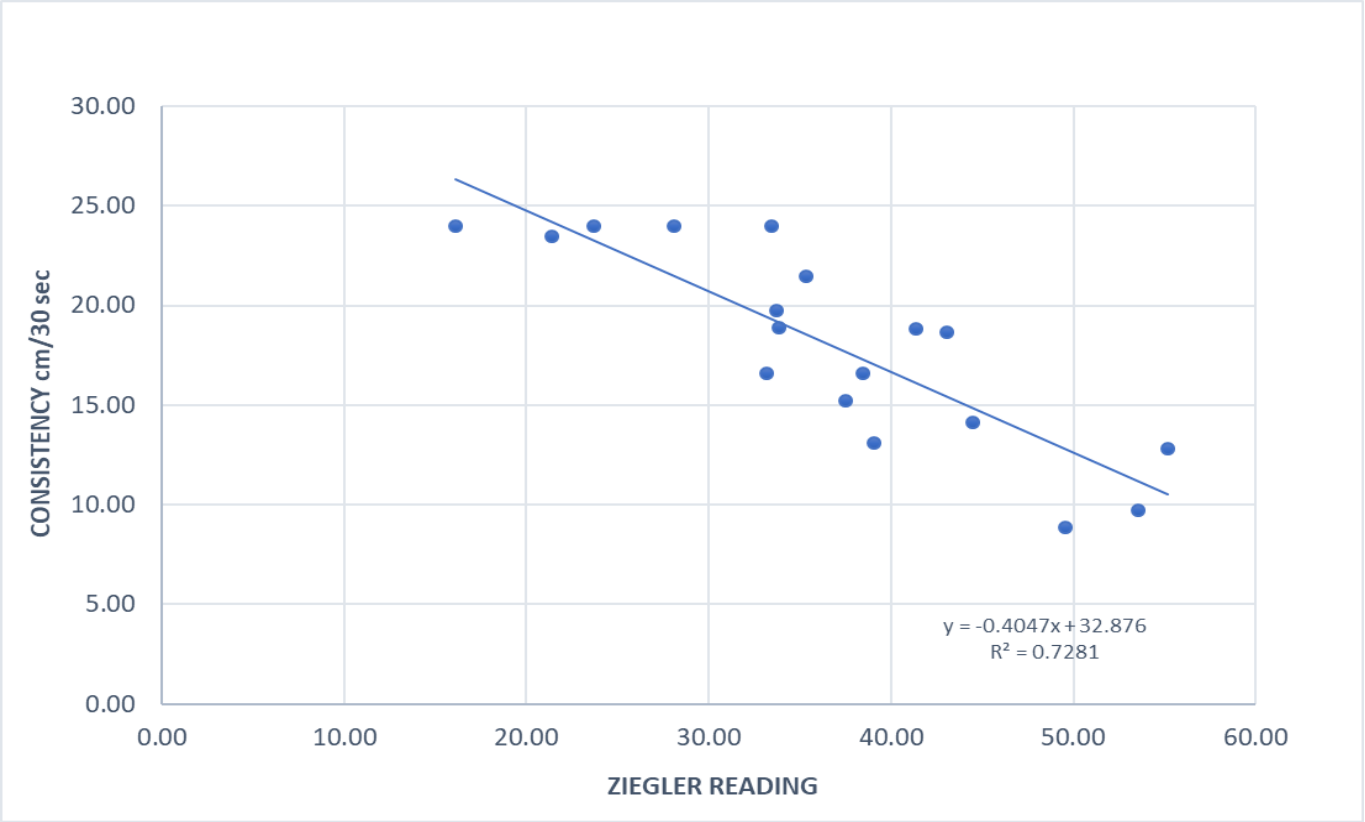


Figure 2. The relationship between Bostwick-type consistency results and Ziegler (viscosity) readings at factory A only.

CONCLUSIONS

- The mud underflow directly from the clarifiers showed similar characteristics of pH, density, and mud % moisture, but showed different behavior for Consistency (flowability), % mud volume, and pol in filter cake.
- Factories A & B had the highest readings in the Consistometer which equates to lighter muds. This was confirmed with the results on % mud volume lowest values (Tables 1 and 2)
- Factories C & D had the lowest readings in the Consistometer which equates to heavier muds. Again, this was confirmed with the results on % mud volume highest values (Tables 3 & 4)
- Factories A & B consistently had the lowest pol values in filter cake (Fig. 1)
- Factories C & D consistently had the highest pol values in filter cake (Fig. 1)
- Factory A (Tray Clarifiers & RVFs) already have an automated mud consistency control in place based on the Ziegler Sugar Consistency Monitor Model 970-C, Consistency Sensor with propeller-type rotor and torque of the Clarifier Drive. A high correlation was found between the Bostwick-type Consistometer readings and the Ziegler readings at this factory. The higher the Ziegler readings, the lower the Consistency readings and the heavier the mud (Fig. 2)

- Factory D (SRT Clarifiers & BVFs) also had a mud consistency control in place, but this was based on using Gravimetric and Flow measurements through an ordinary dP Cell, flowmeters for the incoming juice, and outgoing underflow mud and torque of the Clarifier Drive.
- Overall, the Bostick-type Consistometer proved to be a very simple, reliable, and inexpensive instrument to measure mud flowability at the factory site. It represents a perfect tool for operators to help them control the consistency of the mud and at the same time produce a reference number that can be useful for Instrument personnel to initiate or fine tune their mud consistency control

ACKNOWLEDGEMENTS

The authors thank the management and staff of the four factories for their help in this study.

REFERENCES

Rainey T.J., Thaval O.P., Rackemann, D.W. (2014). Developments in mud filtration technology in the sugarcane industry. In Sugarcane Production, Consumption and Agricultural Management Systems. Eleanor Webb, Ed., New York, 263-292

THE 2018 MOLASSES SURVEY RESULTS

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INTRODUCTION

Since 2001, the Audubon Sugar Institute has analyzed molasses samples provided weekly by each of the eleven Louisiana raw sugar factories. The results of our analyses are used to calculate a "target purity" (TP) and a true purity for the molasses. The TP is the theoretical concentration of sucrose (sugar) where, regardless of effort, no further sugar can be crystallized. The model that is used to calculate the TP originates from South Africa (Rein, 2007), and has been confirmed as representative of the Louisiana industry (Saska et al. 2010).

The true purity is determined by high performance liquid chromatography (HPLC) and is free of the interferences (reducing sugars) that can offset the accuracy of polarimetric determinations (particularly in molasses where purities are very low). The formula for TP is given below, where *RS* is the total reducing sugar (glucose + fructose) determined by HPLC (ICUMSA, 2002) and *Ash* is the approximate sulfated ash via conductivity (Saska et al. 1999).

$$TP = 33.9 - 13.4 \cdot Log_{10} \frac{RS}{Ash}$$

The TP is subtracted from the true purity to give a target purity difference or TPD. The TPD is used by the factories to determine how well they are recovering sugar from their massecuites (which is reflected by residual sugar in the molasses). "True purity" is the sum of the non-crystallizable sugar and that which was crystallized, but was lost across the centrifugals. For this reason, the nutsch should be assayed in order to determine how much sugar is lost across the centrifugals. Generally, a lower TPD indicates greater efficiency as it relates to recovery of sugar.

