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Factory Operations Seminar

Syngenta Pavilion
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April 22, 2014

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HOW DOES MODDUS COMPARE TO GLYPHOSATE FOR RIPENING SUGARCANE

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At the onset of the sugarcane harvest season in mid-September in Louisiana, sugarcane maturity in terms of sucrose accumulation is at its lowest and increases as the season progresses through natural ripening. Application of ripening agents target biochemical processes within the sugarcane plant, resulting in a redistribution of fixed carbon and a shifting of resources into sucrose storage. Use of chemical ripening agents to improve early season sucrose concentration is of critical importance to Louisiana sugarcane processors through improve efficiency and increased daily mill capacity.

Glyphosate has been used as a ripener in Louisiana since 1980 and has become a valuable component of sugarcane production systems. In recent years, however, sugarcane producers have become increasingly concerned with the possible deleterious effects of glyphosate ripener on subsequent ratoon crops; mainly, retardation of regrowth, leaf chlorosis, and reduced shoot population. Furthermore, there is interest in evaluating alternatives to glyphosate for use in sugarcane production programs.

In 2012, the United States Environmental Protection Agency (EPA) granted registration of trinexapac-ethyl (Moddus 2EC®) as a sugarcane ripener. The label states that sugarcane should be harvested 28 to 60 days after trinexapac-ethyl application. For glyphosate sugarcane should be harvested 21 to 49 days after application. Trinexapac-ethyl has been an effective ripener in Brazil and Australia. Unlike glyphosate, trinexapac-ethyl is classified as a plant growth regulator targeting gibberellin biosynthesis.

A replicated, large scale study was conducted on a second stubble field of HoCP 96-540 at Blackberry Farms in Vacherie. Aerial application of Moddus (19 oz/A) was applied on August 19, 2013, and Roundup PowerMax (5.3 oz/A) on September 17, 2013. Plots were harvested October 15, 2013, resulting in a ripener treatment duration of 57 days for Moddus and 28 days for Roundup PowerMax. Cane was harvested by combine and scale weights were obtained from Lafourche Sugar Factory. Core sample analyses for obtaining the yield of theoretical recoverable sugar per ton of cane (TRS) were obtained from both front and rear compartments of all trucks that were part of the experiment. Moddus minimally increased TRS by 4.5% above the nontreated control, whereas, a moderate increase of 10.0 % in TRS was observed for sugarcane treated Roundup PowerMax (Table 1). The 2013, TRS findings are consistent with the 2012 large scale ripener study, where Moddus increased TRS by 4.9 % and Roundup PowerMax

increased TRS by 10.2%. In 2013, sugarcane yield and sugar yield was not statically impacted regardless of ripener treatment. This greatly differs from the 2012 report in which cane treated Moddus had higher sugarcane and sugar yields than the control.

Table 1. Large scale field experiment means comparing the efficacy of the ripeners Roundup PowerMax and Moddus to nontreated second stubble HoCP 96-540 at Blackberry Farms, Vacherie, LA in 2013.

Ripener Treatment	TRS lb/ton	% TRS Increase	Sugarcane Yield Tons/A	Sugar Yield lb/A	% Fiber
Nontreated	172.0 b		35.1 a	6036 a	18.9 a
Moddus (19 oz./ac)	179.8 ab	4.5	33.4 a	5989 a	20.6 a
PowerMax (5.3 oz./ac)	189.2 a	10.0	33.6 a	6349 a	20.5 a

MANAGING DAMAGING FREEZE EVENTS IN LOUISIANA SUGARCANE

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Exposure of sugarcane to damaging frosts occurs in approximately 25% of the sugarcane producing countries of the world, but is most frequent on the mainland of the United States, especially in the state of Louisiana. The frequent winter freezes that occur in the sugarcane areas of Louisiana have forced the industry to adapt to a short growing season (about 7 months) and a short milling season (about 3 months). The nature and extent of damage to sugarcane by a freeze depends on the intensity and duration of the freeze, and the weather conditions after the freeze can control or accelerate deterioration. A series of damaging freezes occurred in Louisiana from November 26-30, 2013 where the low temperatures ranged from -2.2˚C (28˚F) in the southern area of the sugarcane belt to -4.4˚C (24˚F) in the northern area of the belt with the line of demarcation being roughly the areas north and south of the I-10 corridor. The duration of the freeze event below I-10 was approximately 6-10 hours, whereas, the duration north of I-10 was greater than 10 hours. At this point in the harvest, approximately 60% of the 14-million ton Louisiana crop had been processed by the state's 11 factories. Immediately following the November freeze events personnel from the LSU AgCenter, the American Sugar Cane League and the USDA-ARS Sugarcane Research Unit did field inspections of the damage to the crop by the freeze. Visual ratings were taken for all commercial and some candidate varieties for both leaf and stalk cold tolerance in the field.

In the southern area there was minimal damage to the stalk with only the apical meristem or terminal bud affected. However, in the more northern areas, the freeze events affected the entire stalk. With approximately 40% of the crop still remaining in the field and to study the reaction of commercial and candidate varieties to these early freeze events, the Outfield Variety Test at Alma Plantation, Lakeland, LA, located in the northern area above I-10 and US Highway 190, was chosen for the study. The test included the commercial varieties, HoCP 96-540, L 01-283, L 01-299, L 03-371 and HoCP 04-838, and the candidate varieties, Ho 07-613, L 09-112, HoCP 09-804 and Ho 09-840. Ten-stalk samples were hand-cut at ground level but not stripped or topped from each of three replications for the first of three sampling dates, Dec. 12, 19 and 26, 2013. Another set of samples was cut on Jan. 3, 2014 but this time the samples were hand stripped and tops were removed approximately 30 cm (12 in) below the apical meristem (terminal bud). All samples were immediately transported, weighed and processed at the Sugar

Research Station at St. Gabriel using the press method of analyses. Juice samples were analyzed for Brix by refractometer and sucrose by polarimetry and bagasse (residue) samples were analyzed for moisture (by drying). The Brix, sucrose, purity and fiber content of the cane were then calculated from these analyses after which the estimated yield of theoretical recoverable sugar per ton of cane (TRS/TC) was calculated. Juice samples were also analyzed for pH, titratable acidity, total polysaccharides and mannitol. Further, results were compared to actual factory data for daily core juice pH, crusher juice polysaccharides, syrup purities, C massecuite viscosities and sugar yield from Alma Plantation (Lakeland, LA) and syrup purities and sugar yield from the Leighton factory located at Thibodaux, LA (in the southern area below I-10).

Immediately following the field assessment, the LSU AgCenter issued best management practices (BMP) to be used in reducing the impact of the freeze events on sugar yield. Those BMPs stressed the need to deliver high quality cane to the factories free of mud, deteriorated tops and leaves and other trash. The BMP's indicated that growers and processors should not panic as the industry had experienced freeze events of this magnitude many times before. Since areas of higher elevation tend to be warmer, the BMPs recommended that growers should harvest fields with lower elevation first. Also, growers were informed that varieties with poor stalk cold tolerance, i.e., L99-226, L99-233 and L03-371, should be harvested first. Other items discussed in the BMPs included standing vs. down cane, topping height and whether or not one should burn. It also warned of overnight sleeper loads that could lead to increased deterioration.

Reaction of Louisiana Sugarcane Varieties to Freezing Conditions [1]

Resistant	Intermediate	Susceptible
HoCP 04-838* (9)	LCP 85-384 (29)	L 99-226 (59)
	HoCP 96-540 (24)	L 99-233 (70)
	L 97-128 (23)	L 03-371 (61)
	HoCP 00-950† (50)	TucCP 77-42** (64)
	L 01-283 (34)	
	L 01-299 (36)	

[1] Number in parenthesis weighted average of five parameters: sucrose, purity, TRS, ph and titratable acidity. Smaller the number the better the rating for stalk cold tolerance. Rating also a measure of deterioration as measured by dextran concentration.

* Candidate varieties; ** Argentine commercial variety; † Intermediate to Susceptible

Fig. 1. Stalk cold tolerance of Louisiana sugarcane varieties following a freeze event.

Data from the Outfield Test at Alma indicated that most of the parameters measured for the samples with tops and leaves, i.e., pH, titratable acidity, total polysaccharides, TRS/TC, remained relatively stable (unchanged) over the sampling period although it became increasingly impossible to clarify juice samples in the lab with aluminum chloride on the Dec. 26 sampling date. The Alma factory data, however, showed that the core lab juice pH, syrup purity and sugar yield started a slow decline over the same period. On the other hand, total polysaccharides in the crusher juice and C-massecuite viscosity at Alma showed significant declines after the freeze events with the BMPs in place.

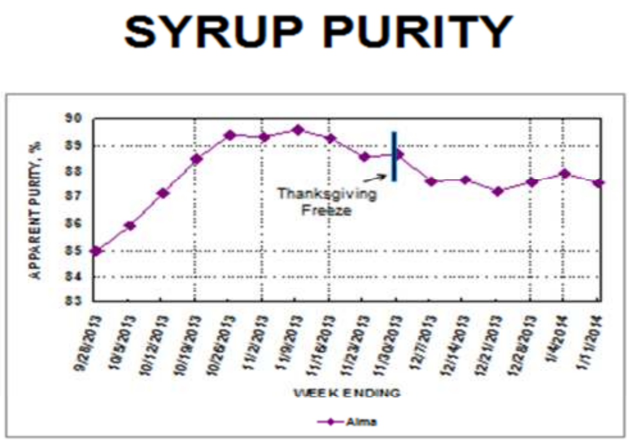

Fig. 2. Syrup purity for Alma Plantation during the 2013-2014 crop harvest before and after the freeze events of Nov. 28-30, 2013.

For the Leighton factory operating south of I-10, syrup purities and sugar yield actually continued to rise in spite of the freeze events and a wet harvest. In general, ambient temperatures following the freeze events were cooler than normal although there was one record daily high temperature of 29˚C (84˚F) on Dec. 5.

SYRUP PURITY

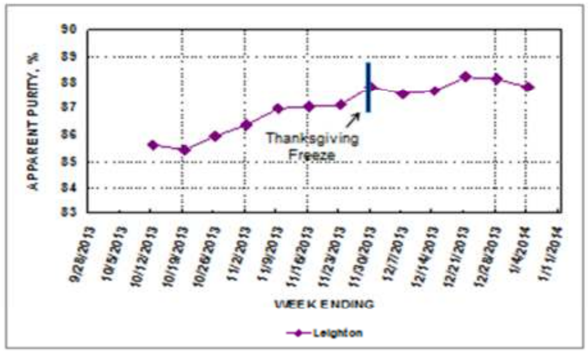

Fig. 3. Syrup purity for Leighton Factory during the 2013-14 crop harvest before and after the freeze events of Nov. 20-30, 2013.

With the BMPs in place, the Alma factory experienced no difficulties in the boiling house without any indication of c-axis elongation of sugar crystals even with the last strike of the 2013-2014, which was processed on Jan. 6. It is interesting to note that on the final sampling date of the Outfield Test, Jan. 3, where tops and leaves were removed, there was no problem in clarifying juice samples in the lab at the Sugar Research Station while at the same time there was a significant reduction in juice pH and total polysaccharides and higher TRS/TC for all varieties in the test from the previous sampling date, Dec 26. All other parameters remained the same as the Dec. 26 sampling dates when all tops and leaves were not removed. These data showed that the BMPs implemented at the time of the freeze proved to be an effective tool in mitigating the effects of the freeze events of the magnitude that occurred on Nov. 28-30 and that factories could continue to operate with minimal problems in the boiling house so long as the frozen tops and leaves were removed. Even with these BMPs in place, however, it appears that the freeze events of Nov. 28-30 reduced overall state sugar yields by approximately 5.0 kg/tonne (10 lbs/ton) and by removing the top 30 cm (10 in) of the stalk, field yields were reduced by approximately 6.75 tonnes/ha (3 tons/ac) such that the overall loss in sugar yield per hectare for the 2013-2014 crop amounted to about 33.6 kg/ha (30 lbs/ac).

Fig. 4. Photo of normal sugar crystals with no c-axis elongation taken on the last day of the 2013-14 crop harvest at Alma Plantation on Jan. 6.

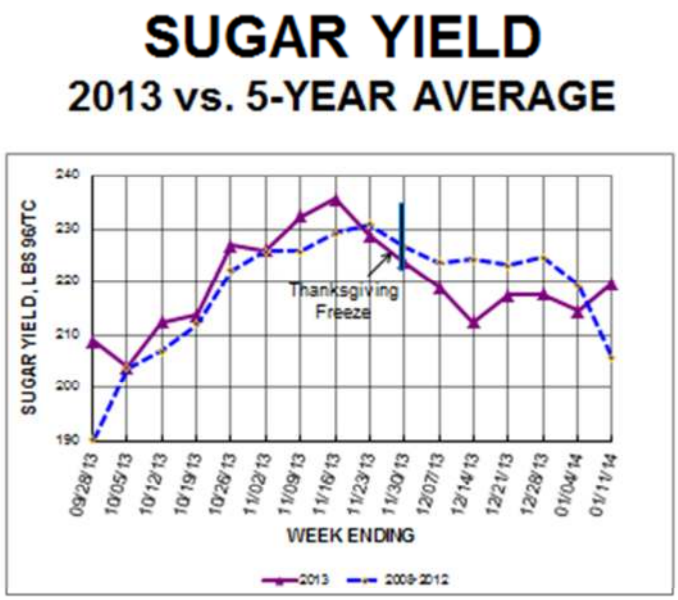

Fig. 5. Sugar yield for the 2013-14 crop harvest vs. five year average.

FILTER STATION OPERATIONS

H. Birkett and J. Stein

INTRODUCTION

The two important issues regarding filter cake in the production of raw sugar are that of pol losses and capacity (handling large quantities of filter cake or mud). The recent introduction of belt filters to the sugar industry is of interest to all Louisiana raw sugar mills. The objectives of the project are to review filter operations in general and compare belt filters with that of the traditional rotary drum filters.

METHOD

In an effort to compare belt filters with rotary drum filters several areas of interest were investigated over the 2012 and 2013 crops. These included pol losses, bagacillo ratio (bagacillo % feed/mud solids % feed), filter retention (mud in filter cake/mud in feed), filter capacity (filter cake production and removal of mud), belt wash water loss and flocculant usage.

Samples of the following were collected at all Louisiana factories: clarifier underflow, feed to filters, filtrate, filter cake and belt wash water. The underflow and filter feed were analyzed for Brix, pol, moisture and bagacillo content. Mud filtrate samples were analyzed for Brix, pol and sediment. Filter cake was analyzed for pol, moisture and bagacillo.

Brix and pol determinations were based on standard sugar laboratory methods (Chen & Chou 1993). Moisture was measured by drying a known amount of sample at 105°C for 24 hours or until constant weight. Sediment was determined using the standard core lab method for juice (Birkett 1998). Bagacillo content was determined by washing a known quantity of sample with water through a 200-mesh screen until washings appeared clear. Large quantities of water were used, however, dirt was still retained by the bagacillo. This led to necessary ash determinations (Birkett & Stein 2004). Actual bagacillo % sample was determined after taking into account the remaining ash in the washed sample.

RESULTS AND DISCUSSION

Figure 1 shows the pol % filter cake average of 3.47. Pol % filter cake from drum filters ranged from 1.00 to 7.36 and averaged 4.08% pol. Belt filters had an average pol % filter cake of 2.36 with a range of 0.46 to 5.86%.

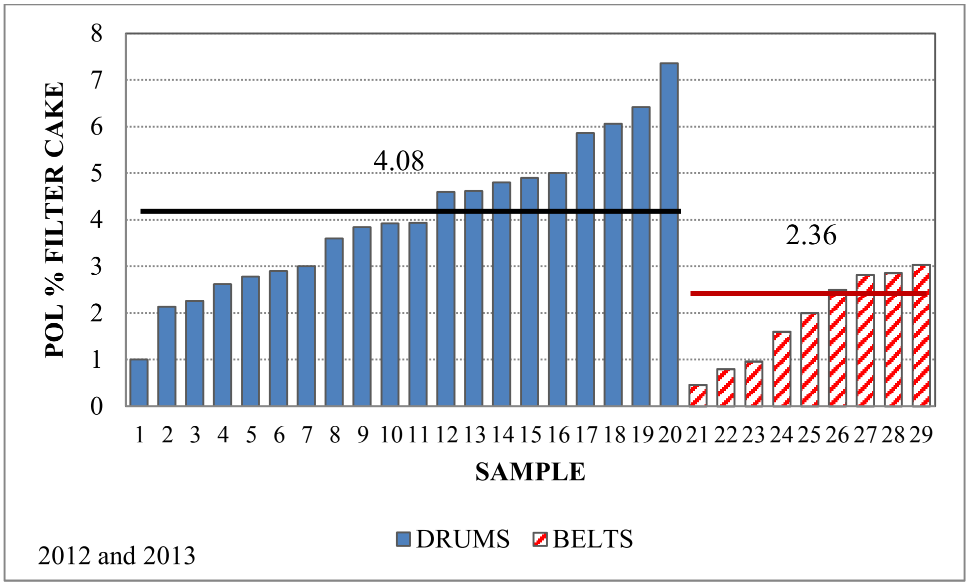

Figure 1. Pol % filter cake for drum and belt filters.

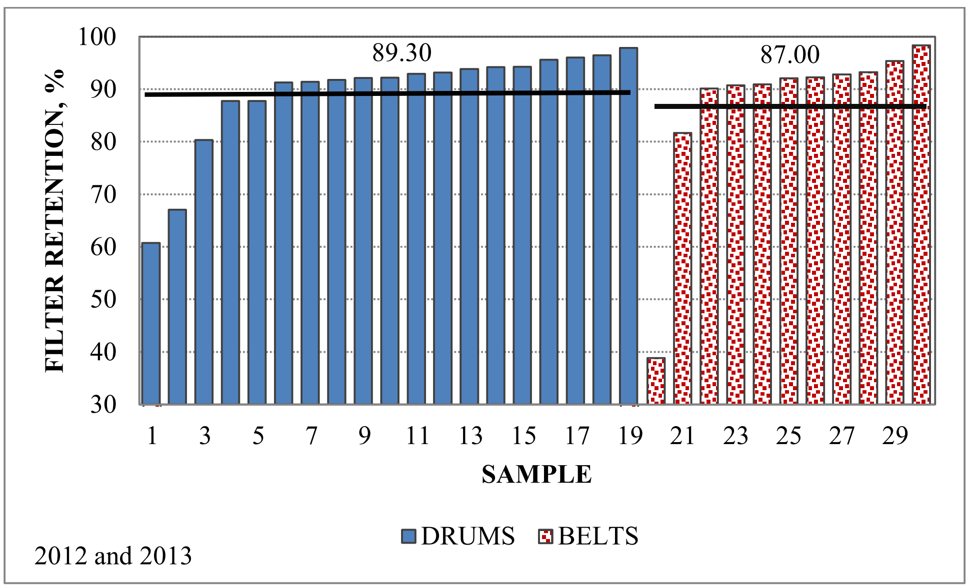

Figure 2. Filter retention for drum and belt filters.

The retention of all filters tested averaged 88.48% as shown in Figure 2. Retention varied from 38.89 to 98.38%. Retention rates above 90% are desirable.

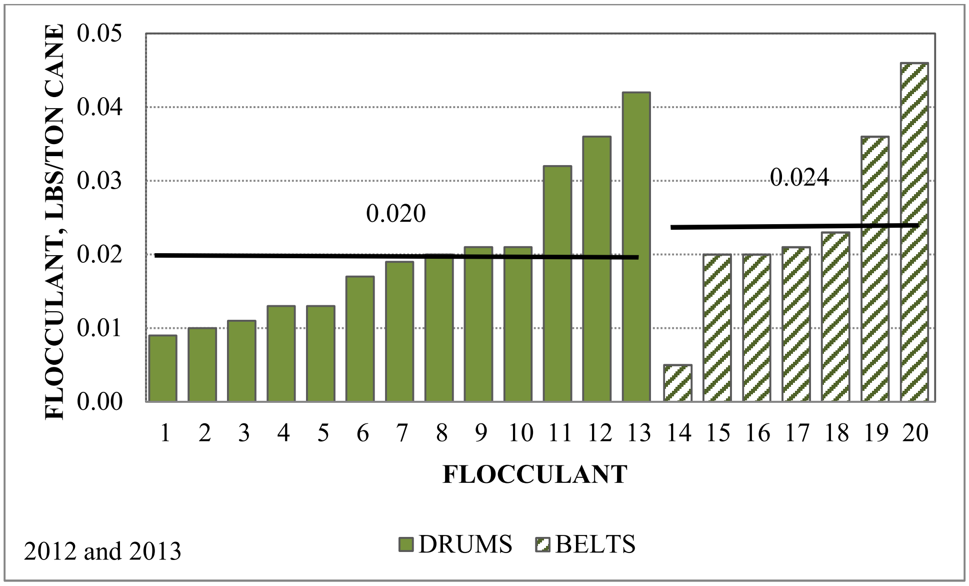

Figure 3. Crop average flocculant usage at the filter station.

Crop flocculant usage averaged 0.022 lbs./ton cane as shown in Figure 3. Belt filters used a little more flocculant (0.024) on average than drum filters (0.020).

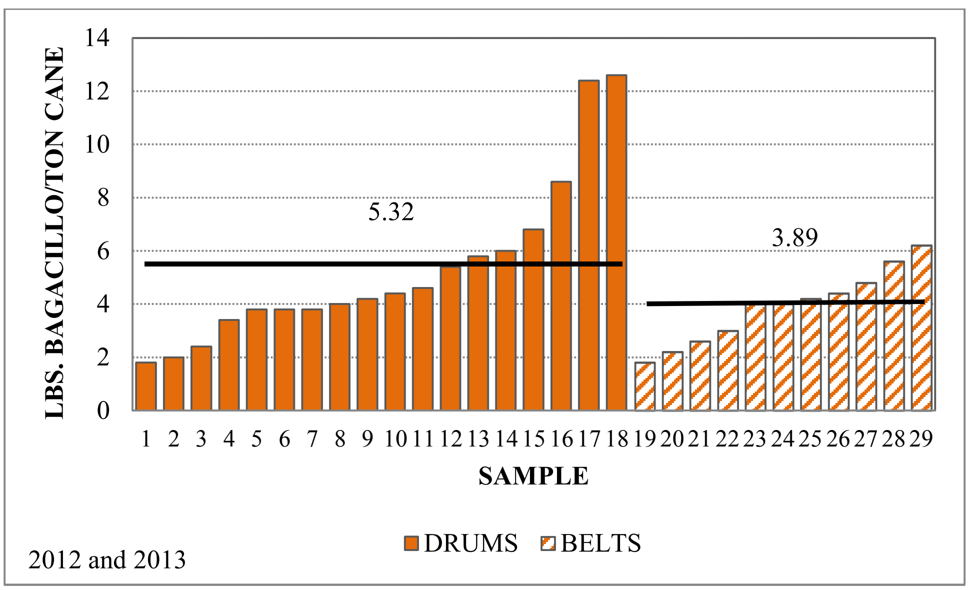

Figure 4. Bagacillo per ton of cane for drum and belt filters.

Figure 4 gives the bagacillo per ton cane for drum and belt filters. Drum filters averaged 5.32 lbs. bagacillo/ton cane ranging from 1.8 to 12.6. Belt filters averaged 3.89 lbs./ton cane of bagacillo with a range of 1.8 to 6.2 lbs./ton cane.The overall average was 4.78 lbs. bagacillo/ton cane.

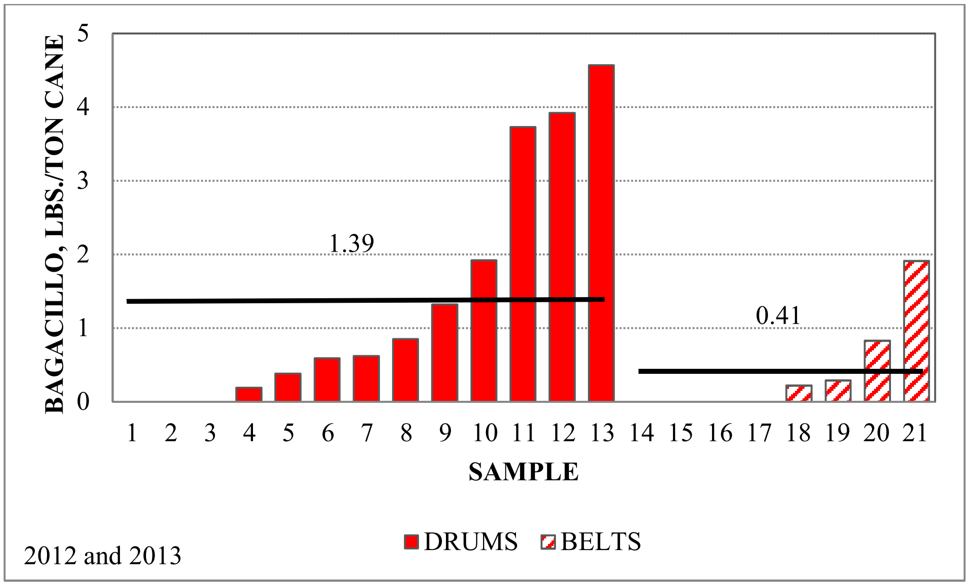

Figure 5. Pounds bagacillo added per ton cane for drums and belts.

Pounds of bagacillo/ton cane added to filter cake averaged 0.76 overall. Shown in Figure 5 is the amount added for drums, ranging from 0 to 4.57 and averaging 1.39 lbs./ton cane. The addition of bagacillo for belt filters averaged 0.41 lbs./ton cane. Less than 30% of bagacillo in filter cake was added as screened bagacillo from mill-run bagasse with the vast majority provided from the clarifier underflow.

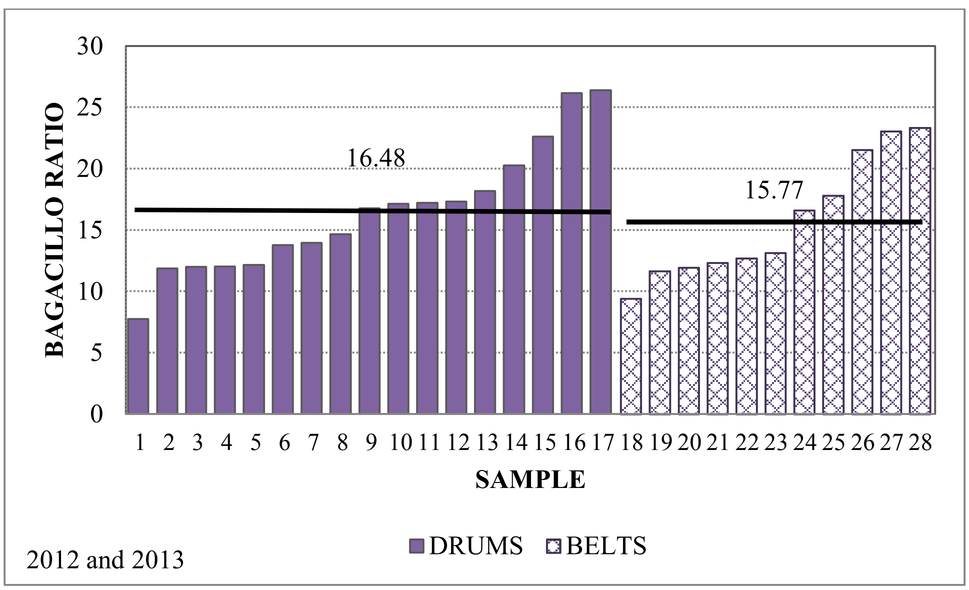

Figure 6. Bagacillo ratio for drum and belt filters.

The overall bagacillo ratio averaged 16.20 which was similar for both the drum filters (16.48) and belt filters (15.77) as displayed in Figure 6. Internationally, bagacillo ratios of 80 are recommended. To achieve, this ratio about five times as much bagacillo as currently used would be required.

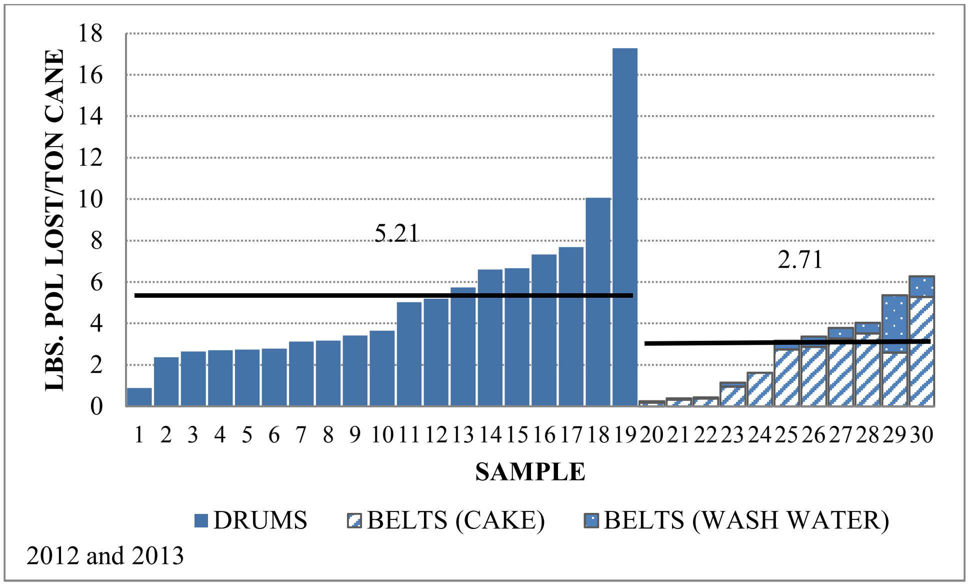

Figure 7. Pounds pol lost per ton of cane with belts based on 100 gpm wash water.

Figure 7 shows that on average 5.21 lbs. pol/ton of cane are lost in filter cake for rotary drum filters. Belt filters lost on average 2.71 lbs. pol/ton cane. This included losses in the belt wash water based on a rate of 100 gallons per minute.

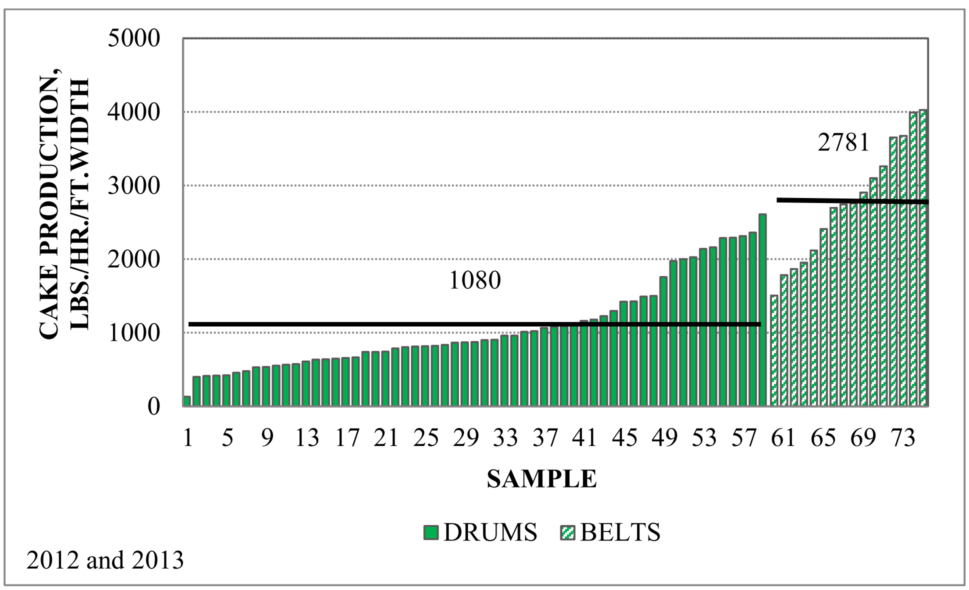

Figure 8. Filter cake production for both drum and belt filters.

The overall average filter cake production was 1443 lbs./hr./ft. width as shown in Figure 8. Drum filters averaged 1080 lbs. filter cake and ranged from 128 to 2606 lbs. filter cake/hr./ft. width. Belt filters averaged 2781 lbs./hr./ft. width, almost three times the amount for drums, and ranged from 1509 to 4027 lbs./hr./ft. width.