EXPERIMENTAL

Composite samples of final molasses (seven days) were sent to the Audubon Sugar Institute weekly from each of the eleven mills in Louisiana. The 2018 survey season stretched from 09-30-18 until 01-13-19. A total of 193 molasses and syrup samples and 10 juice samples were analyzed in duplicate for the 2018 season.

Analyses included:

1. Refractometer Brix (ICUMSA GS4-13)
2. Sucrose, glucose and fructose by HPLC (ICUMSA GS7/4/8-23)
3. Sucrose via polarimetry*
4. Conductivity ash (ICUMSA GS1/3/4/7/8-13)

*Because we measure sugar using HPLC, we perform a direct polarization of molasses, syrup, and juice samples which are clarified using Octapol^{TM} (Baddley Chemical) so that we can obtain a pol/sucrose ratio.

A double-blind quality control (QC) was performed each week. Briefly, a large sample of molasses is collected during the first week of the season. This sample is sub sampled into enough small containers to last the season (approximately 28-30 samples). Each week, two of these subsamples are pulled and included randomly into the weekly sample set. Each sample in the weekly set is mixed thoroughly and subsampled into containers identical to those used for the QC. A number is applied to each container, and the identity of each sample is kept in confidence until the analyses are complete.

RESULTS AND DISCUSSION

The 2018 season was longer than usual and operated for 16 weeks. The 2018 season maximum TPD weekly average was 12.5 and the minimum was 3.3. Throughout the season, the TPDs demonstrated the usual trend of decreasing TPD. The Industry average TPD for 2018 was 5.8. (Figure 1).

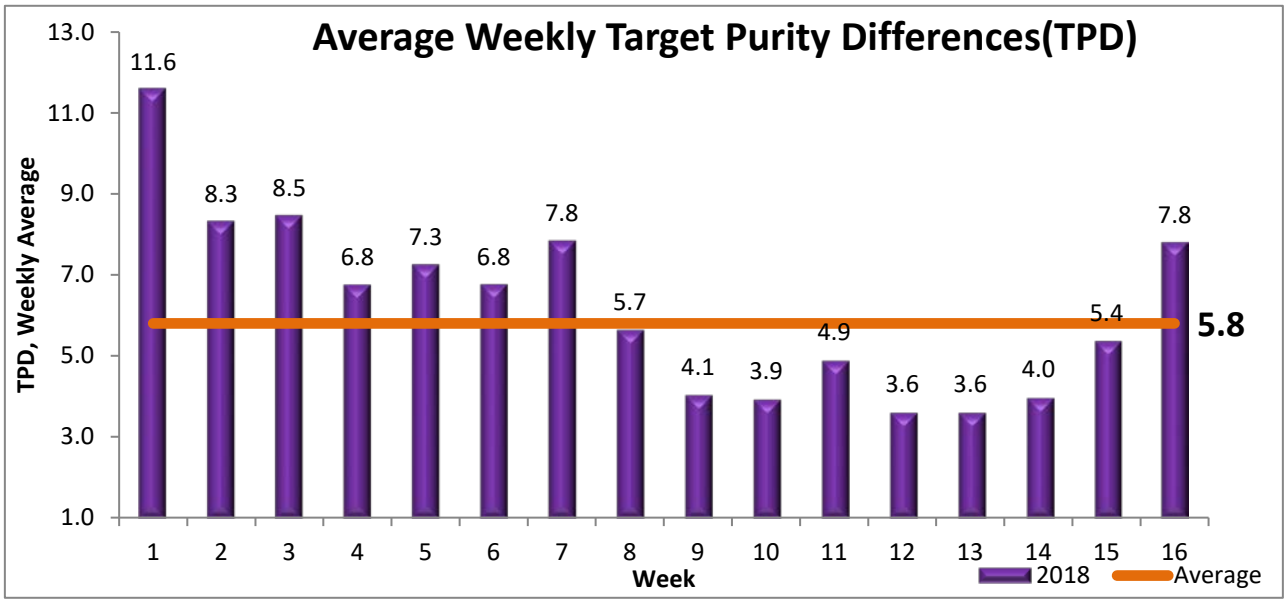

Figure 1. 2018 Average Weekly Target Purity Difference

The conductivity ash component for the 2018 season had a minimum value of 13.3%. Towards the middle of season, the ash increased to the maximum value of 18.1% and slightly decreased thereafter. The conductivity ash weekly average was 16.3% (Figure 2).

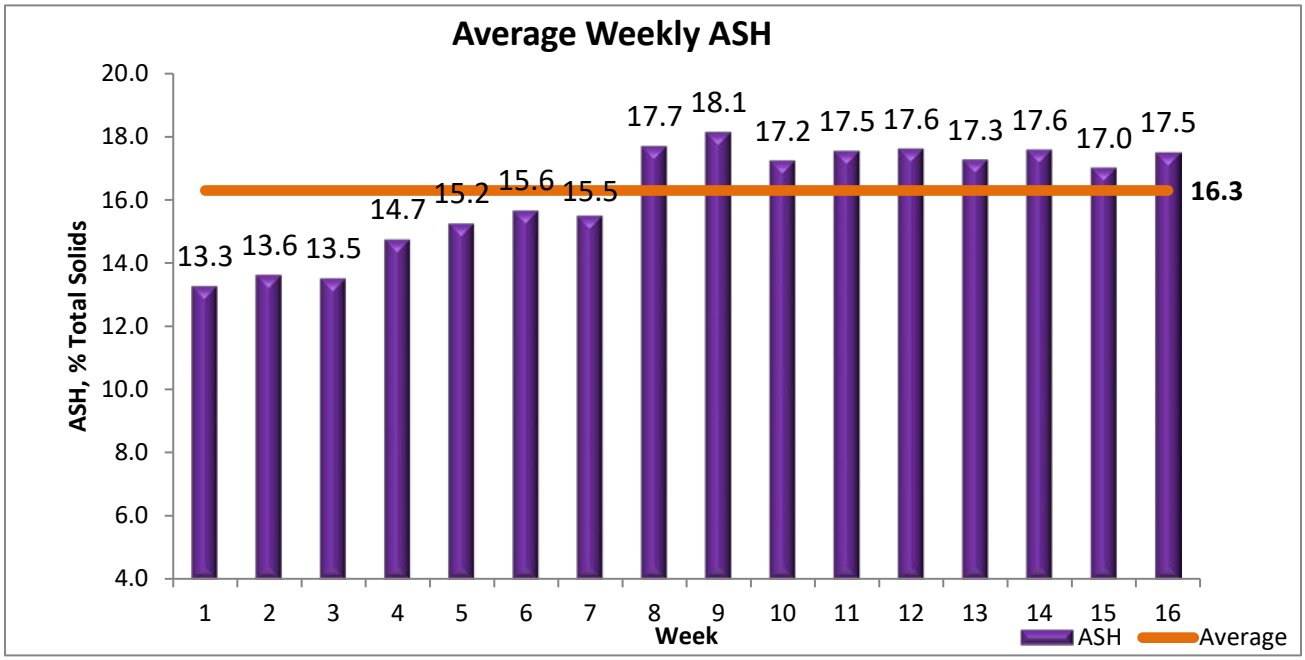

Figure 2. 2018 Average Weekly Conductivity Ash.