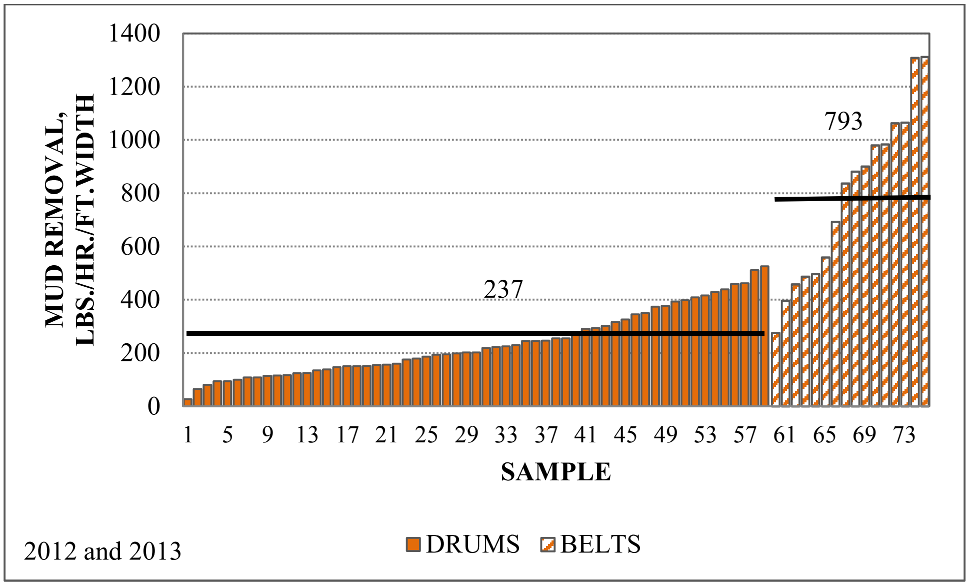

Figure 9. Amount of mud removal for drum and belt filters.

Mud removal averaged 356 lbs./hr./ft. width for all filters tested. Figure 9 shows the average mud removal for drum filters was 237 lbs. mud/hr./ft. width and ranged from 26 to 526 lbs. Mud removal for belt filters is also shown, averaging 793 lbs. mud/hr./ft. width and varying from 275 to 1311 lbs./hr./ft. width.

SUMMARY

In general, filter operations are highly variable with much scope for improvement. Most of the bagacillo is obtained from the underflow with very little coming from bagasse screening. The bagacillo ratio (bagacillo/mud) of 16% is very low. Filter retention of 88% is generally very good.

Regarding belt filters only, the capacity seems to be very high with sugar losses comparable to that of drums. Maintenance costs have yet to be determined. Options for disposal of belt wash water should be considered.

ACKNOWLEDGEMENTS

We would like to express our thanks and appreciation to the American Sugar Cane League and all of the participating factories and personnel for their support of this project.

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FILTRATE CLARIFICATION DEMONSTRATION PLANT

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The clarification of filtrate using a stand-alone dedicated clarification unit has been proposed as a way to eliminate the recycle of filtrate to the mixed juice clarifiers. In a standard clarification process, the filtrate juice from the mud filters is sent to the juice heaters by mixing with the incoming raw juice and consequently returned to the clarifiers for impurity removal. In filtrate clarification, instead of the filtrate being returned to the standard clarifiers, a separate juice heater, lime addition, and clarifier is added solely for processing filtrate. The clear juice from the filtrate clarifier is forwarded directly to the evaporators, while the mud from the filtrate clarifier is returned to the mud mixer. Past projects for filtrate clarification utilizing settling clarifiers (Conijn, 1962; Gayle, 1956) and flotation clarifiers (Bento, 2006) have been reported. The current research effort is a continuation of the work performed by Audubon Sugar Institute during the 2012-2013 filtrate clarification trials (Grimaldo, 2013).

The trials conducted by Grimaldo included batch settling tests and a pilot plant clarifier unit that was installed on-site at a sugar mill. The research focused on optimal chemical dosage rates with an emphasis on suspended solids reduction and turbidity reduction. The unit consisted of a plate and frame heat exchanger, hot liming with lime saccharate, and a 4.5 ft diameter clarifier. The pilot clarifier was operated at flowrates of 40 gpm and 60 gpm, with a residence time of 10 to 12 minutes. Operation of the unit was on a non-continuous basis, in which the unit was started, allowed to reach steady state, samples were taken, and the unit was liquidated daily. The clear filtrate and mud that was obtained were mixed and returned to the filtrate.

The current project is a scale up of this process with the goal of having a unit that can be reliably operated online on a continuous basis. The unit consisted of a plate and frame heat exchanger and a 6 ft diameter trayless LLT clarifier. The filtrate comprised of juice being sourced from horizontal belt filters, with a brix typically around 8. The clear filtrate was forwarded to the evaporators, and the mud was returned back to the filtrate going to the clarifiers. It was found that the incoming filtrate had a phosphate concentration as low as 10 ppm, thus addition of phosphoric acid was required to promote settling. A 75% concentration phosphoric acid was used to bring the phosphate concentration to a level higher than 300 ppm. The phosphoric acid was added upstream of the juice heater. The plate and frame juice heater was a narrow gap two-pass heater with automatic flow reversal to reduce plugging. The filtrate was heated to 215-218^{o}F. Hot liming using lime saccharate was utilized to adjust the pH of the filtrate to pH 7.5. The filtrate was then flashed and introduced into the clarifier.

The clarifier was able to operate successfully without floating solids up to 110 gpm, or a 15 minute residence time, with the limitation being juice heating capacity. Typical operating flowrate ranged from 50 gpm to 70 gpm, which gave a residence time of 23-33 minutes. This flowrate was chosen to enable enough tolerance for fouling and control of steam pressure fluctuations that could affect the heat transfer in the juice heater over multiple runs.

In operating the clarifier, it was found that adequate mud control was the major factor contributing to stable, reliable operation. The clarifier was designed with a steep (45^{o}) cone, without a scraper system, and a screw pump with a VFD was used to control mud withdrawal. Initially, phosphoric acid was added to bring the phosphate level to 400 ppm, lime was added to adjust the pH to 7.5, and the mud pump speed was adjusted to control mud level and mud consistency. A daily run typically consisted of a 4-5 hour online operation of the unit. Flocculant dosage rates were set per daily run, with the goal of determining the window of optimal flocculant rates needed for stable mud level control, where the mud is dense enough for the mud flowrate to be within the mud pump's capabilities, while having a mud thin enough that it can flow to the mud outlet and be removed. After trial and error over multiple runs, a flocculant dosage rate that gave successful mud withdrawal over multiple runs was found and the unit was left online for operation by the mill's operators for a longer term analysis of stability. Turbidity measurements during this long term run can be viewed in figure 1.

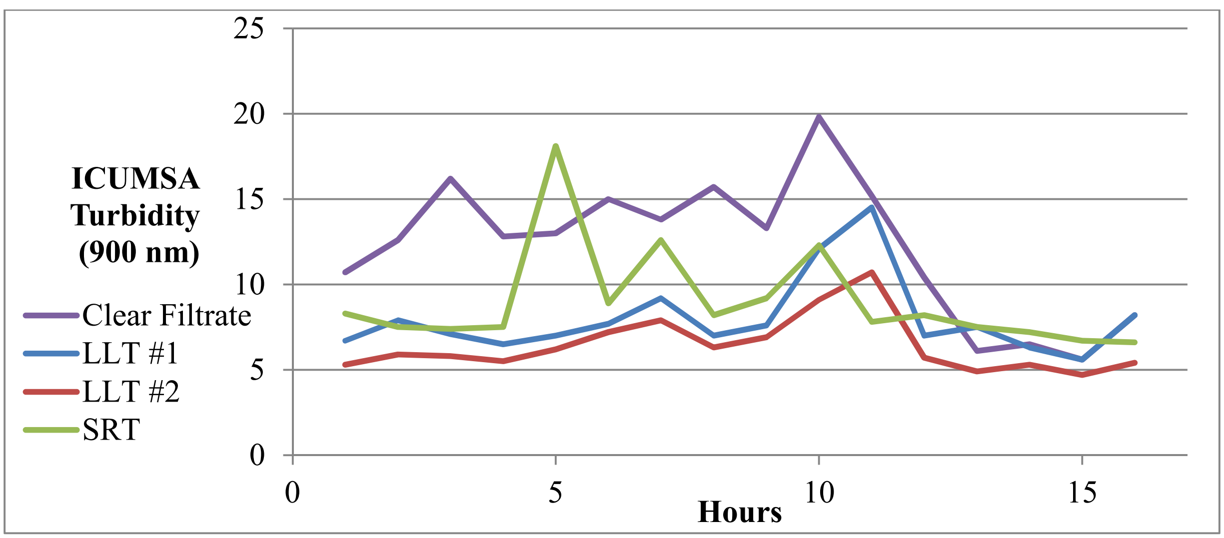

Figure 1. ICUMSA turbidity measurements comparing the clear filtrate to the clear juice turbidity in the mixed juice clarifiers over a 16 hour run.

The clarifier was stable over the run, but was taken offline due to high mud levels at the 16th hour. The turbidity of the clear filtrate was higher than the clear juice in the mixed juice clarifiers, though deemed of acceptable quality by mill personnel for forwarding to the evaporators. Also note, the filtrate clarifier is of an LLT design, similar to the two LLT clarifiers processing mixed juice.

After the 16 hour run, a mud scraper system was added to increase the predictability of the mud density. Though this was successful in increasing the operational reliability of the next set of operations, a reduction in the phosphoric acid, reducing the phosphate concentration from 400 ppm to 300 ppm had a dramatic effect of getting a more consistent mud concentration. This agrees with past research on phosphoric acid addition to mixed juice which found that a minimum of final mud volume is encountered near 300 ppm, with final mud volume levels increasing from above and below this concentration (Carter, 1966). With the decrease in phosphoric acid addition, flocculant addition was also decreased while obtaining a higher mud concentration in the underflow.

A second long term run was attempted in which the system was operated by the mill's clarifier operators, (figure 2). The run consisted of 22 hours and was disrupted by a mill stop at the 22nd hour. Significant fouling to the heat exchanger occurred during the course of this test, in which the flowrate of the filtrate had to be reduced from 60 gpm to 20 gpm. Lack of automatic control of the phosphoric acid and manual pH control most likely resulted in the system not being operated optimally given the reduction in filtrate flowrate that took place during the test, requiring a proportional decrease in chemical addition rates. Notice at the 15th hour that the system seemed to stabilize and be in control for 6 hours, after having a constant rise in turbidity in the hours before (even if the measurement at the 6th hour is considered and outlier or inaccurate sample/measurement).

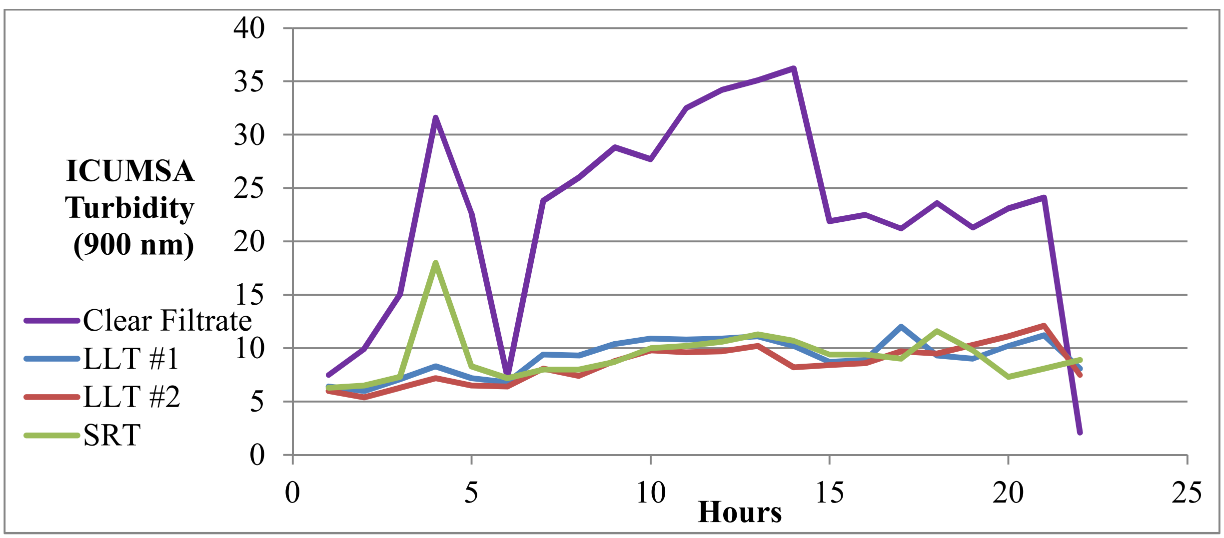

Figure 2. ICUMSA turbidity comparing the filtrate clarifier's clear juice to the clear juice from the mill's mixed juice clarifiers.

After this test, the heat exchanger was cleaned using caustic and underwent a high pressure, high flow flush with water. This allowed the system three more 5 hour daily runs before significant heat exchanger fouling made the system inoperable. The juice heating capacity dropped from 110 gpm to 20 gpm during the three day period. This fast rate of fouling may necessitate the use

of a different design of heat exchanger more resistant to plugging in future applications, such as a shell and tube or a wide gap plate and frame instead of the narrow gap plate and frame used.

CONCLUSIONS

It was found that increasing the phosphates may be necessary when attempting filtrate clarifications. The phosphate concentration can have a major effect on mud withdrawal, and adequate control of phosphoric acid addition is needed to have a consistent mud flow. The turbidity of the test runs ranged between 150-400 NTU, which is higher than that of most mixed juice clarifiers, but generally acceptable. The rate of fouling of the heat exchanger was alarming after some operating time on the unit, and changes should be made to take potential plugging into consideration. The end result of this project is that operational and potential design changes have been discovered that can help the operation future filtrate clarifiers more dependable.

ACKNOWLEDGEMENTS

I would like to thank Sterling Sugars personnel, especially Mr. Luis Acevado, Mr. Danny King, and Mr. Jacques Giraud for assisting in the design, construction, operation, and troubleshooting of the demonstration unit. I would also like to thank Crompion International, especially Mr. George Schaffer, Angel Proano, and Luis Lopez, for funding, design, and construction support. I also thank the American Sugar Cane League for supporting the advancement of technology in the sugar industry.

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STARCH ANALYSIS OF CLARIFIED JUICE AND SYRUP (Project 13-121)

Giovanna M. Aita

INTRODUCTION

Project 13-121 was funded by the American Sugar Cane League in 2013 and it was a continuation of Project 12-121 (Starch Analysis of First Expressed Juice and Raw Sugar) with aim at finding some answers to a common concern among sugar processors on the discrepancies of starch levels found in raw sugar. It is unclear whether this difference starts in the juice as it enters the factory or it is due to how efficient processing methodologies are employed at each mill. The objective of this project was to analyze for the presence of starch in the clarified juice, syrup from the last body evaporator and syrup from the storage tank during early and late grinding season.

ACCOMPLISHMENTS

All eleven mills participated in this study. Samplings of clarified juice and syrup were taken simultaneously at all mills on Wednesday, October 23rd (early grinding season) and on Wednesday, December 4th (late grinding season). Dr. Aita met with chief chemists from all eleven mills to coordinate the sampling of materials and discuss sampling methodologies. Sampling instructions were provided to each mill and discussed in detail with each chief chemist and personnel. Labeled sampling containers were personally delivered by Dr. Aita to all mills. A survey was developed to gather additional information at the time of sampling (i.e. weather conditions, clarified juice and syrup flow rates, last body evaporator temperatures, syrup storage capacity, use of amylase). Samples and surveys were collected from mills at the end of each sampling date. Samples were analyzed for starch content at the Audubon Sugar Institute. An update report and final report were submitted to the ASCL Funding Committee by the end of grinding.

RESULTS

Project 13-121 evaluated the starch levels from clarified juice and syrup from the last body evaporator and storage tank as per request from the ASCL Funding Committee during early and late grinding. Tables 1 and 6 summarize the weather conditions at the time of sampling for season 2013. Data on starch analysis is presented in Table 2 (during early grinding) and Table 7 (during late grinding). Tables 3, 4, 8, and 9 summarize clarified juice and syrup flows, last body evaporator temperatures and syrup storage capacity. Amylase concentrations and its point of injection at each mill are presented in Tables 5 and 10. Overall starch concentrations during early grinding of clarified juice and syrup were higher than those observed during late grinding. Starch levels discrepancies were observed in the clarified juice, syrup from last body evaporator and

syrup storage tank among all mills. Amylases were applied at two points (syrup tank/evaporator) and concentrations/brand varied among mills.

ACKNOWLEDGEMENTS

Sincere appreciation and gratitude is extended to all sugar factory managers, chief chemists and laboratory personnel for their time and support spent on this project. The author also thanks her research team (Dr. Patrisha Pham, Mr. Zenghui Qiu, Ms. Akanksha Kanitkar, Mr. Fang Deng, Ms. Jing Cao, and Mr. Saeed Oladi) for conducting the starch analysis and the staff from the chemistry laboratory (Ms. Chardcie Verret, Dr. Derek Dorman and Mrs. Shyue Lu) at the Audubon Sugar Institute for instrumentation.

Table 1. Weather conditions at the time of sampling during early grinding.

Sugar Factory	Weather Conditions	
1	Sunny	55-70F
2	Sunny	55-70F
3	Sunny	55-70F
4	N/A	N/A
5	Sunny	55-70F
6	Sunny	55-70F
7	Sunny	70-85F
8	Sunny	55-70F
9	Sunny	70-85F
10	Sunny	55-70F
11	Sunny	70-85F

N/A= Information not available.

Table 2. Starch concentrations from clarified juice and syrup during early grinding.

Sugar Factory	Starch (ppm/Brix)		
	Clarified Juice	Syrup Last Body Evaporator	Syrup Tank Storage
1	1367	1028	344
2	857	175	343
3	898	409	457
4	680	308	362
5	906	270	543
6	1297	323	426
7	1057	421	412
8	1134	102	219
9	1193	238	275
10	944	1053	215
11	1291	1118	179

Table 3. Clarified juice and syrup flow rates, and last body evaporator temperatures during early grinding.

Sugar Factory	Clarified Juice Flow Rate	Clarified Juice Brix	Syrup Flow Rate	Syrup Brix	Last Body Evaporator Temperature (F)
1	2296 GPM	14.7	368 GPM	64.4	150
2	2647 GPM	13.0	594 GPM	62.0	148
3	2560 GPM	15.1	500 GPM	59.4	151
4	N/A	N/A	N/A	N/A	N/A
5	1200 GPM	13.5	N/A	58.0	140
6	2749 GPM	14.2	106 Ton/h	53.2	150
7	2500 GPM	13.3	N/A	65.8	150
8	2700 GPM	12.9	N/A	63.0	150
9	N/A	13.5	N/A	57.0	175
10	N/A	15.8	N/A	68.6	146
11	2210	12.7	454	51.7	134

N/A= Information not available.

Table 4. Syrup flow rates and syrup tank storage capacity during early grinding.

Sugar Factory	Syrup Tank Capacity (CFT)	Syrup Storage Tank to Pans		
		Flow Rate	**Temperature (F)**	**Brix**
1	1600	330 GPM	140	64.4
2	1938	439 GPM	150	63.0
3	4682	500 GPM	140	57.2
4	N/A	N/A	N/A	N/A
5	4144	N/A	130	57.6
6	3200	2430 LbPM	116	60.5
7	8785	350 GPM	150	65
8	4100	2430 LbPM	116	62.3
9	3743	N/A	170	56.8
10	6190	N/A	N/A	N/A
11	3374	N/A	132	51.75

N/A= Information not available.

Table 5. Amylase concentrations and point of injection during early grinding.

Sugar	Amylase			
Factory	**Used**	**Application**	**Brand**	**Concentration**
1	Yes	Syrup Tank	Termamil	2.5 PPM
2	Yes	Evaporator	Protech Magnazyme S	0.015 GPM
3	Yes	Evaporator	Protech	5 PPM
4	N/A	N/A	N/A	N/A
5	Yes	Evaporator	Protech	9 PPM
6	Yes	Evaporators	Protech	0.01 GPM
7	Yes	Evaporators	Protech	8 PPM
8	Yes	Evaporators	Protech	0.01 GPM
9	Yes	Evaporators	Protech Magnazyme S	0.018 GPM
10	Yes	Evaporators	Protech Magnazyme S	0.018 GPM
11	Yes	Syrup Tank	Protech	0.03 GPM

N/A= Information not available.

Table 6. Weather conditions at the time of sampling during late grinding.

Weather Conditions	
Sunny	70-85F
Sunny	55-70F
Overcast	70-85F
N/A	N/A
Sunny	70-85F
Overcast	55-70F
Sunny	70-85F
Overcast	55-70F
Sunny	70-85F
Overcast	55-70F
Sunny	70-85F

N/A= Information not available.

Table 7. Starch concentrations from clarified juice and syrup during late grinding.

Sugar Factory	Starch (ppm/Brix)		
	Clarified Juice	**Syrup Last Body Evaporator**	**Syrup Tank Storage**
1	274	420	N/A
2	243	<100	<100
3	338	133	113
4	316	159	<100
5	256	102	243
6	688	185	352
7	437	387	<100
8	219	194	179
9	253	188	<100
10	156	158	120
11	241	406	178

N/A= Information not available.

Table 8. Clarified juice and syrup flow rates, and last body evaporator temperatures during late grinding.

Sugar Factory	Clarified Juice Flow Rate	Clarified Juice Brix	Syrup Flow Rate	Syrup Brix	Last Body Evaporator Temperature (F)
1	1230 GPM	15.7	442 GPM	56.1	150
2	2750 GPM	12.6	600 GPM	58.0	145
3	2560 GPM	14.6	500 GPM	62.1	140
4	N/A	N/A	N/A	N/A	N/A
5	1340	13.7	N/A	54.4	138
6	2750 GPM	13.6	110 Ton/h	61.0	150
7	2600 GPM	12.5	N/A	66.6	150
8	2700 GPM	14.1	N/A	56.0	150
9	510 Ton/h	14.1	121 Ton/h	59.0	160
10	N/A	14.7	N/A	63.7	138
11	1884 GPM	14.6	255 GPM	53.8	136

N/A= Information not available.

Table 9. Syrup flow rates and syrup tank storage capacity during late grinding.

Sugar Factory	Syrup Tank Capacity (CFT)	Syrup Storage Tank to Pans		
		Flow Rate	Temperature (F)	Brix
1	1600	442 GPM	140	56.1
2	1938	450 GPM	145	58.0
3	4682	500 GPM	140	57.0
4	N/A	N/A	N/A	N/A
5	4144	N/A	130	N/A
6	3200	N/A	N/A	61.6
7	8785	350 GPM	150	65.0
8	4100	2430 LbPM	116	56.2
9	3743	N/A	120	60.0
10	6190	N/A	135	65.0
11	3374	N/A	130	52.2

N/A= Information not available.

Table 10. Amylase concentrations and point of injection during late grinding.

Sugar Factory	Amylase Used	Application	Brand	Concentration
1	Yes	Syrup Tank	Termamil	1 PPM
2	Yes	Evaporator	Protech Magnazyme S	0.020 GPM
3	Yes	Evaporator	Protech	5 PPM
4	N/A	N/A	N/A	N/A
5	Yes	Evaporator	Protech	4 PPM
6	Yes	Evaporators	Protech	0.01 GPM
7	No	Evaporators	Protech	N/A
8	Yes	Evaporators	Protech	0.01 GPM
9	No	Evaporators	Protech Magnazyme S	N/A
10	Yes	Evaporators	Protech Magnazyme S	0.018 GPM
11	No	Syrup Tank	Protech	N/A

N/A= Not applicable.

SIZE MATTERS – CRYSTAL SIZE ANALYSIS FOR LOUISIANA SUGAR MILLS I

I. Tishechkina, F. Ehrenhauser and D. Aragon

INTRODUCTION

Audubon Sugar Institute has been analyzing sugar crystal size distributions for the Louisiana Sugar Industry since 2007. Currently more than 300 samples from nearly all mills are analyzed per year. In order for all mills to take full advantage of this valuable service, it makes sense to take a closer look at what crystal size is, the procedures we use and the potential benefits that these analyses could bring to the factories.

PRINCIPLE AND METHODOLOGY OF CRYSTAL SIZE ANALYSIS

We analyze sugar samples with the CILAS Particle Size Analyzer (Model 1180) (Figure 1), which measures particles in a range of 0.04-2,500.00 µm.

Figure 1: CILAS Particle Size Analyzer

The particle size measurement is based on the principle of light diffraction. To analyze submicron particles, CILAS uses laser diffraction. The observation of the diffraction pattern at finite distance is done through a lens (L) placed between the laser source and the detector. The diffraction patterns of particles having the same size converge at the same point with respect to their relative location with the lens (Figure 2). Based on the intensity of the diffracted laser light at a certain point in the detector the number of particles of this size can be evaluated. As the laser does not take into account orientation, a single particle size, the diameter of an equivalent sphere is obtained.

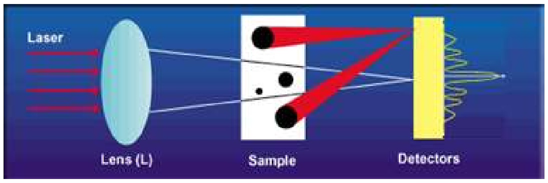


Figure 2: Principle of laser diffraction used in the PSA

Clearly a distribution of particle sizes is obtained. In order to characterize this crystal size distribution the following terminology (shown in Figure 3) is used:

Diameter at 10% and 90%:

This is the diameter value for which the cumulative result is 10% or 90%. Cumulative distribution function is a function that gives the probability where a random variable is less than or equal to the independent variable of the function. In our case it is a value at D (10%) and D (90%).D (10%) or D (90%) means that 10% or respectively 90% of all the measured crystals are below this crystal size. These two values give you therefore approximations of the smallest and the largest diameter contained in your sample.

Median Size:

Median size is the diameter value for which the cumulative results are 50%. This means that half of the particles in the sample are above the median size and half of the particles are below the median size.

Mean Diameter or Average Diameter:

Mean diameter (also called **Mean Size D [4,3]**) is the statistical mean value of particles' diameters contained in the sample.

Mode:

The most frequent value in a data set.

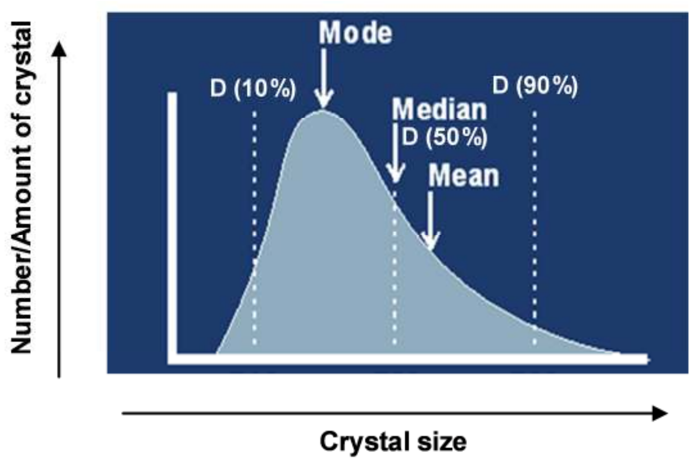


Figure 3: The main characteristics used in sugar crystal size distribution

Standard Deviation

The standard deviation (SD) shows how much variation or dispersion exists from the average. A low standard deviation indicates that the data points tend to be very close to the mean (also called expected value); a high standard deviation indicates that the data points are spread out over a large range of values.

Coefficient of Variation:

The coefficient of variation (CV) is defined as the ratio of the standard deviation of distribution to the Mean crystal size:

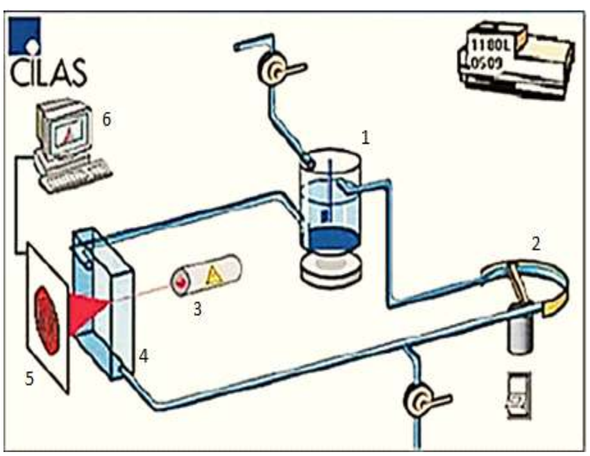
$$CV = \frac{SD}{MEAN} \quad \text{Equation (1)}$$

Histogram:

The histogram gives us the size distribution for different classes of sizes. It usually resembles a mountain-like shape in the graphs.

HOW DOES THE ANALYZER WORK?

First, a dispersed sample goes into a metal cup filled with the medium fluid and then circulates inside the closed system. It passes through the "glass" cell where the laser beam passes through the sample. A very sophisticated detector on the other side of the cell (Figure 4) captures results of the laser diffraction and computer software calculates the particle size distribution.



1. Metal res
   the samp
2. Pump
3. Laser so
4. "Glass" c
5. Detector

1. Metal cup with the sample
2. Pump
3. Laser source
4. "Glass" cell
5. Detector

Figure 4: The CILAS PSA scheme

Audubon Sugar Institute developed a unique procedure for the CILAS Particle Size Analyzer to analyze sugar samples. Several types of samples could be analyzed (Table1).

Table 1: Sample types and preferable sample conditions

SAMPLE TYPE	PREFERABLE CONDITION
Seed slurry	-
C grain	must be freshdelivered within 24 h
C massecuite	delivered within 24 h
C sugar	preferably not moist
B massecuite	preferably delivered within 24 h
B sugar	-
A sugar	-

Receiving the sample in preferable timeline end condition helps to minimize the result error.

We use two different mediums to disperse samples: Isopropanol and saturated sugar solution. Isopropanol works only for seed slurry. To disperse any other sample we use a saturated aqueous sugar solution. Each medium has its own challenges (Table 2).

Table 2: Mediums used for sample dispersion

MEDIUM FLUID	CHALLENGES
1. Isopropanol	price of isopropanol
2. Saturated aqueous sugar solution	saturation changes with lab and sample conditions (lab temperature, sample moisture) preparation time spoiling of the solution overtime hardening of the sample (C massecuite)

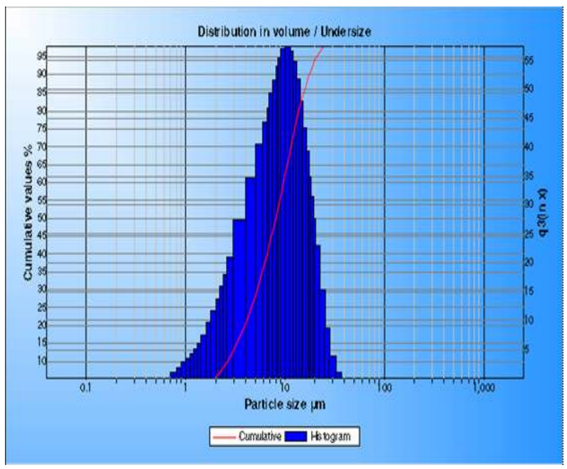
The biggest challenge is a change of saturation level in the solution, e.g. the solution becoming unsaturated or oversaturated. The first condition can result in dissolving some small crystals; the second can lead to the forming of small crystals. Both situations are undesirable, as they may shift the crystal size distribution and cause error in the results. We are trying to avoid these kinds of errors as much as possible and asking the mills personal to follow our recommendations for sample conditions as closely as possible; if followed, we will be able to provide the sugar mills with reliable results in a timely manner.

EXAMPLES OF CRYSTAL SIZE ANALYSIS

To illustrate the type of data sent to the sugar mills and how these results might benefit them, we present several examples of crystal size distribution that have been done at Audubon Sugar Institute in previous seasons.

a) **Seed Slurry**

We analyzed several Seed slurries during the 2013-2014 grinding season. Figure 5 shows a good crystal size distribution for one of the slurries. The crystal size distribution for the seeding material was tight, indicating a good start for boiling. The slurry was prepared from ball-milled, commercial white-sugar.



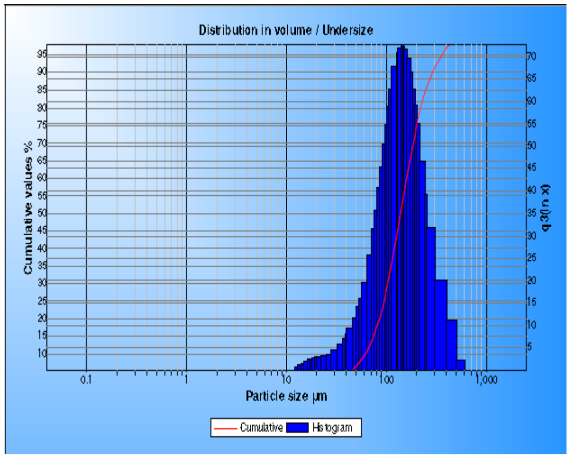
Concentration: 9
d (10%): 2.64 µm
d (50%): 8.07 µm
d (90%): 17.45 µm
D (4,3): 9.211 µm
D (3,2): 5.475 µm
Span: 1.833
Mode: 11.000 µm
Deviation: 5.899 µm

CV: 0.64 (calculated using Equation (1))

Figure 5: Seed slurry crystal size distribution

b) **C grain**

This season we included analysis of C grain samples. Our cooperation with the Lafourche Sugar Mill gave both sides a satisfying result. Figure 6 shows the best distribution for the mill during the season. In general, the target mean size should be around 150 microns and expectable CV should be close to 0.55. This year’s results show that C grain analyses can become routine for the mills.