In the 2018 season, the reducing sugars increased up to week 9. The maximum was 14.4% and the minimum occurred near the beginning of the season with 9.9. The reducing sugars weekly average was 12.8% (Figure 3).

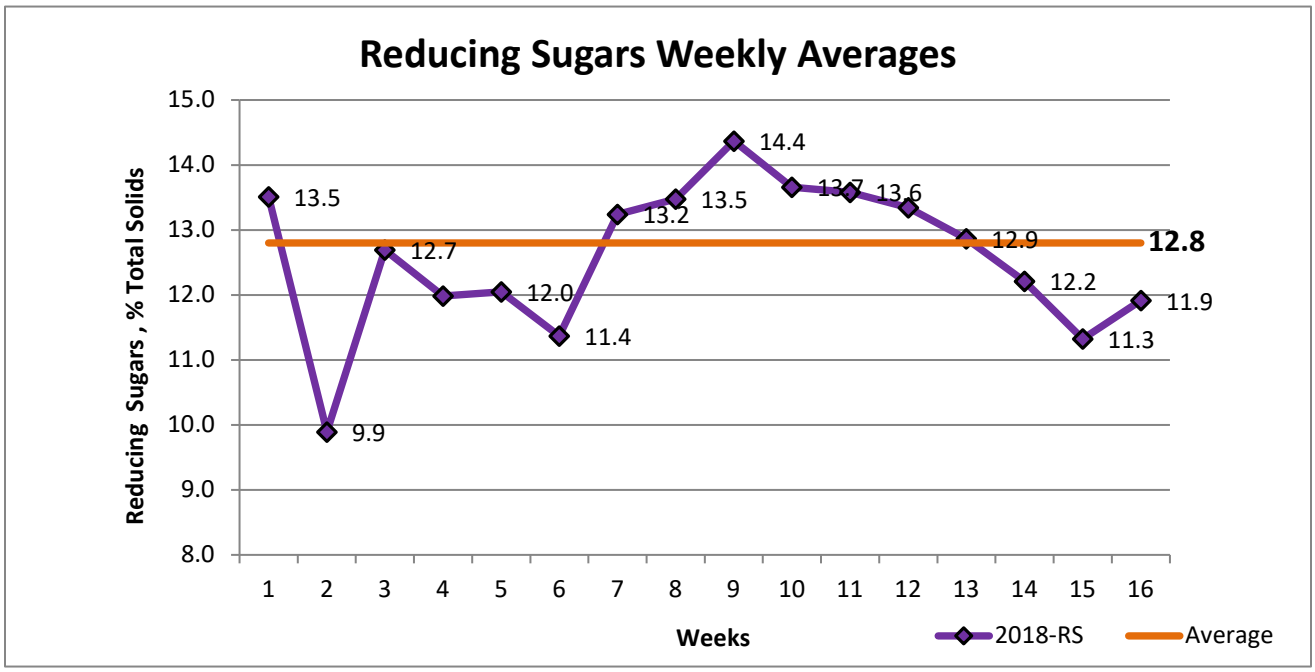

Figure 3. 2018 Reducing Sugars Weekly Averages

Comparing the results from the 2018 season to the results from the previous seasons showed the yearly average TPD of 7.9. This is demonstrated in Figure 4**.** The 2018 season maximum TPD was 12.5. The minimum TPD for 2018 was 3.3 (Table 1).

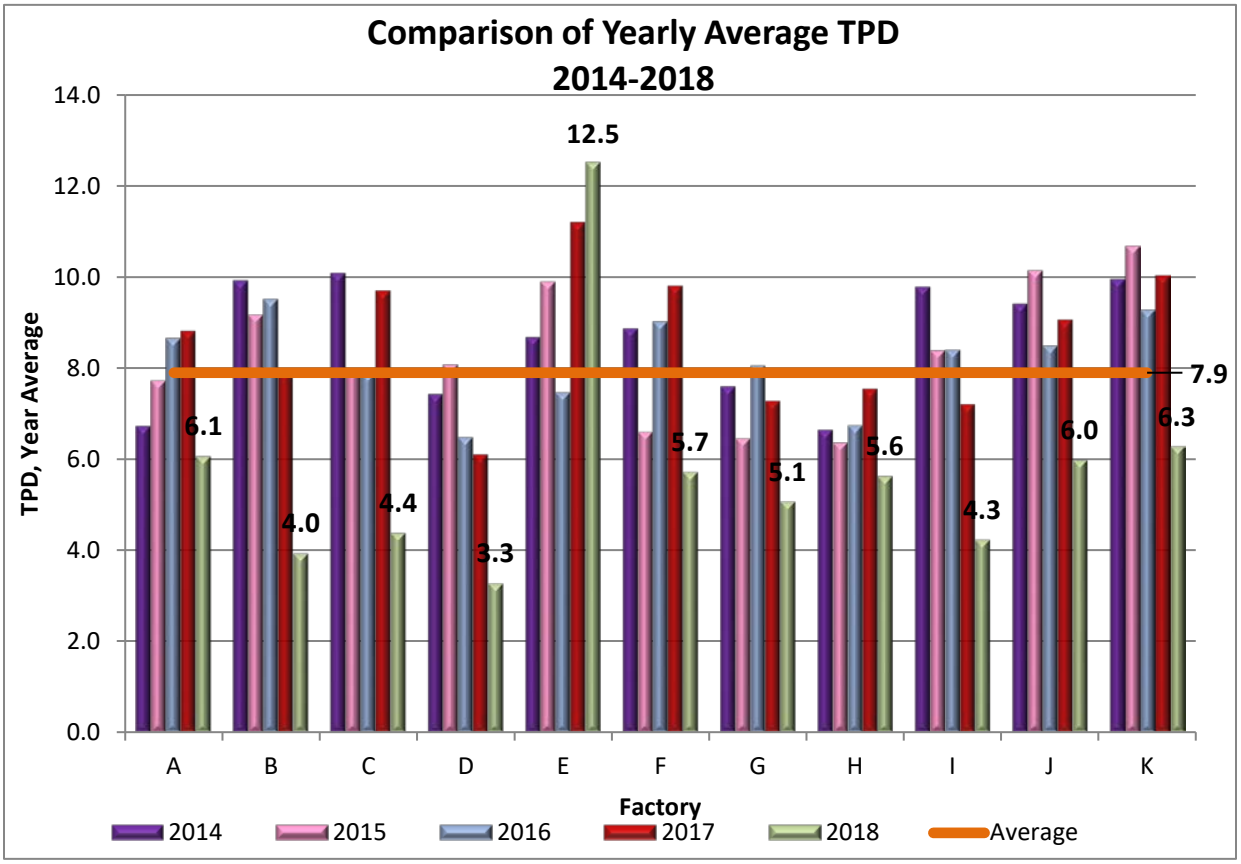

Figure 4. Comparison of Yearly Averages TPD 2014-2018.

Table 1. Summary of Yearly TPD 2014-2018.

TPD Data Summary for 2014-2018			
Year	**TPD Minimum**	**TPD Maximum**	**TPD Average**
2014	6.6	10.1	8.6
2015	6.4	10.7	8.3
2016	6.5	9.5	8.2
2017	6.1	11.2	8.3
2018	3.3	12.5	7.9

The sugar cane juice from the sugar mill has been analyzed for the past 9 years. Results in Table 2 shows the summary from the analyses. Over the last nine seasons, the average Brix was 14.4%, a true purity average was 88.6%, and reducing sugars 5.2%.