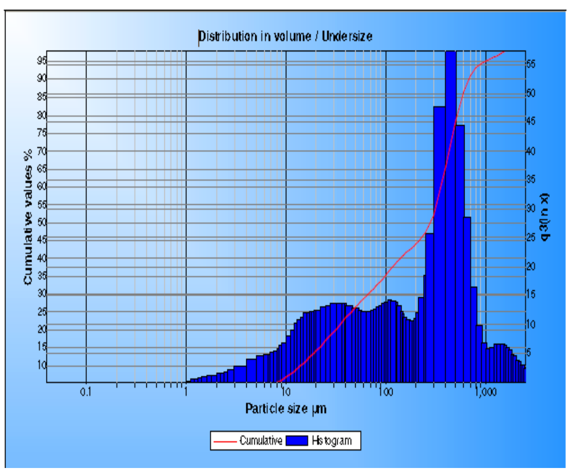
Concentration: 7
d (10%): 61.33 µm
d (50%): 138.65 µm
d (90%): 289.45 µm
D (4,3): 160.021 µm
D (3,2): 106.887 µm
Span: 1.654
Mode: 145.000 µm
Deviation: 95.346 µm

CV: 0.59 (calculated using Equation (1))

Figure 6: C grain crystal size distribution

c) **C Massecuite**

Figure 7 presented an abnormal crystal size distribution (CV 1.1). After analyzing the sample slide under microscope we saw the reason: the massecuite sample had a lot of false grains (Figure 8). Microscopic analyses revealed another issue with the massecuite: presence of elongated crystals, indicating high levels of dextran. This particular case illustrated the importance of another complimentary tool for the mill. A microscope at the factory may help to discover the presence of high levels of dextran even before the lab analysis would inform about it.



Concentration: 8
d (10%): 14.03 µm
d (50%): 279.04 µm
d (90%): 676.64 µm
D (4,3): 332.061 µm
D (3,2): 34.736 µm
Span: 2.375
Mode: 500.000 µm
Deviation: 366.258 µm

CV: 1.1 (calculated using Equation (1))

Figure 7: C Massecuite crystal size distribution

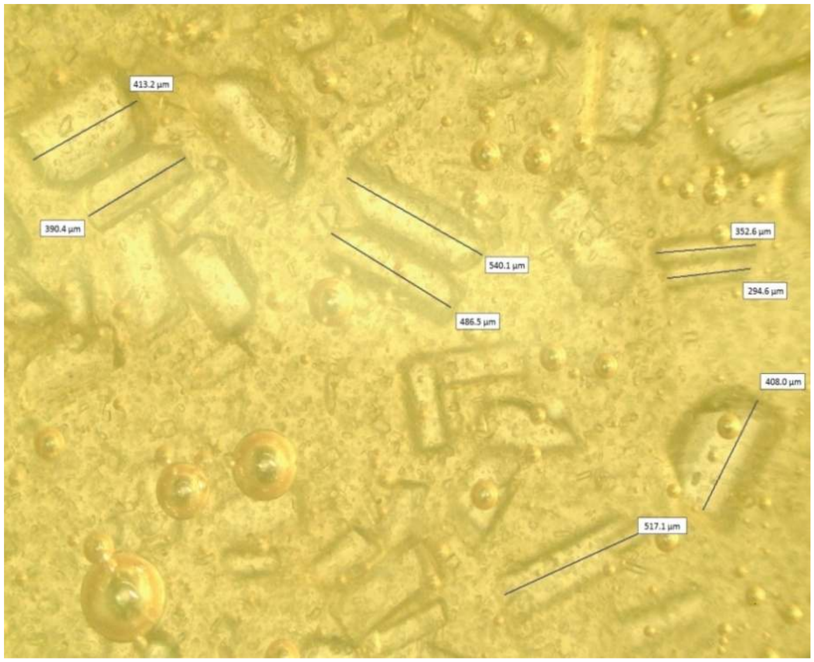
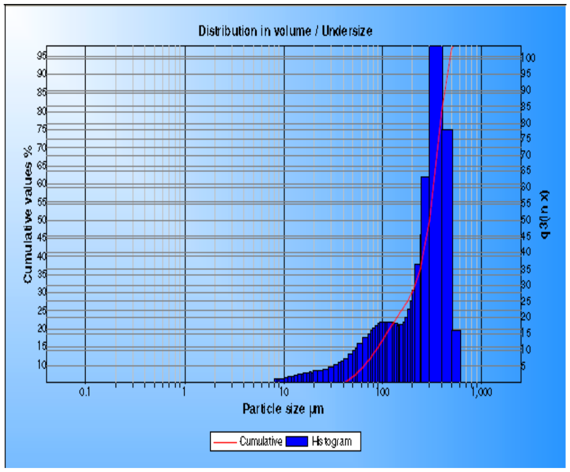


Figure 8: False grain in C Massecuite

d) **C Sugar**

C sugar crystal size distribution depends a lot on the C massecuite distribution. In the centrifugals, C massecuite becomes C sugar. Its distribution might differ from corresponding massecuite. In theory, smaller crystals should pass through the screen, and C sugar ought to have a better distribution than C massecuite. But inside the centrifugal, C sugar crystals can be overwashed, broken and so on, so the distribution might indicate problems in the C centrifugal machine. Figure 9 shows a distribution with some second generation crystals (small bump at 100 µm in Figure 9), which resulted in a higher CV. The second generation of crystals often develops from false grains.

r measurements of the last two seasons for some C sugars D 90% has shifted from 450 micron to 550 micron, while the range for small crystals stayed the same. This lead to a CV increase from 0.45-0.50 to 0.5-0.6 and up.



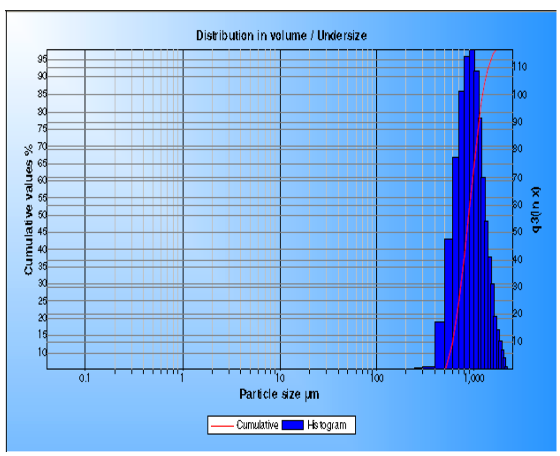
Concentration: 8
d (10%): 65.57 µm
d (50%): 300.50 µm
d (90%): 459.19 µm
D (4,3): 277.703µm
D (3,2): 136.240 µm
Span: 1.310
Mode: 400.000 µm
Deviation: 141.187 µm

CV: 0.51 (calculated using Equation (1))

Figure 9: C sugar crystal size distribution

e) **Raw Sugar**

The raw sugar we analyzed this season had a range of mean crystal sizes from 800 microns to 900 microns and CVs from 0.33 to 0.38. The bigger sugar was not always the best sugar. As always, a good sugar is sugar with a combination of a good mean crystal size and a good CV. For raw sugar, we would like to see a CV closer to 0.30. Figure 10 showed a good crystal size distribution with some room for improvement on the left side.



Concentration: 9
d (10%): 567.94 µm
d (50%): 886.92 µm
d (90%): 1348.62 µm
D (4,3): 929.225m
D (3,2): 835.729 µm
Span: 0.880
Mode: 1000.000 µm
Deviation: 305.681 µm

CV: 0.33 (calculated using Equation (1))

Figure 10: A sugar crystal size distribution

The examples presented here demonstrate that utilizing the knowledge about the crystal size distribution could help Louisiana sugar factories to:

- evaluate the quality of the seeding material
- control the C grain development and make correction to the seeding if necessary
- monitor C pan and crystallizer performance
- improve the C machines' and B machines' performance
- assess the final product

In conclusion, we would like to emphasize that crystal size analyses have already become a useful tool in optimizing the boiling house performance and minimizing the sugar loss for several sugar factories.

ACKNOWLEDGMENTS

We would like to thank the American Sugar Cane League for providing funding for this project, and all Louisiana's sugar mills for their continuous support.

SIZE MATTERS - CRYSTAL SIZE ANALYSIS FOR LOUISIANA SUGAR MILLS II 2013/14 Season

F. Ehrenhauser, I. Tishechkina and D. Aragon

During the 2013/14 season more than 300 different samples were obtained from Louisiana sugar mills and analyzed. The Cilas 1800 particle size analyzer provides the ability to assess the entire size spectrum of crystals ranging from 0.04 µm to 2,500 µm. Typical samples analyzed were seed slurry, A-,B-, and C- sugars and massecuites. The following presents a short summary giving an overview of some aspects of the obtained samples.

SEED SLURRY ANALYSIS

Proper seeding is essential for efficient crystallization. Three different slurry preparation methods were in use in Louisiana during the 2013/14 season. Slurry prepared directly from powdered sugar and isopropyl alcohol (IPA), slurry ball-milled from granular sugar and IPA and slurry ball-milled from powdered sugar and IPA. These different preparations have all the potential to yield suitable slurries depending on the need of the sugar factory.

Figure 1 shows the particle size histograms of several sampled slurries during the season. Figure 1a and 1b show powdered sugar slurries. Interestingly enough not every powdered sugar is identical. Powdered sugar is prepared by dry-milling granular sugar in a (hammer) mill and adding up to 5% (m/m) starch as anticaking agent. The label 10X denotes no specific size but only the times the sugar is sent through the mill. Usually, there exists an upper specification on the particle size, but there is no control limit on the small, finer particles. Figure 1a shows the slurry obtained from Domino's powdered 10X sugar as it is sold in a 1 lbs box, whereas Figure 1b shows also Domino's powdered sugar sold in 2 lbs bags. Clearly, the distribution is quite different. The boxed sugar has a substantial fraction of very small crystals, whereas the bagged sugar is exhibiting a more narrow size range, without the ultrafine crystals. Other powdered sugars (Western Sugar and Milliana) were also measured and their particle sizes exhibited different characteristics. From these samples we can conclude that the selection of the powdered sugar is relevant. Especially the sustainability of the particle size distribution during the season is challenging and as such the powdered sugar for the entire season should be bought from the same production lot. Storage of powdered sugar is also key, as Louisiana's climate causes caking and the sugar should therefore be stored in a dry place (e.g. closed drum).

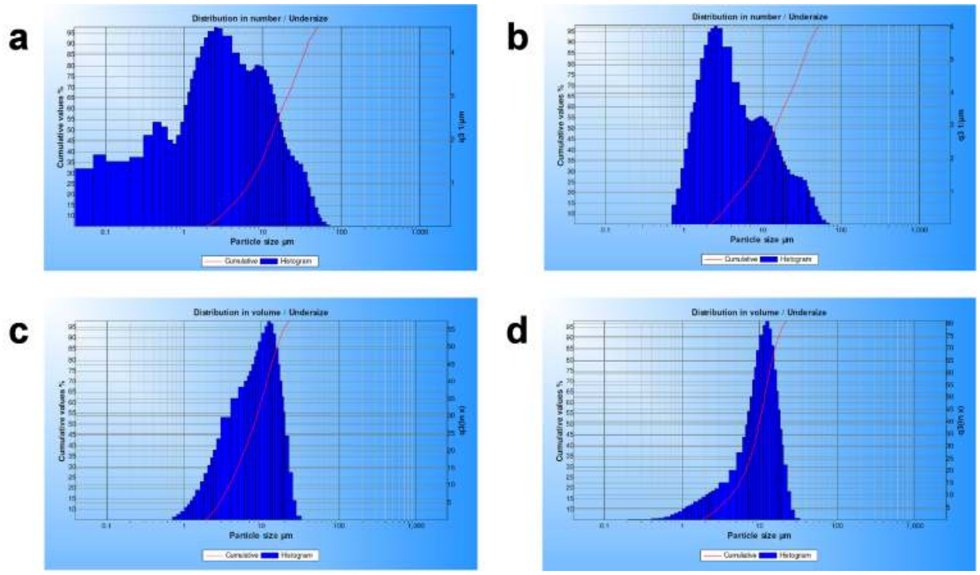


Figure 1. Crystal size distributions of (a) Domino Sugar powdered (10X) sugar sold in 1-lbs box, (b) Domino Sugar powdered (10X) sugar sold in 2-lbs bag, (c) ball-milled granulated sugar and (d) ball-milled powdered sugar

Figure 1c and Figure 1d show ball-milled seed slurries. Figure 2a shows a seed slurry which has been produced from granular sugar. The particle size is comparable to the one of the ball-milled powdered sugar. The choice of the seed slurry and preparation method is challenging and will vary from mill to mill. A narrow (small CV) particle size distribution is the key feature of any good slurry. The desired particle size range depends on the configuration of the pan floor and the graining procedure. As such for some mills, ball-milled fine slurry material is ideal, whereas others prefer powdered sugar. The most important feature for any slurry preparation is reproducibility throughout the season to provide consistency.

C-SUGAR ANALYSIS

Four mills have sent regularly samples (from 1 up to 49 samples per mill and season) of C-sugar from different C-sugar centrifugals for analysis during the last three seasons. Figure 2 shows an overview of the measured samples. Figure 2a shows the average median of the obtained C-sugars which centers around 250 µm. The error bars in the graph denote the standard deviation of the median of the measured sugars and are a measure for the reproducibility during the season. Mill III has the least deviation in the particle size of their C-sugar for the 2013/14 season. CVs for the C sugar can be found in Figure 2b. The values range between 0.5 and 0.65. A more narrow distribution and therefore lower CV might be desirable, but difficult to achieve. Generally, it is difficult to establish common trends for the shown values. Particle size rises for mills II and II, whereas for mill I and IV no trend can be determined. CV values are rising for mills II and IV. For mill I no clear trend in CV is recognizable and for mill III a decrease can be seen. Generally, larger median crystal sizes and lower CV's should improve recovery and purgability at the centrifugals.

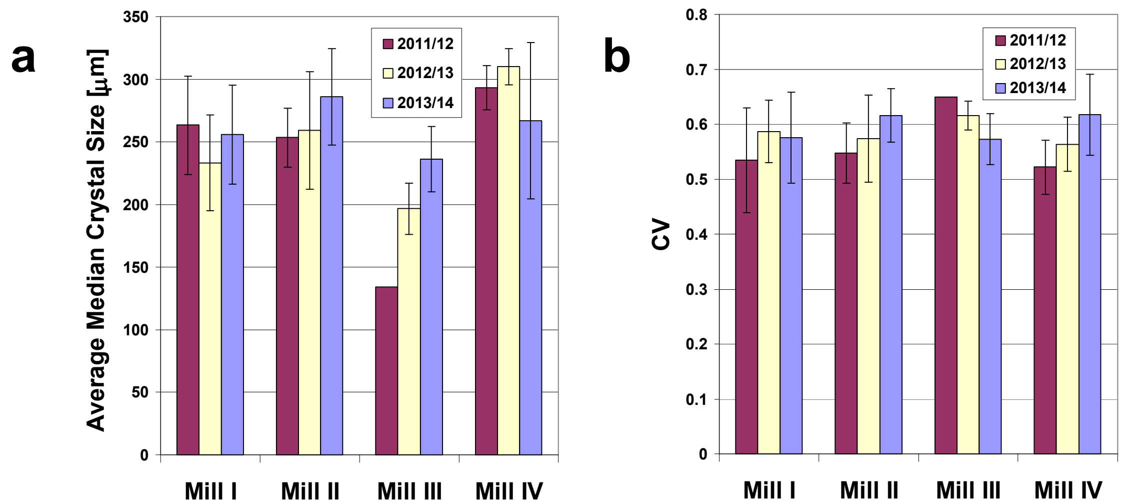


Figure 2. (a) Average median crystal size and (b) CV of C-sugar for mills I-IV during the 2011/14 seasons.

CENTRIFUGAL PERFORMANCE

A unique evaluation of the centrifugal performance was possible at one mill by evaluating nutsch purity and comparing with actual (centrifugal) molasses purity. As the fine nutsch filter captures virtually all crystals of the massecuite, while the centrifugal recovery is subject to losses, the difference between the purity obtained by nutsch filtration and the actual purity of the centrifugal molasses is a measure of the experienced sugar losses to molasses.

Figure 3 shows the purity difference plotted versus the median crystal size (D50%) and the 10-percentile crystal size (D10%). There is no clear trend between the purity difference and the particle size recognizable. Even though a trend is not visible, the absence of small purity differences for small particle size distributions (grey shaded area) indicates that by increasing crystal size the potential to minimize losses is given.

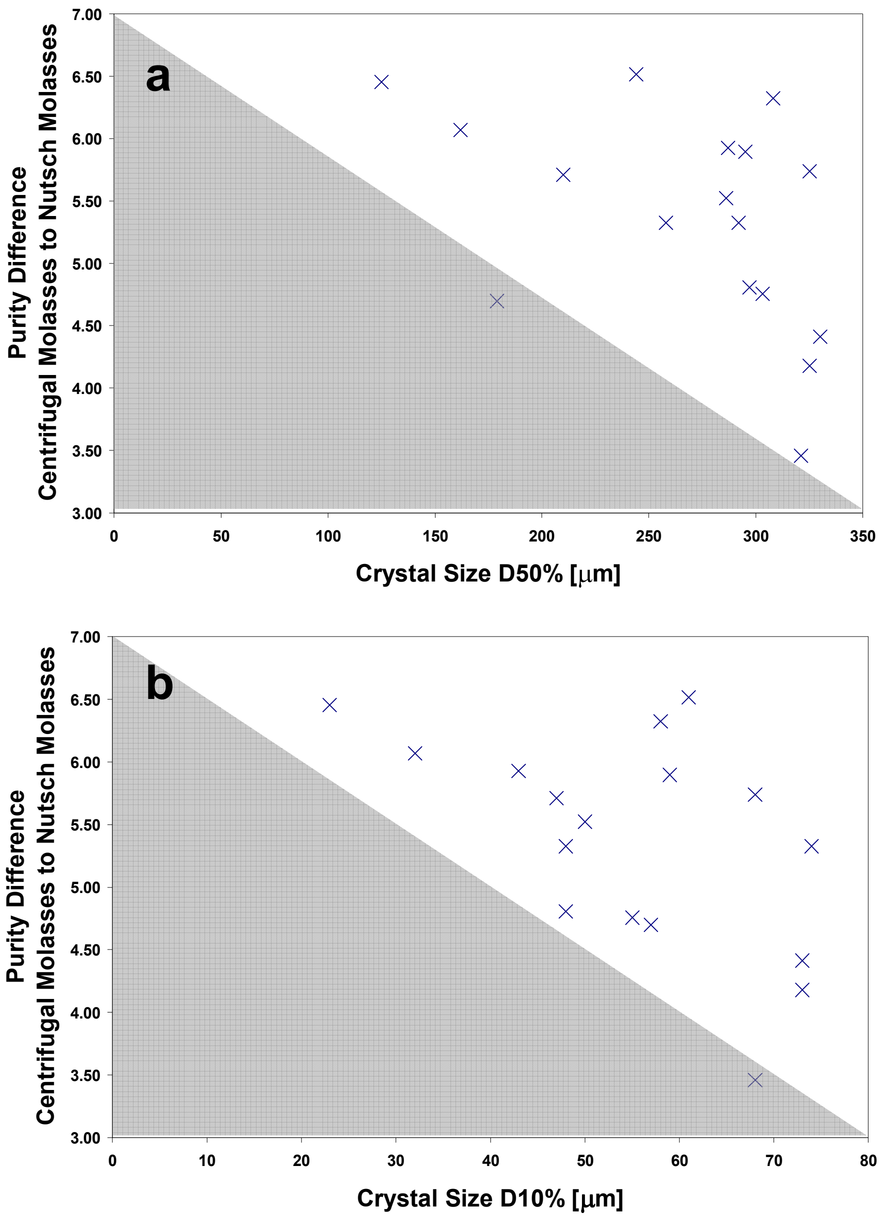


Figure 3. Purity difference between nutsch molasses and centrifugal molasses in dependence of (a) the median (D50%) and the 10-percentile (D10%)

For crystal size distributions with larger (300 µm) median sizes low purity differences can be achieved. However, not all large-sized C-sugars yielded low losses. These enhanced losses compared to the potential results are due to washing and breakage, which were not accounted for in this work.

SUMMARY

The measurement of the crystal size distribution enables the assessment of the crystallization process, the centrifugal operation and seed slurry preparation. Seed slurry should be prepared in consistent manner in order to provide a stable basis for further optimizations. C-sugar crystal size distributions in the four measured mills show no common trend; however, best potential centrifugal recovery can be achieved by aiming at larger crystal sizes and uniform (low) crystal size distribution.

ACKNOWLEDGEMENTS

The authors would like to thank the American Sugar Cane League and the Louisiana sugar mills for their continuing support and contributions for this research.

IMPROVEMENTS OF RAW SUGAR QUALITY USING DOUBLE PURGE OF C-MASSECUITES PERFORMANCE COMPARISON

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In 2013 Louisiana sugarcane crop season, three factories integrated double purge of C-magma to their three-boiling crystallization schemes, corroborating (like in 2012) color improvements on raw sugar up to 50% when comparing to a factory using single centrifugation (1,400 CU compared to 2,800 CU, at pH 8.5 using 1.2 µm GF filter). New BMA k3300 centrifuges operated at 1680 rpm and 1800 rpm were applied on second centrifugation. The double purge system was run controlling purity of the first magma around 82 and purity of the affined second magma between 90 and 92. The purity of the second wash molasses ranged between 63 and 67, and, it was sent to A or B molasses tanks. Like with a three boiling scheme, A and B massecuites were seeded with footings, prepared from the affined high purity magma, returning a high polarization (~ 99.2°Z) and low color raw sugar (1,000 – 2,000 CU). One factory reduced color approximately 50% while the other two factories reduced color about 36%. Double purge system and boiling house configuration and settings differed slightly between factories. Three years (2011 – 2013) of boiling house indices were compared for each factory. Indices of C-massecuite production (ft^3/ton cane), sugar lost on final molasses (pol final molasses % cane) and sugar recovery (pol sugar % cane) do not distinctly show a positive or negative effect for the integration of the double purge system. All three factories continuously ran the double purge of C-magma system, almost to the end of the 2013 season. Figure 1 shows pictures taken in 2012/2013.

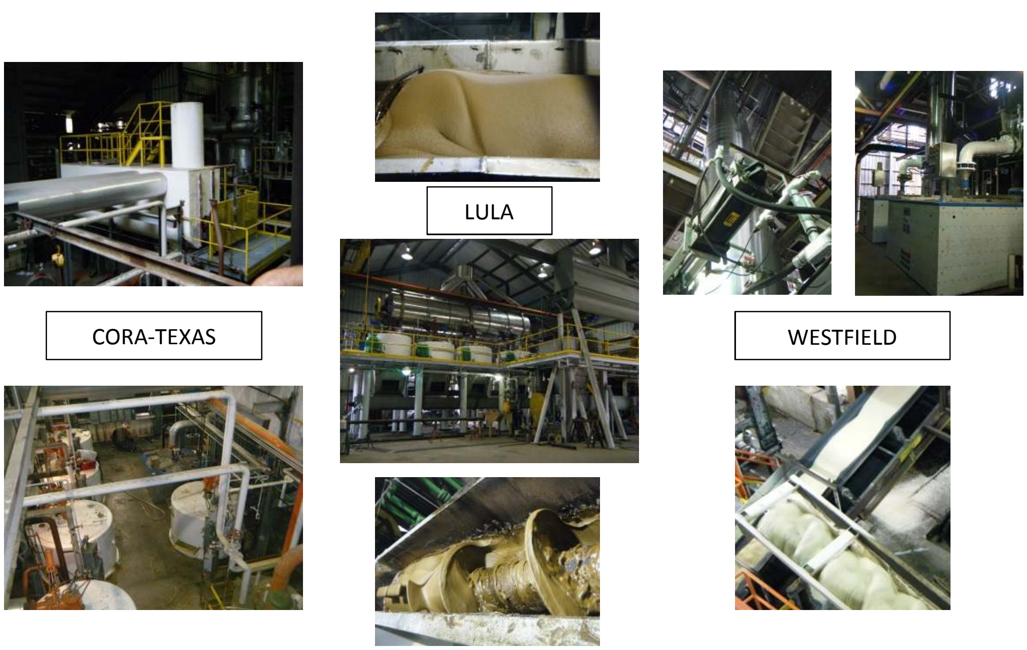

Figure 1. Double purge installations at Cora-Texas, Lula and Westfield

INTRODUCTION

Hugot (1986) described the double purge of C-magma using batch centrifuges and highlighted that grain size uniformity and mother liquor viscosity are conditions required for a good performance of the second centrifuges. In the first purge the C-sugar is separated from the molasses with "little or no water" (Meade and Chen 1977). Double purge molasses will be mixed with B molasses or it will be stored in an independent tank, but it is not recommended to recirculate the second wash to an upper crystallization stage. The first C-sugar is discharged into a mixer where it is converted in magma by mingling the sugar with higher purity molasses, clarified juice or water. Diluted B molasses (70 °Brix) at 70 °C (158 °F) is suggested for the preparation of the first magma. The resulting first magma is again centrifuged washing with water and steam. The second C sugar is mingled with syrup (30% syrup for 70% C sugar) and sent to the pan floor. First magma purity values from 80 – 85 and Second magma purity values from 88 – 92 are recommended for the production of high polarization raw sugar (Hugot 1986). Milner and Houstonk (1997) recommends to use for the first purge a standard centrifuge (30° basket) equipped with magma mixing and with open bottom, operating at no more than 2000 G (G-factor) to reduce crystal breakage and for the second purge, centrifuges (25° basket) can operate at 1200 to 1600 G. Approximately one to three is the capacity ratio between second purge to first purge centrifuges, because of the higher capacity of the second centrifugation stage (easier task). There exist centrifuges built with two stages (double centrifuges) but two individual centrifuges offer more flexibility to change capacity at each stage to optimize results and the maintenance of each separation stage (Milner and Houstonk 1997). The mixing and scrubbing of crystals before the second centrifugation releases the molasses layer attached to the crystal (Meade and Chen 1977; Milner and Houstonk 1997). The double purge system changes such first magma purity, magma preparation, wash molasses purity and second magma purity all effect non-sugars recirculation, raw sugar quality and capacity of low grade pans (Hugot 1986; Perez, Moreno et al. 1997). It was found in Hawaii that if the purity of the first magma drops from 75 to 65, the required capacity of C pans increases on 30% (Hugot 1986). Using second purge wash molasses instead of water to prepare the first magma requires more capacity for the whole double purge system (magma preparation and double purge centrifuge), and produces a second magma with higher color and higher turbidity (Perez, Moreno et al. 1997). Milner and Houstonk (1997) state that the use of cold water reduces the color on affined raw sugar. Makina (2000) states that on the case of sugar beet production, a single purge can reduce color on 94 – 96 while with double purge the color reduction is about 96 – 98% for low grade massecuites. Perez, Moreno et al. (1997) compared the use of double purge molasses or water on magma preparation, for the production of plantation white sugar with sulfitation and a VHP boiling scheme (B magma as footing for A strike and melting of C sugar). It was found that the quality of white sugar in terms of color and turbidity were significant different when comparing first magma preparation with 2nd molasses or with water (Table 1). A system producing ~19 m^3 / hour (667 ft^3 / hour) of C-massecuite, requires 18 m^3 (634 ft^3) mixer; three continuous centrifuges (one standby) with capacity of 6.8 m^3 / hour-centrifuge (240 ft^3 / hour-centrifuge); two magma

pumps (one standby) with capacity of 16.8 m^3 / hour-pump (594 ft^3 / hour-pump, 74 gpm) and; two molasses pumps (one standby) with capacity of 34.2 m^3 / hour-pump (594 ft^3 / hour-pump, 150 gpm) (Perez, Moreno et al. 1997).

Table 1. Double purge parameters and sugar quality preparing magma with 2nd molasses and with water (Perez, Moreno et al. 1997)

Year	**1 (magma – using 2nd molasses)**				**2 (magma – using water)**			
Parameter	Brix	Purity	Color	Turbidity	Brix	Purity	Color	Turbidity
1st C Sugar	92.8	77.6	26036	9315	92.9	79.4	22138	7392
2nd C Sugar	92.2	85.9	14252	5723	92.5	89.6	9782	4305
2nd Molasses	82.8	61.6						
White Sugar			161	69			141	53

The double purge technology can be implemented for factories operating with three-boiling system to improve raw, white and refined sugar quality. The double centrifugation system to improve raw sugar quality lost popularity around 1960. Double purge of C-magma may be important for some raw sugar factories in Louisiana where it is necessary to improve raw sugar quality at a minimum cost.

RESULTS

In 2013 Lula Sugar Factory, Westfield Sugar Factory and Cora-Texas Mfg. Co., Inc. implemented the double purge system, giving an opportunity to evaluate the performance of the system for different system configurations and boiling house operational parameters. Table 2 shows some of the differences in double purge in the three factories. Cora-Texas and Lula feed the prepared first magma from a tank equipped with mixer; Westfield installed a cylindrical vertical tank without a mixer. Cora-Texas and Lula installed a molasses preparation tank controlling level and Brix; Westfield added water in-line to the wash molasses, directing this stream to the A or B molasses tanks at the centrifuge floor. Primary wash molasses were sent to A molasses at Lula and Westfield and, they were sent to B molasses at Cora-Texas. Lula increased the rpm for the second purge from 1475 to 1684 rpm for the 2013 season.

Table 2. Main components of the double purge system on Cora-Texas, Lula and Westfield

	Cora-Texas	Lula	Westfield
Magma Preparation	Water Heat & Mix 3 rpm	Water Mix 7.8 rpm	Water No Mix
Centrifuge	1,800 rpm	1,684 rpm	1,684 rpm
Molasses Preparation	Water Level & Brix	Water Level & Brix	N.A.
Return molasses	B molasses	A molasses	A molasses

Simulation of capacity requirements at different syrup purities for each factory were produced by applying the estimated best double purge parameters found in 2012. Figure 2 illustrates the results obtained from boiling house models on Sugars^{TM} and from calculation of pans capacity requirements for each factory (Birkett Report) comparing single purge (3B) and double purge, blending double purge molasses with B molasses (DPB). Maximum grinding rates (tons cane per day, TCD) for each factory were used for the simulation (critical condition): 12,000 TCD for Lula, 13,500 TCD for Westfield and 18,000 TCD for Cora-Texas. These factories had enough installed capacity, but the low grade pan capacity was critical for Lula at low syrup purities. During the 2013 crop season Lula ground above 12,000 TCD cane at least for 10 days, Westfield and Cora-Texas ground cane above these critical values some few days. At grinding rates above the critical values the capacity of the boiling house is compromised, affecting sugar yields per strike and increasing sugar losses.

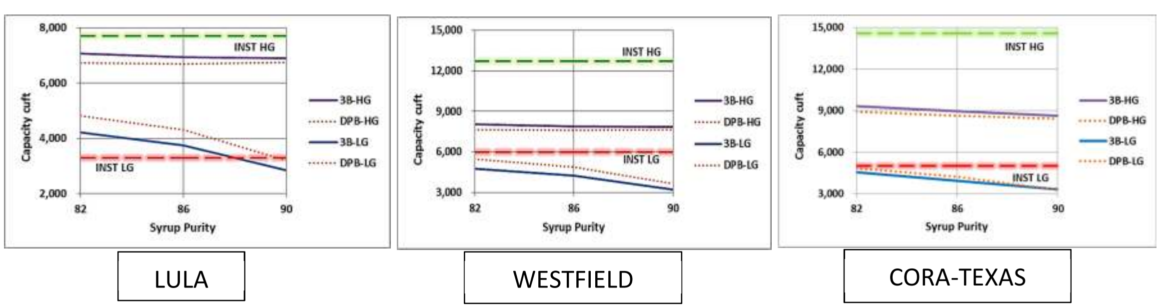

Figure 2. High grade (HG) and low grade (LG) pans required and installed (INST) capacity at each factory (Lula 12,000 TCD, Westfield 13,500 TCD and Cora-Texas 18,000 TCD) for different syrup purity (82, 86, 90), simulated with Sugars^{TM} for a three-boiling scheme with single purge and with double purge of C-magma

Figure 3 shows daily variations of purity for the double purge system in each factory. Lula operated the system controlling second magma purity around 92; the purity of the first magma was controlled around 82 to avoid problem for excess of B molasses and; the purity of the double purge molasses was around 65. Lula and Westfield controlled the second magma purity around 90 and first magma around 82.