Table 2. Summary of Juice Survey 2010-2018**.**

Juice Survey Summary for 2010-2017			
Year	**Ref. Brix (%Juice)**	**True Purity (% Juice)**	**Reducing Sugars (%Juice)**
2010	14.5	89.3	3.8
2011	15.1	87.2	5.0
2012	14.6	88.1	3.6
2013	14.1	91.1	10.8
2014	14.8	88.7	4.1
2015	13.5	88.9	3.2
2016	15.1	88.9	5.2
2017	14.5	88.8	6.1
2018	13.5	86.4	4.8
Average	**14.5**	**88.9**	**5.2**

CONCLUSIONS

The seasonal average TPD was 5.8 for the 2018 season, which was a decrease from the 2017 season. The ash increased for the 2018 season to 16.3%. The reducing sugars remained somewhat the same for the 2018 season at 12.8% from 12.9% from the previous season.

For the 2018 season, some of the TPDs were the lowest observed when compared to the previous seasons.

During the middle 2018 season, cold temperatures (early freeze) occurred in some areas. This had an unusual effect on the purity of the final molasses. The final molasses also had high levels of reducing sugars and low purities. The differences can be attributed to a wide range of factors which included weather conditions and harvest conditions, cane maturity and increased awareness at the cane delivery/mill level.

For the 2018 season, the juice had an average Brix of 13.5%, a true purity of 86.4%, and reducing sugars of 4.8%.

REFERENCES

Baddley Chemical. (2001). Octapol and Octapol Plus. *http://www.baddley.com/octapol.htm*
ICUMSA. (1994). Method GS4-13: The Determination of Refractometric Dry Substance (RDS %) of Molasses – Accepted. ICUMSA Methods Book. Verlag Dr. Albert Bartens KG-Berlin. Supplement 2003.

ICUMSA. (2002). Method GS7/4/8-23: The Determination of Sucrose, Glucose and Fructose by HPLC - in Cane Molasses - Official -and Sucrose in Beet Molasses – Official. ICUMSA Methods Book. Verlag Dr. Albert Bartens KG-Berlin. Supplement 2003.

ICUMSA. (1994). Method GS1/3/4/7/8-13: The Determination of Conductivity Ash in Ra Sugar, Brown Sugar, Juice, Syrup and Molasses - Official. ICUMSA Methods Book. Verlag Dr. Albert Bartens KG-Berlin. Supplement 2003.

Rein P. (2007). Cane Sugar Engineering. Verlag Dr. Albert Bartens KG-Berlin, p 459.

Saska M., Goudeau S., and Andrews, L. (1999). Molasses Exhaustion and Target Purity Formulas. Sugar J. 62, 20-24.

Saska M., Goudeau S., and Beyene F. (2010). Exhaustibility of Louisiana Final Molasses and the Target Purity Formula: The 2009-2010 Season Results. Ann. Meet. Am. Soc. Sug. Cane Technol., La. Div. presentation, Feb. 1-3.

CRYSTAL SIZE ANALYSIS FOR LOUISIANA SUGAR FACTORIES 2018/19 GRINDING SEASON

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INTRODUCTION

Crystal size analysis has been provided as a service by the Audubon Sugar Institute to all Louisiana sugar factories since 2007. In May 2018, a Malvern Mastersizer 3000 laser diffraction particle size analyzer was purchased with a Board of Regents Enhancement Award. Standard Operating Procedures for the Malvern Mastersizer 3000 were developed, and seed slurry and C-sugar samples, collected and analyzed during the 2017/18 grinding season, were analyzed multiple times with the new acquired equipment to check for accuracy and equipment sensitivity. This new particle size analyzer was used during the 2018/19 grinding season to accurately measure the particle size and monitor the size distribution of both seed slurry and C-sugar samples of all eleven factories.

RESULTS AND DISCUSSION

Seed slurry particle size distribution results for each factory are summarized in Table 1 and Figure 1.

Table 1. 10-percentile, 50-percentile (median), 90-percentile, Mean, and CV-values of crystal size distributions for seed slurries collected during the 2018/19 grinding season (average values ± standard deviation).

Factory	Number of Samples	D 10% [µm]	D 50% [µm]	D 90% [µm]	Mean [µm]	CV
A	6	2.70 ± 0.44	6.72 ± 0.97	16.3 ± 1.84	8.69 ± 1.44	0.88 ± 0.40
B	10	3.35 ± 0.18	9.02 ± 0.90	21.1 ± 2.77	10.9 ± 1.29	0.69 ± 0.05
C	12	2.81 ± 0.20	7.46 ± 0.83	19.6 ± 3.23	9.60 ± 1.20	0.80 ± 0.09
D	9	3.07 ± 0.08	8.40 ± 0.45	19.9 ± 1.51	10.2 ± 0.62	0.71 ± 0.07
E	12	4.33 ± 0.31	18.5 ± 0.71	54.9 ± 2.86	24.9 ± 1.18	0.86 ± 0.04
F	12	3.19 ± 0.20	8.55 ± 0.46	19.2 ± 0.90	10.1 ± 0.48	0.67 ± 0.05
G	11	5.28 ± 0.53	19.6 ± 1.54	54.4 ± 3.33	25.5 ± 1.75	0.81 ± 0.03
H	13	4.92 ± 0.90	19.5 ± 1.19	55.4 ± 4.35	25.5 ± 1.50	0.81 ± 0.05
I	12	3.17 ± 0.15	7.93 ± 0.42	17.4 ± 0.93	9.30 ± 0.47	0.64 ± 0.05
J	10	2.95 ± 0.14	7.97 ± 0.31	18.4 ± 0.52	9.50 ± 0.30	0.67 ± 0.02
K	7	4.94 ± 0.44	17.6 ± 0.62	46.6 ± 2.70	22.2 ± 1.10	0.77 ± 0.02
Overall	114	3.70 ± 0.97	11.9 ± 5.50	31.2 ± 17.3	15.1 ± 7.52	0.76 ± 0.08

The average mean crystal size for seed slurry was 15 µm with a 10-percentile (D10%) of 4 µm, a 50-pencentile (D50%) of 12 µm, and a 90-percentile (D90%) of 31 µm. Particle size distribution means ranged from 9 to 26 µm, with D10%, D50%, and D90% observed values ranging from 3 to 5 µm, 7 to 20 µm, and 16 to 55 µm, respectively.