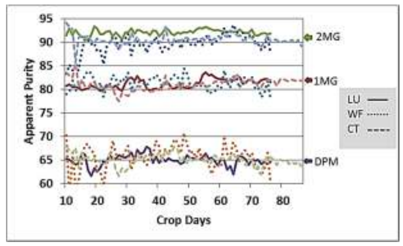

Figure 3. Daily average variation (daily factory report, 2013) for purities of double purge magma (2MG), first magma (1MG) and double purge molasses (DPM) on Lula (LU), Westfield (WF) and Cora-Texas (CT).

Figure 4 shows the daily variation and composite weekly variations of whole color (1.2 µm, pH 8.5) for the factories with double purge of C-magma. Adjustments on performance of the double purge system, at Cora-Texas, on the middle of the season helped in reducing color on raw sugar. Whole color was on average 1,200 CU at Lula, 1,500 CU at Westfield and 1,600 CU at Cora-Texas after parameters adjustment (first magma Brix and purity).

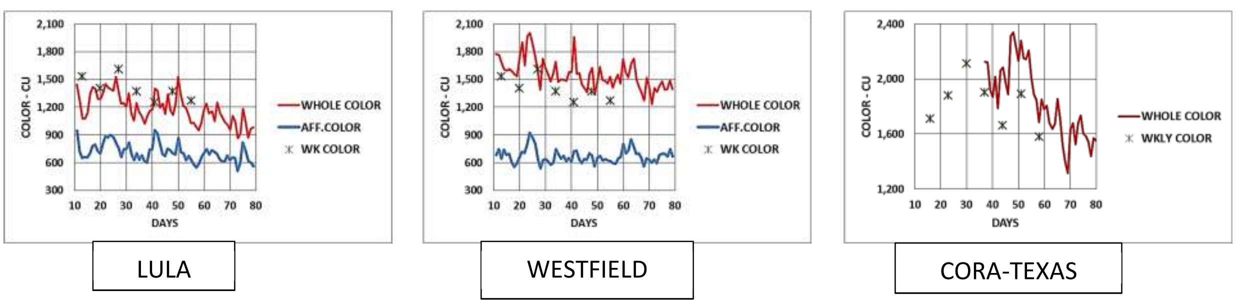

Figure 4. Daily and weekly, whole and affined color analysis at Lula, Westfield and Cora-Texas during 2013 sugarcane crop season

The improvement in whole color of raw sugar was also evaluated comparing weekly composite samples of syrup and raw sugar for the three factories with double purge system, and an additional factory (Alma Plantation) operating with single purge of C magma. Figure 5 shows the average results obtained from the analysis of the weekly composite samples. Compared to Alma, it can be seen that Lula improved raw sugar color and conductivity ash on 50% and 62% respectively while the improvement for Cora-Texas and Westfield for both parameters were 36% and 41%.

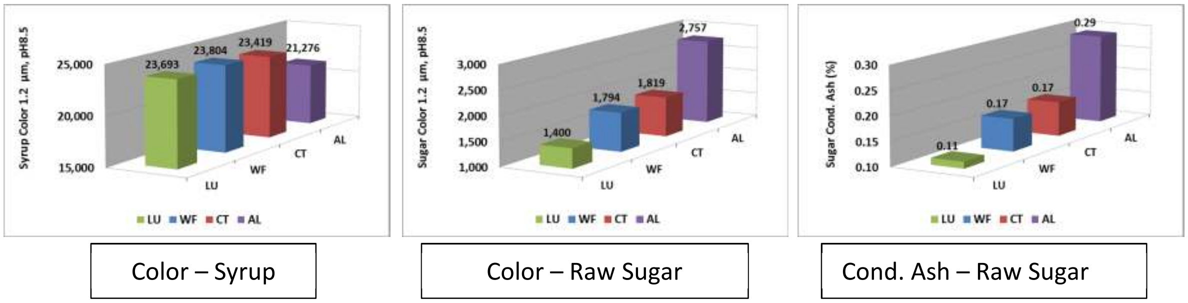

Figure 5. Means of 9 weekly composite analysis of syrup color (pH 8.5, 1.2 µm, CU), raw sugar color (pH 8.5, 1.2 µm, CU) and raw sugar conductivity ash (%) for factories with double purge of C-magma (Lula-LU, Westfield-WF and Cora-Texas-CT) compared to a factory with single purge (Alma-AL)

Figure 6 compared some performance parameters for three crop seasons (2011 – 2013) for each factory (Factory Reports). On average (2012 and 2013 with double purge) Lula processed 5% more C-massecuite than in 2011. On the other hand, Westfield processed 5% less and Cora-Texas 1% more comparing 2012 and 2013. Increments or reduction cannot be attributable to the double purge system. Same conclusion is reached with respect to boiling house parameters like sugar recovered % pol in juice (Lula 0.9% higher, Westfield 0.6% lower and Cora 1.2% lower), final molasses production per ton of cane (Lula 7% lower, Westfield 1% lower and Cora 3% lower) and sugar lost to final molasses (Lula 13% less, Westfield 9% more and Cora 1% more). Operational factors and boiling house equipment changes plays also an important role on the behavior of all of these parameters. For instance, boiling house can also be affected by water input with syrup and with the dilution of molasses. Comparing 2012 and 2013 Lula and Westfield had extra water input with syrup of approximately 16 & 10 % while Cora-Texas had a brix reduction (more dilution water) on A and B molasses of 4 & 3%.

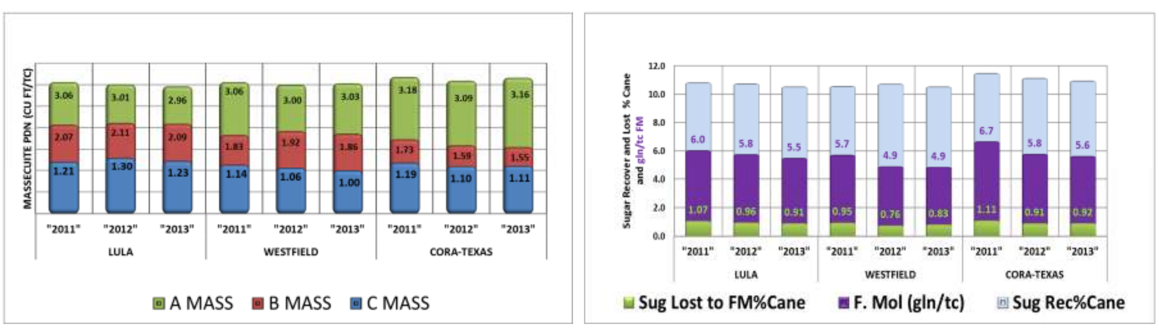

Figure 6. Lula, Westfield and Cora-Texas massecuite production (ft^3/tc) and boiling house performance parameters (sugar recovered, sugar lost and molasses production) comparison for three years.

CONCLUSIONS

Since operational conditions may affect also the performance of the boiling house it cannot be deduced that double purge altered either positively or negatively the C-massecuite production and the recovery (or lost) of sugar of any of the mills. Cora-Texas, Lula and Westfield operated the double purge system continuously from the second week to almost the last week of the 2013 season achieving significant results on reduction of whole color (36% to 50%) and conductivity ash (41% to 62%) of raw sugar.

ACKNOWLEDGEMENTS

Thanks to the American Sugar Cane League for the funding and to each one of the managers, engineers and lab personnel of Cora-Texas, Lula and Westfield. Special mention to:
M. Daigle, G. Carline, L. McAdams, E. Garcia, R. Whitelaw, T. Newchurch and J. Grisaffe- Lula-Westfield, LLC
A. Monge, Scott Kessler, C. Schudmak and J. Sanchez- Cora-Texas Manufacturing Co.
B. Montes – Alma Plantation LLC
V. Kochergin – Amalgamated Research LLC
C. Verret and I. Tishechkina – Audubon Sugar Institute

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THE MOLASSES AND JUICE SURVEY

C. Verret, D. Dorman and S. Lu

INTRODUCTION

Since 2001, the Audubon Sugar Institute has analyzed the molasses provided weekly by each of the 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 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) via 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 massecuite (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.

MATERIALS AND METHODS

Composite samples of final molasses (seven day) were sent to us weekly from each of the 11 mills in Louisiana. The 2013 survey season stretched from 10-06-13 until 01-12-14. A total of 207 samples were analyzed in duplicate for the 2013 season. Including standards, this totaled 579 samples for 2013. 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 clarified using Octapol^{TM} (Baddley Chemical) so that we can obtain a pol/sucrose ratio.

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 25-28 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/DISCUSSION

The 2013 season operated for 15 weeks. The 2013 season maximum TPD weekly average was 10.7 and the minimum was 6.2. Throughout the season, the TPDs were up and down, a possible reason for this was the weather conditions. The industry average TPD for 2013 was 8.0. (Figure 1)

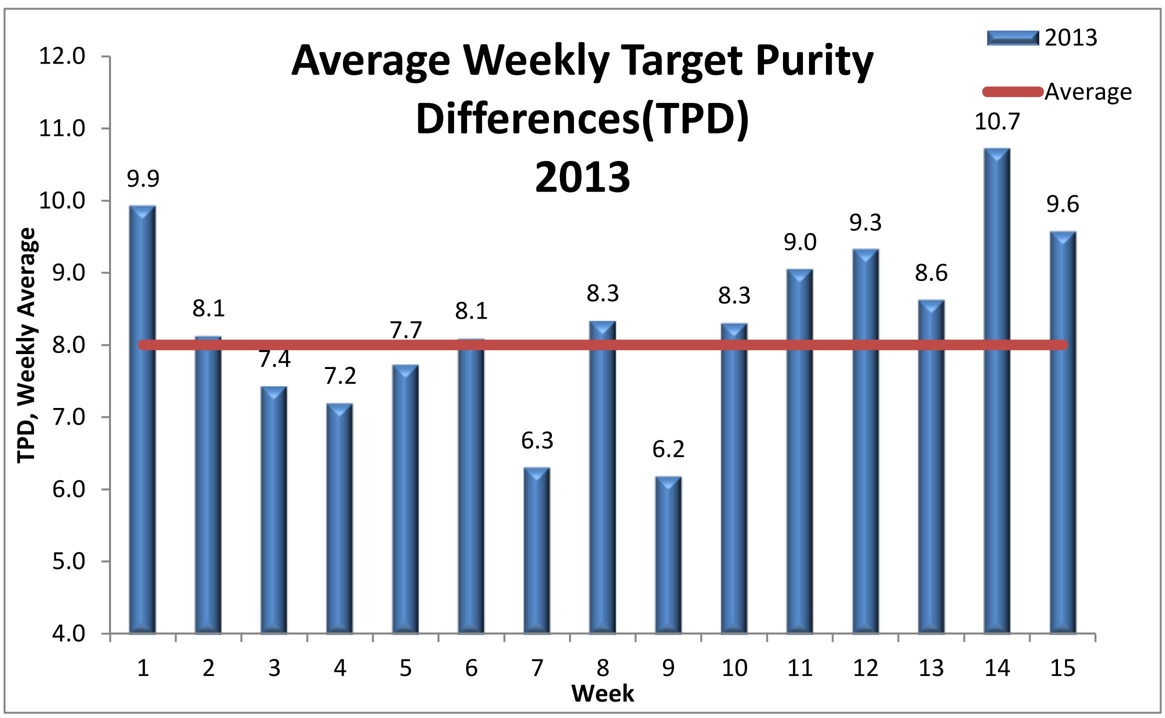

Fig. 1 – 2013 Average Weekly Target Purity Differences

The conductivity ash component for the 2013 season started at the minimum value of 13.1. As the season continued the ash increased to the maximum value of 16.0 and then decreased towards the end of the season. The conductivity ash average was 15.0. (Figure 2)

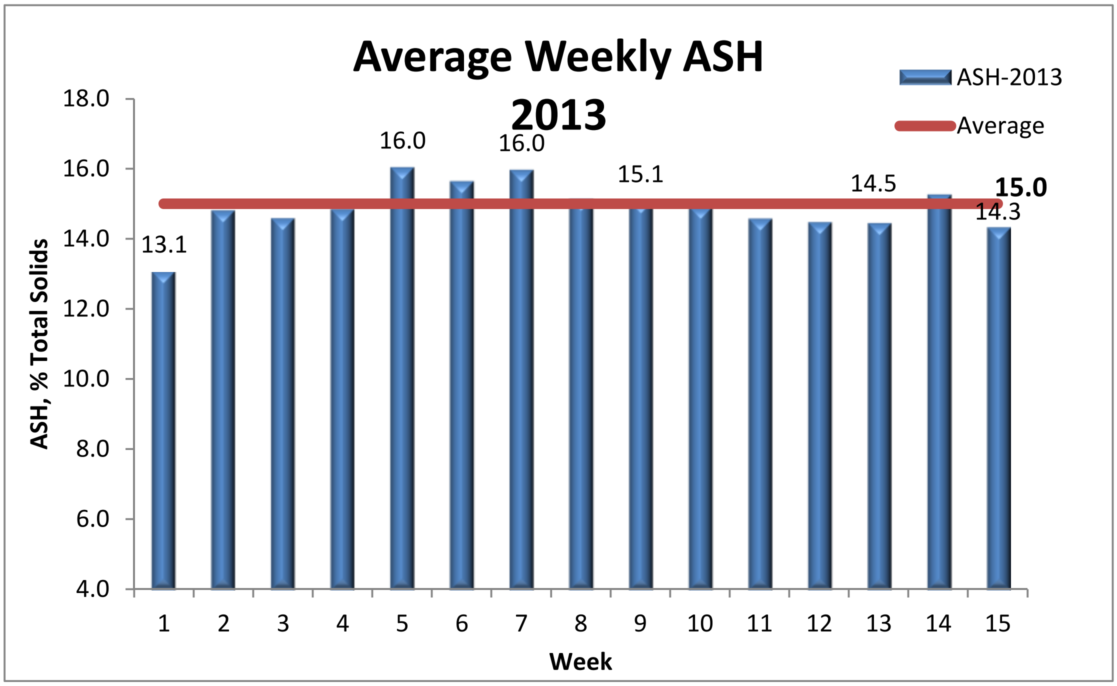

Fig. 2 – 2013 Average Weekly Conductivity Ash

In comparing TPD to ash, it demonstrated that as the conductivity ash increased or decreased the TPD decreased or increased. Lower ash leads to lower target purities, which will lead to higher TPDs.
(Figures 3 & 4)

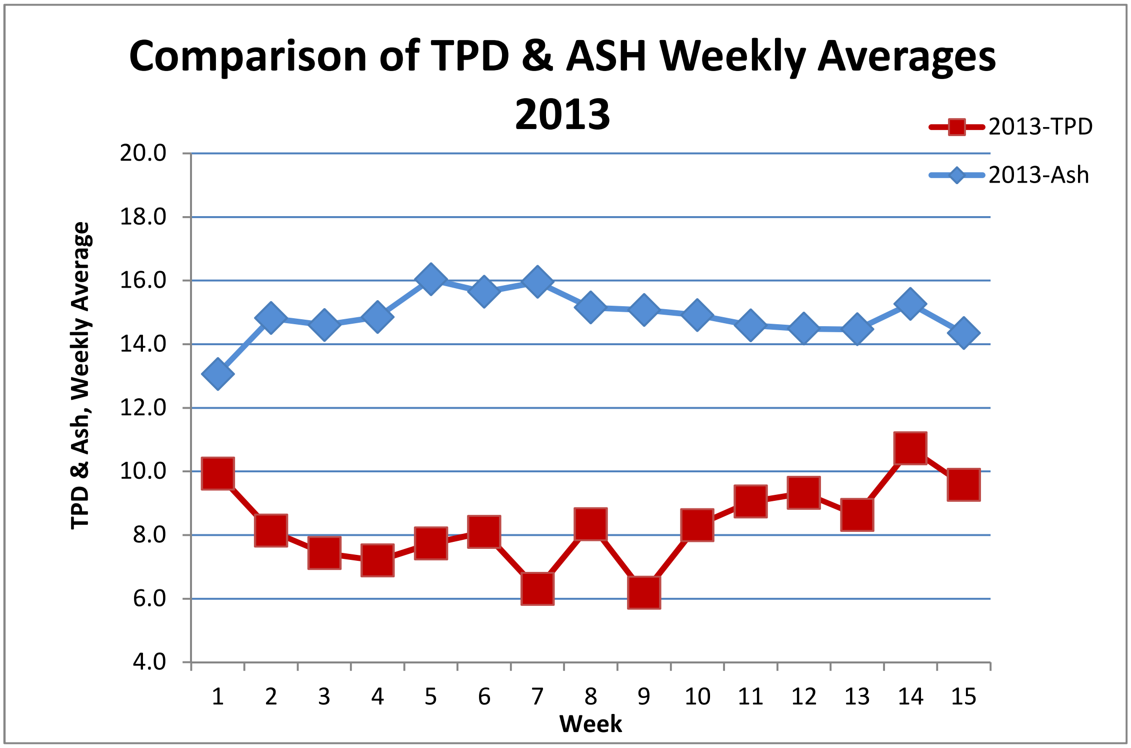

Fig. 3 – 2013 TPD & Ash comparison

In general, there has been a significant downward trend, relative to time, in the amount of reducing sugar in final molasses. In the beginning of the 2013 season, the reducing sugar was showing the decreasing trend. The maximum was 15.3; usually the minimum occurs towards the end of the season. However, this season the minimum of 11.2 occurred in the middle and slightly increased throughout the rest of the season. (Figure 4)