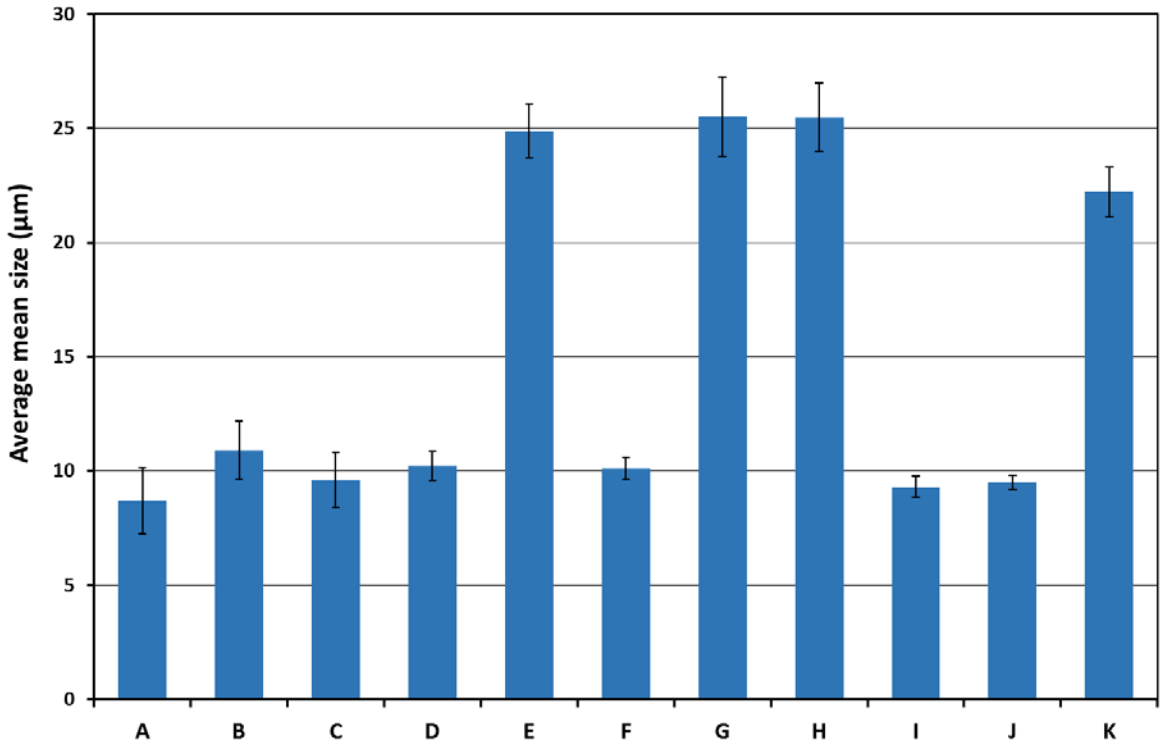

Figure 1. Average mean crystal size of seed slurry samples for factories A-K collected during the 2018/19 grinding season.

C-sugar particle size distribution results for each factory are summarized in Table 2 and Figure 2. The average mean crystal size for C-sugar was 232 µm with a D10% of 81 µm, a D50% of 222 µm, and a D90% of 396 µm. Crystal size means of collected C-sugar samples ranged from 172 to 291 µm, with D10%, D50%, and D90% observed values ranging from 35 to 113 µm, 155 to 281 µm, and 333 to 484 µm, respectively.

Table 2. 10-percentile, 50-percentile (median), 90-percentile, Mean, and CV-values of crystal size distributions for C-sugars collected during the 2018/19 grinding season (average values ± standard deviation).

Factory	Number of Samples	D 10% [µm]	D 50% [µm]	D 90% [µm]	Mean [µm]	CV
A	15	75.69 ± 18.2	206.0 ± 24.8	360.0 ± 42.7	213.6 ± 25.6	0.50 ± 0.04
B	22	91.04 ± 8.48	213.4 ± 29.6	367.0 ± 53.6	222.0 ± 30.0	0.47 ± 0.02
C	20	35.32 ± 15.99	155.5 ± 16.56	333.0 ± 20.20	172.0 ± 13.55	0.68 ± 0.13
D	24	77.05 ± 16.06	220.5 ± 26.22	388.3 ± 46.61	228.2 ± 27.04	0.51 ± 0.04
E	24	82.51 ± 22.64	235.7 ± 21.59	401.8 ± 34.70	241.3 ± 22.35	0.49 ± 0.03
F	29	88.27 ± 25.45	230.2 ± 21.04	389.0 ± 33.18	236.3 ± 22.36	0.48 ± 0.04
G	18	79.88 ± 14.04	195.2 ± 25.86	345.3 ± 43.27	205.0 ± 25.91	0.49 ± 0.03
H	26	113.0 ± 51.18	266.2 ± 93.19	482.1 ± 78.84	282.9 ± 73.79	0.58 ± 0.34
I	18	63.93 ± 15.84	195.7 ± 24.90	361.8 ± 37.39	206.3 ± 24.24	0.54 ± 0.04
J	12	110.7 ± 40.26	281.7 ± 24.28	484.3 ± 36.02	291.5 ± 26.8	0.46 ± 0.05
K	17	71.71 ± 28.66	241.9 ± 54.46	444.5 ± 79.80	252.5 ± 53.34	0.56 ± 0.05
Overall	225	80.83 ± 21.38	222.0 ± 35.12	396.1 ± 52.49	231.97 ± 34.78	0.52 ± 0.06

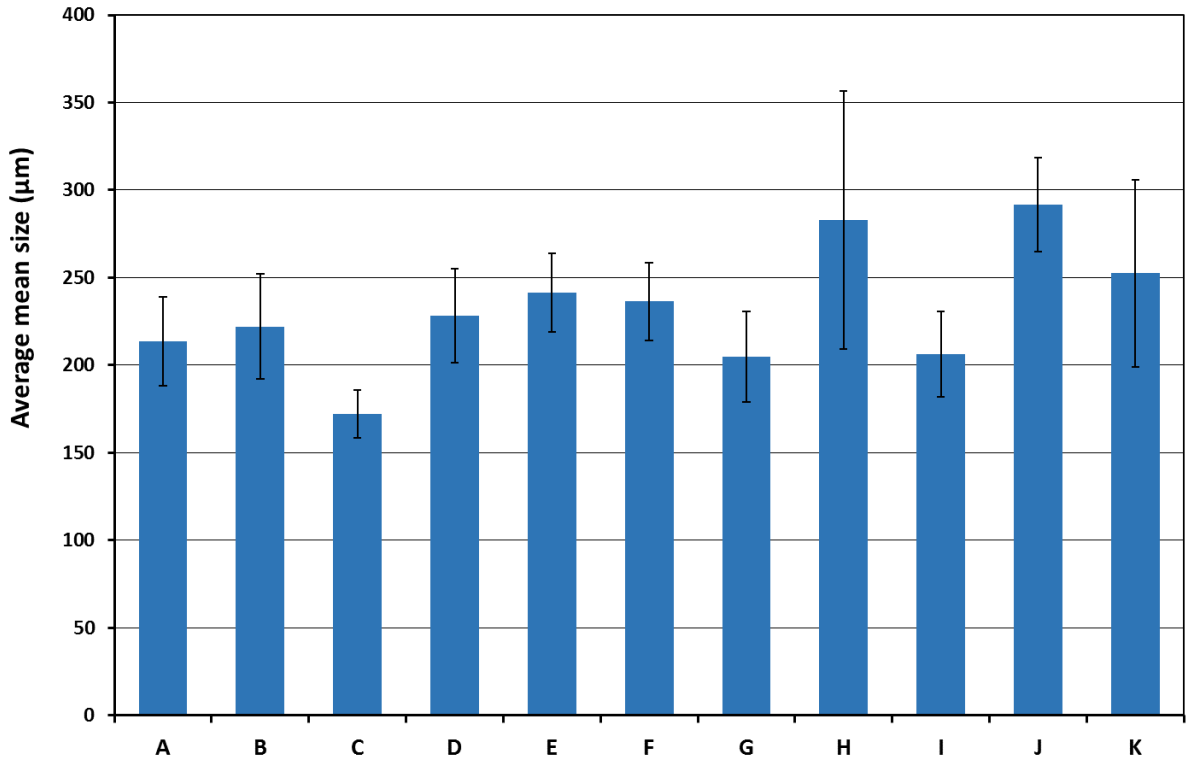

Figure 2. Average mean crystal size of C-sugar samples for factories A-K collected during the 2018/19 grinding season.

The average mean, D10%, D50%, D90%, and coefficient of variance (CV)-values for C-sugar samples collected during the past five grinding seasons are summarized in Table 3. An improvement in the size distribution of C-sugar crystals can be observed, with mean values getting close to the desired 250 µm. The average mean crystal size for C-sugar increased from 196 µm (2014/15 season) to 232 µm (2018/19 season). Size increments were also observed for D10%, D50%, and D 90%, with crystal sizes increasing from 31 to 81 µm, 180 to 222 µm, and 392 to 396, respectively. It is worth mentioning that CV values have gradually decreased over the years, from 0.70 (2014/15 grinding season) to 0.52 (2018/19 grinding season), an indication of a more consistent crystal size distribution.

Table 3. Grinding season summary of 10-percentile, 50-percentile (median), 90-percentile, Mean, and CV-values of C-sugar crystal size distribution (average values ± standard deviation).

Grinding Season	Number of Samples	D 10% [µm]	D 50% [µm]	D 90% [µm]	Mean [µm]	CV
2014/15	127	31 ± 12	180 ± 34	392 ± 28	196 ± 25	0.70 ±0.07
2015/16	377	32 ± 15	184 ± 50	405 ± 41	203 ± 35	0.71 ± 0.11
2016/17	247	41 ± 16	202 ± 47	409 ± 44	214 ± 34	0.65 ± 0.10
2017/18	194	59 ± 16	203 ± 36	407 ± 26	219 ± 25	0.60 ± 0.07
2018/19	225	81 ± 21	222 ± 35	396 ± 52	232 ± 35	0.52 ± 0.06

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DO LATE-SEASON APPLIED SYNTHETIC AUXIN HERBICIDES INFLUENCE STARCH AND COLOR IN COMMERCIAL SUGARCANE CULTIVARS?

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INTRODUCTION

The synthetic auxin herbicides dicamba and 2,4-D are primarily used as a selective herbicides to control broadleaf weeds by mimicking plant growth hormones. Susceptible plants respond by uncontrolled growth which leads to plant death. Most grasses, including sugarcane, are not susceptible to labeled-use rates of dicamba or 2,4-D. Dicamba and 2,4-D are often applied aerially to sugarcane from June through August to control vining weeds such morningglory spp. and tievines and commercially acceptable control is achieved when the herbicide dose is matched with weed size (Siebert et al. 2004). In a low-light environment, morningglory spp. climb towards light as they compete with sugarcane for light. Tendrils wrap around the sugarcane stalk where they provide stability for additional growth. When not controlled, a dense mat is formed over the crop canopy reducing light quality and quantity necessary for photosynthesis and slows harvesting efficiency.

In Louisiana, chemical ripening is necessary to increase sucrose concentration in early-season harvested sugarcane. The use of glyphosate, an herbicide that inhibits aromatic amino acid production, as a chemical ripener has been widely adopted by sugarcane growers in Louisiana. However, increased starch accumulation in sugarcane tissues in certain sugarcane cultivars treated with glyphosate has been documented (Eggleston et al. 2007, 2017). Starch accumulation was greater in green leaves and the middle portion of the stalk of LCP 85-384 treated with glyphosate when compared to starch accumulation in the aforementioned tissues not exposed to glyphosate (Eggleston et al. 2007). Little or no information is available on the possible effects of late-season applied synthetic auxin herbicides on starch and color quality. The objective of this research was to evaluate starch and color quality following a late-July treatment of 2,4-D (Weedar64® applied at 1,120 g ae ha^{-1}), dicamba (Sterling Blue® applied at 560 g ae ha^{-1}), and a pre-mixture of dicamba plus 2,4-D (Brash® applied 289 g ae ha^{-1} plus 831 g ae ha^{-1}, respectively), to HoCP 96-540, L 01-299, and HoCP 04-838 sugarcane cultivars.

EXPERIMENTAL

Total, Insoluble, and Soluble Starch.

Juice samples were analyzed for total, soluble, and insoluble starch using the microwave-assisted sonication/iodometric USDA Research method (Cole et al. 2016). The Brix in juice was first measured and starch was assayed in triplicate; concentrations are quoted as average ppm on a Brix basis. Brix was measured using an Index Instruments (Kissimmee, FL, USA) TCR 15-30

temperature-controlled refractometer accurate to ± 0.01 Brix, and results expressed as the mean of triplicates.

Color

Color was calculated according to the official ICUMSA method GS2/3-9 (1994) for sugarcane products, with slight modifications. Juices (10 mL) were diluted in triethanolamine/hydrochloric acid buffer (10 mL; pH 7) and filtered through a 0.45 µm PVDF filter. Color was also measured at pH 4, 8.5, and 9 by adjusting the pH with HCl and NaOH solutions, respectively. Results are expressed as the mean of triplicates.

RESULTS AND DISCUSSION

Total starch

An abundance of starch in the streams of the sugar factory and refinery is known to cause processing difficulties. Typically 250 ppm starch (using ICUMSA GS-17 method) in raw sugar is often quoted as the maximum amount compatible with satisfactory refining conditions in Louisiana (Eggleston et al. 2017). It is also now known that insoluble starch is very persistent across the whole factory, as well as in raw and even refined sugars (Cole et al. 2013), which has negative implications on amylase factory applications as well as syrup/liquor viscosity and sugar recovery. Fortunately, a new USDA Starch Research method is now available to measure both insoluble and soluble starch (Cole et al. 2016), which was used in this study to measure total, insoluble, and soluble starch (Figures 1 to 3).

In this study, the cultivar by synthetic auxin treatment interaction influenced total starch (ppm $Brix^{-1}$) (Figure 1). Total starch for L 01-299 and HoCP 04-838 was not influenced by herbicide treatment; however, total starch was 60% greater when HoCP 96-540 was treated with dicamba when compared to 2,4-D. Eggleston et al. (2007) reported total starch varied by sugarcane cultivar. A similar amount of total starch was present in cultivars L 01-299 (535 ppm $Brix^{-1}$) and HoCP 96-540 (712 ppm $Brix^{-1}$); however, total starch in HoCP 04-838 was 49 to 99% greater than L 01-299 and HoCP 96-540 (*data not shown*).

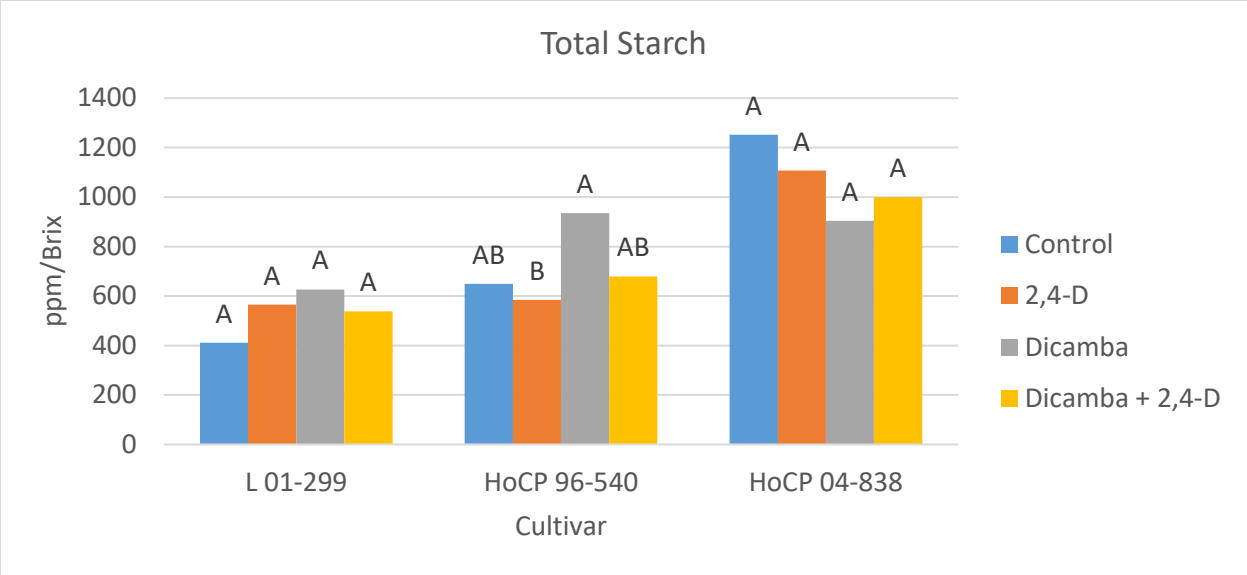

Figure 1. Total starch for L 01-299, HoCP 96-540, and HoCP 04-838 treated to the synthetic auxin herbicides 2,4-D (Weedar64®), dicamba (Sterling Blue®), and a pre-mixture of dicamba + 2,4-D (Brash®). Treatment means within cultivar that are followed by the same letter are not statistically different according to adjusted Tukey's test at $\alpha \leq 0.1$. Data were log-transformed and means were back-transformed for presentation.

Soluble Starch

Soluble starch was calculated by subtracting insoluble starch from total starch (Cole et al. 2016). Most starch in extracted sugarcane juices is insoluble because the juice has not been subjected to heat (Eggleston et al. 2017). In this study, however, small but significant amounts of soluble starch were detected (Figure 2). This may be due to natural amylase (diastase) enzymes solubilizing some starch in the plant itself and could have been influenced by high rains, although further studies are needed to confirm this.

The cultivar by synthetic auxin treatment interaction influenced soluble starch for cultivars L 01-299 and HoCP 96-540 (Figure 2). For cultivar L 01-299, all synthetic auxin treatments increased soluble starch 61% or more when compared to the non-treated control. A different response was observed with HoCP 96-540 where dicamba containing herbicides increased soluble starch 54 to 91% more than 2,4-D. The herbicide treatments failed to increase or decrease soluble starch for HoCP 04-838.

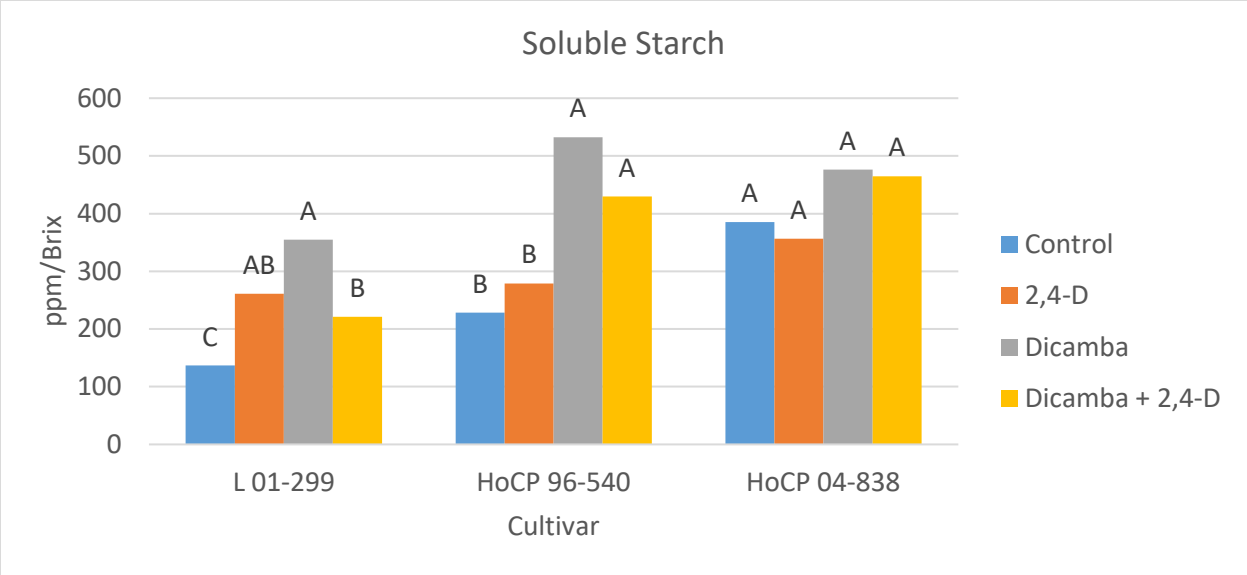

Figure 2. Soluble starch for L 01-299, HoCP 96-540, and HoCP 04-838 treated to the synthetic auxin herbicides 2,4-D (Weedar64®), dicamba (Sterling Blue®), and a pre-mixture of dicamba + 2,4-D (Brash®). Treatment means within cultivar that are followed by the same letter are not statistically different according to adjusted Tukey's test at $\alpha \leq 0.1$. Data were square root transformed and means were back-transformed for presentation.

Insoluble Starch

Similar to the parameters total starch and soluble starch, the cultivar by synthetic auxin treatment interaction influenced insoluble starch, especially for HoCP 04-838 (Figure 3). The synthetic auxin herbicide treatment effect on insoluble starch for cultivars L 01-299 and HoCP 96-540 was not observed. Although the synthetic auxin herbicide treatment did not influence soluble starch for HoCP 04-838, insoluble starch was sensitive to the synthetic auxin herbicide treatment. Dicamba containing treatments reduced insoluble starch 38 to 51% when compared to the non-treated control.

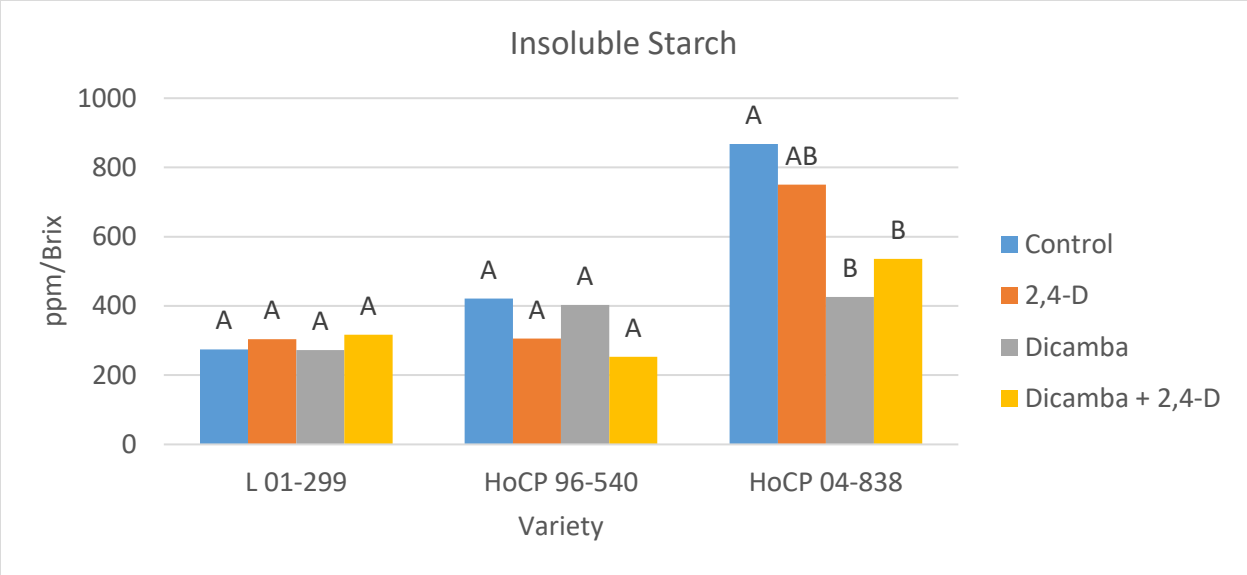

Figure 3. Insoluble starch for L 01-299, HoCP 96-540, and HoCP 04-838 treated to the synthetic auxin herbicides 2,4-D (Weedar64®), dicamba (Sterling Blue®), and a pre-mixture of dicamba + 2,4-D (Brash®). Treatment means within cultivar that are followed by the same letter are not statistically different according to adjusted Tukey's test at $\alpha \leq 0.1$. Data were log-transformed and means were back-transformed for presentation.

Color

The color of the juice was measured at pH 4, 7, 8.5, and 9 according to ICUMSA protocols. As expected, colors at pH 4 and 7 were lower than at pH 8.5 and 9 (Figure 4), because color intensity depends on the solution pH due to changes in molecular structure, ionization, and association-dissociation equilibria (Eggleston et al. 2017). The changes are not linear and color intensity changes most steeply in the pH range 6 to 8. This is one of the reasons that some U.S. sugarcane refiners measure color at pH 8.5 – a value at which the rate of color change has diminished. In this study, late-season applied synthetic auxin herbicides did not influence color at any pH level (Figure 4).

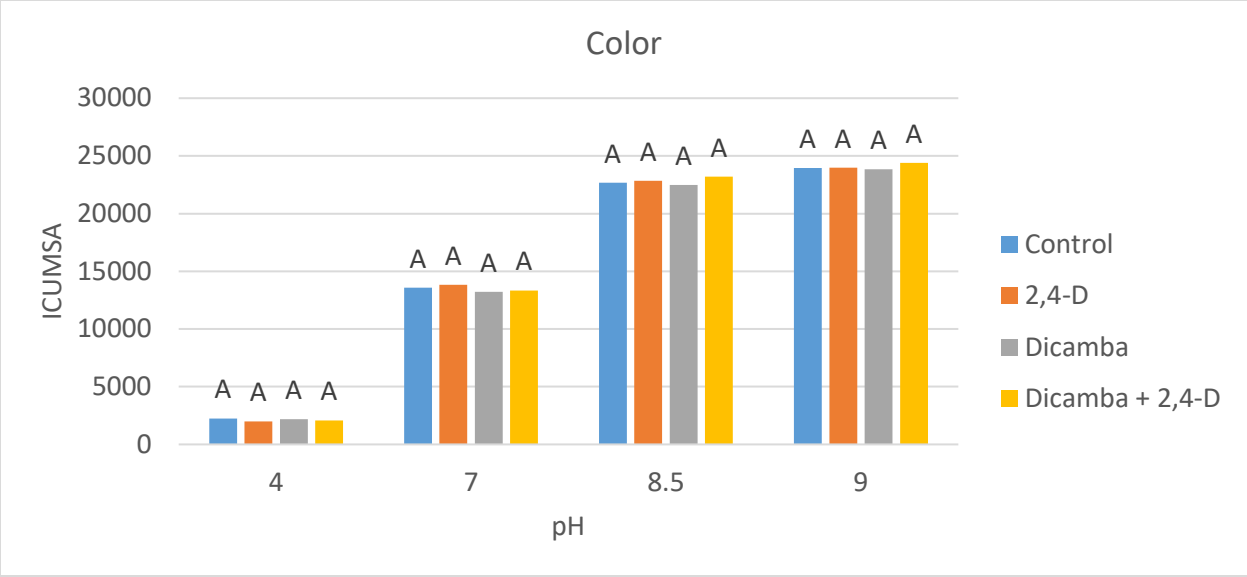

Figure 4. Influence of synthetic auxin herbicides 2,4-D (Weedar64®), dicamba (Sterling Blue®), and a pre-mixture of dicamba + 2,4-D (Brash®) on color at pH levels of 4, 7, 8.5, and 9. Treatment means within pH level that are followed by the same letter are not statistically different according to adjusted Tukey's test at $\alpha \leq 0.1$.

CONCLUSIONS

High concentrations of insoluble starch and soluble starch can cause processing difficulties (Cole et al. 2013). The influence of synthetic auxin herbicides on soluble and insoluble starch depended on the sugarcane cultivar treated. All synthetic auxin herbicides increased soluble starch for L 01-299; however, only dicamba and the pre-mix formulation of dicamba plus 2,4-D increased soluble starch for HoCP 96-540. Insoluble starch accumulation was not influenced by the synthetic auxin herbicide for both L 01-299 and HoCP 96-540. To control vining broadleaf weeds and to minimize increases in soluble and insoluble starch in L 01-299 and HoCP 96-540, results from these data suggested 2,4-D may be a better fit. Dicamba and the pre-mix formulation of dicamba plus 2,4-D reduced insoluble starch for HoCP 04-838 and the herbicides had no effect on soluble starch when compared to the non-treated check. There is an added benefit of controlling vining broadleaf weeds with synthetic auxin herbicides and lowering insoluble starch for cultivar HoCP 04-838, particularly when dicamba is applied.

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REFERENCES

Cole M., Eggleston G., Gilbert A., Rose I., Andrzejewski B., St. Cyr E., Stewart D. (2013). The presence and implication of soluble, swollen, and insoluble starch at the sugarcane factory and refinery. International Sugar Journal, 115, 844-851.

Cole M., Eggleston G., Gilbert A., Chung Y. (2016). Development of an analytical method to measure insoluble and soluble starch in sugarcane and sweet sorghum products. Food Chemistry, 190, 50-59.

Eggleston G., Triplett A., Didier K., Petrie E., St. Cyr, E., and Myers G. (2017). How chemical ripeners of sugarcane affect the starch and color quality of juices. Proc. 2017 Sugar Industry Technol. Mtg, Taiwan, LXXVI, No. 1174, 1-11.

Eggleston G., Viator R., Grisham M. (2007). Glyphosate ripener effects on the processing quality of different sugarcane tissues. Proc ISSCT 26, 1460-1467

Siebert J.D., Griffin J.L., Jones C.A. (2004). Red morning glory (*Ipomoea coccinea*) control with 2,4-D and alternative herbicides. Weed Technol. 18, 38-44