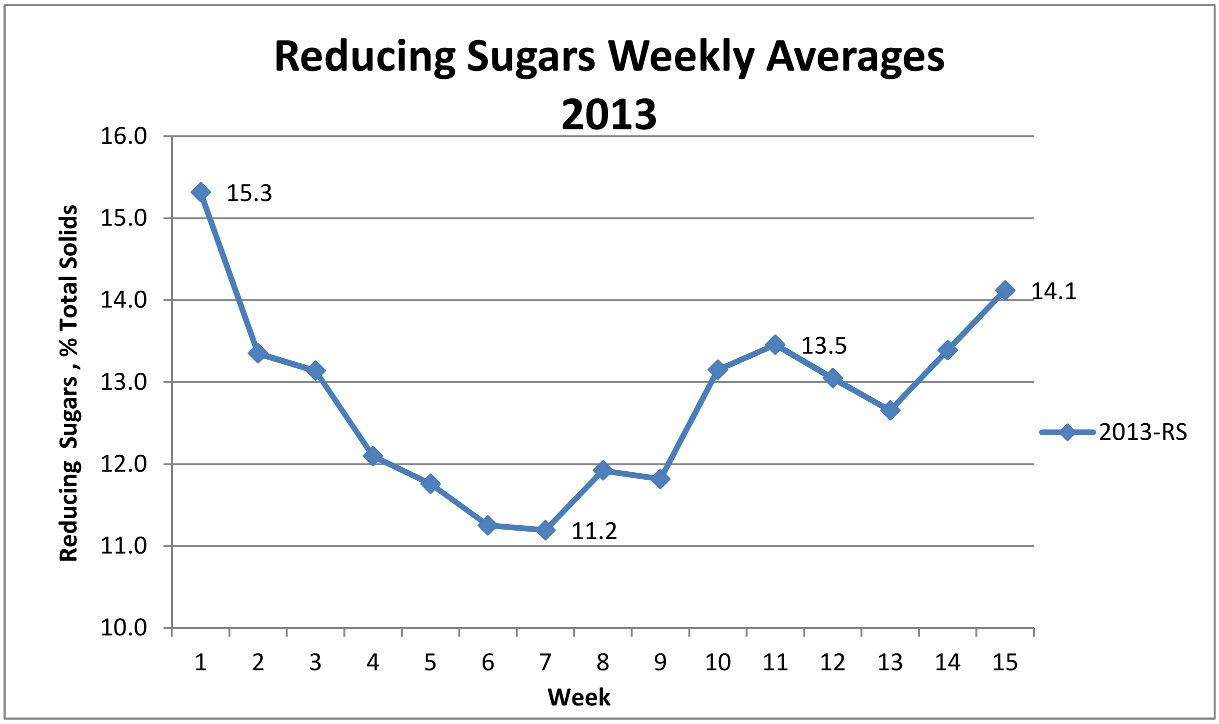

Fig. 4 –2013 Reducing Sugars Weekly Averages

Comparing the results from the 2013 season to the results from the 2011and 2012 seasons showed the downward trend of the yearly average TPD. This is demonstrated in **Figure 5.** The 2013 season maximum TPD was 10.1. The minimum TPD for 2013 was 5.8. (Table 1) The comparison of the weekly averages of TPD and the reducing sugars shows increasing towards the end of the season. (Figures 6 & 7)

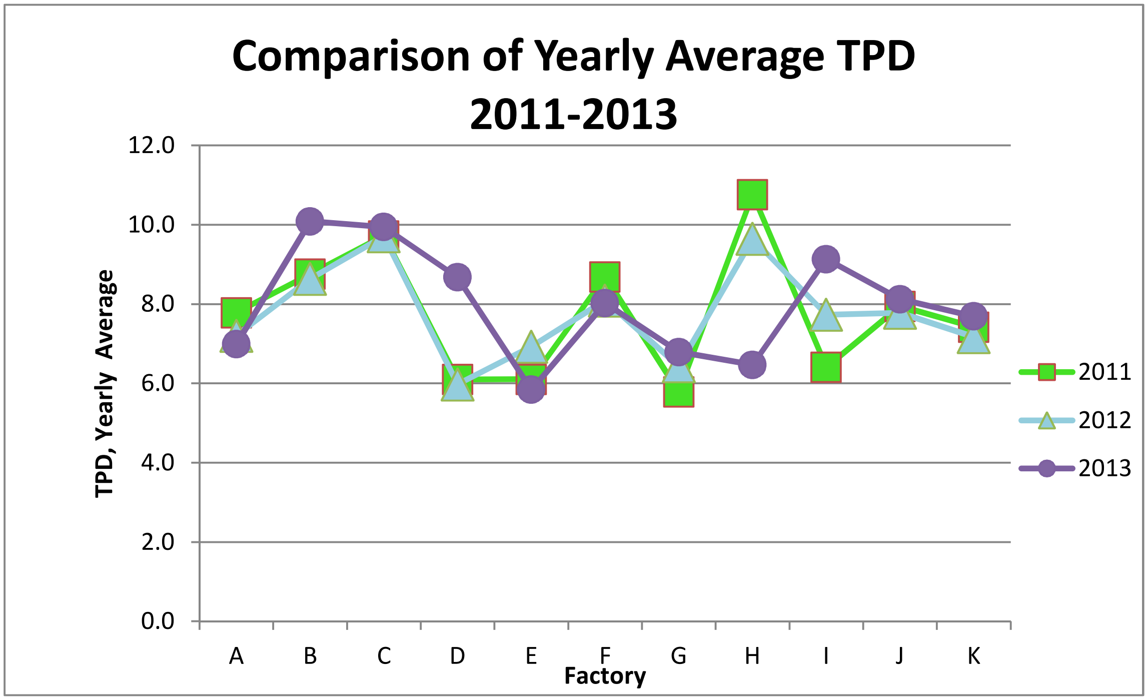

Fig. 5 – Comparison of Yearly Averages TPD 2011-2013

TPD Data Summary for 2011-2013			
Year	**TPD Minimum**	**TPD Maximum**	**TPD Average**
2011	5.8	10.8	7.8
2012	6.0	9.7	7.8
2013	5.8	10.1	8.0

Table 1 – Summary of Yearly TPD 2011-2013

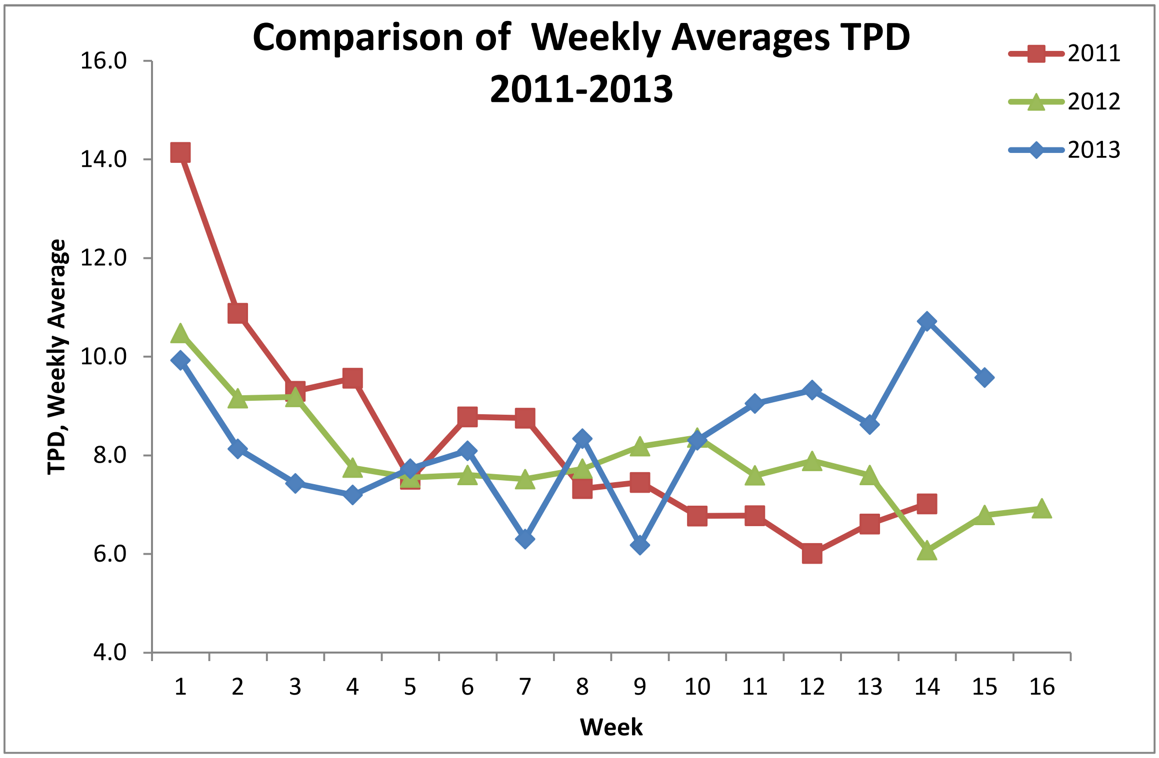

Fig. 6 – Comparison of Weekly Averages TPD 2011-2013

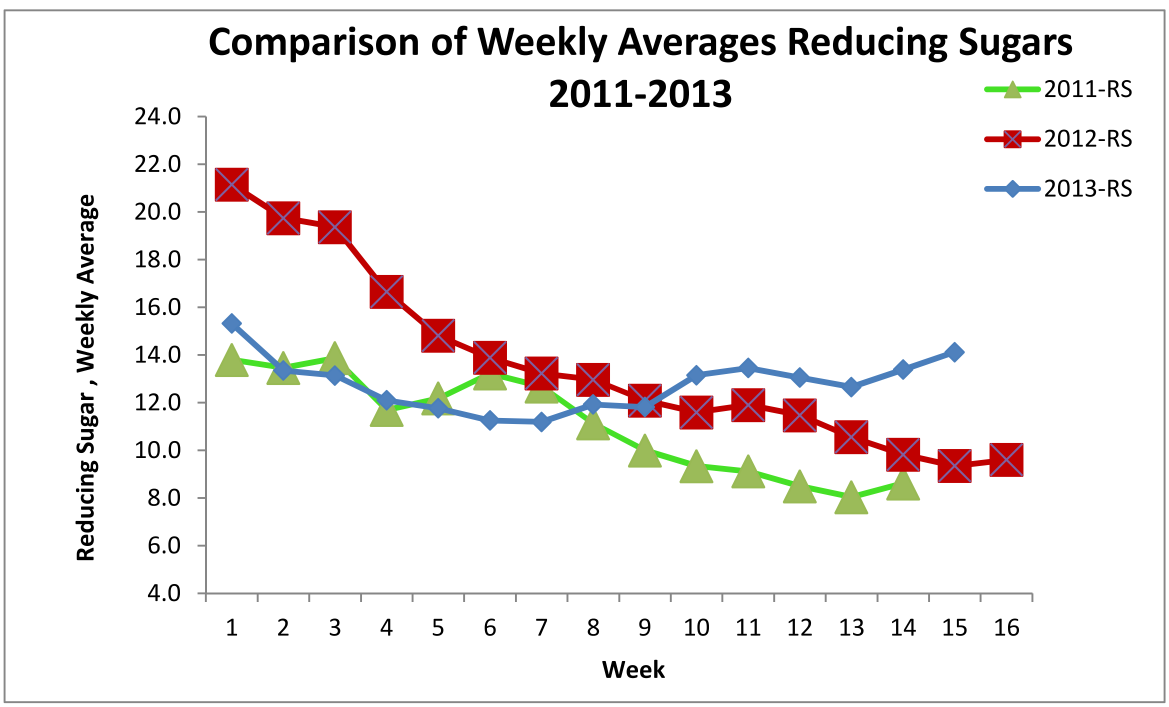

Fig. 7 – Comparison of Weekly Averages Reducing Sugars 2011-2013

CONCLUSIONS

The seasonal average TPD was 8.0 for the 2013 season. The ash increased for the 2013 season to 15.0%. The reducing sugars decreased for the 2013 season to 12.5% from 13.1% from the previous season.

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.

The mills are conscious of their TPD and are continuing to improve which is an encouraging trend.

Historical data suggests that a TPD of five remains a rational and encouraging objective for the 2013 grinding season.

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APPLICATIONS OF PROCESS MODELING AND SIMULATION IN LOUISIANA'S SUGAR MILLS

Daira Aragon

INTRODUCTION

Factory models can be used in combination with plant data during the grinding season or in the downtime to assist plant managers and engineers to develop mechanisms to maintain and/or improve process operations. There are an increasing number of applications of process models; however, in the scope of this paper only simulation applications considered promising and practicable in the sugar industry will be addressed: balancing sugar, steam and energy consumption, evaluating changes to equipment and plant layout and evaluation of changes in operating conditions.

MODELING AND SIMULATION

Simply said, a model is a representation of the reality. Models are either physical, such as equipment made to scale (i.e. pilot plants), or mathematical. Mathematical process models constitute the core for simulation, control and optimization; hence, developing a valid and accurate model is the key for the successful implementation of these methodologies not only in the sugar industry but also in any other type of process. It is also important to understand that models are not 100% accurate, as they are often simplified, especially for complex processes where the phenomena involved are not known exactly. In a process industry, it is the plant personnel who know the process best. Therefore, the modeler must work hand-in-hand with them to arrive at a model that represents the process behavior correctly and provides meaningful answers to the questions given by its purpose.

Steps involved in development of a mathematical process model are presented in figure 1. In first place, an objective or purpose for the model is define; this objective dictates which system to model (individual equipment, section of the plant, or entire plant) and simplifications allowed. Then, the model equations are written using available knowledge, both from plant engineers and operators and from theory. Plant data is later used to validate and refine the model, so that it produces accurate and reliable results. Finally, simulations are performed to give answer to the questions posed by the stated purpose.

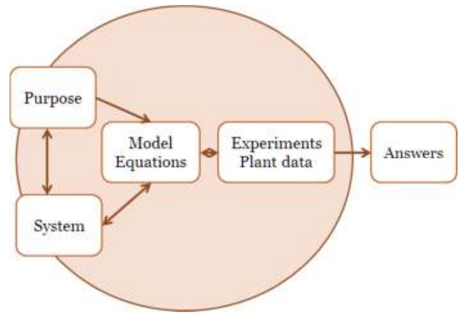

Figure 1. Steps in development of mathematical process models

Simulation can be defined as the use of a process model to obtain a response given certain input values; for example, obtaining the quantities of juice, bagasse, mud, filter cake and sugar produced by a sugar mill for an input cane with a pol of 14 and 12% fiber. The final result from the simulation can be a single value, a set of values over time or an animation. Usually, results for material, steam and energy balances are single values for each variable (steam consumed by evaporators and pans, sugar lost in bagasse and filter cake, raw sugar produced, among others), results for process control are values over a period of time, and results for designing equipment through fluid dynamics are animations.
There is a number of software available for modeling and simulation. Some of them, known as equation-oriented software, require writing the model equations directly. Matlab is an example of this type of software. Others programs, known as flowsheeting software, such as Sugars™ and ASPEN, use predefined equipment where only certain parameters are changed. While both types of software can produce good models and results, equation-oriented programs require deeper knowledge on the fundamental phenomena driving the processes and on programming logic. Figure 2 shows a snapshot of the user interface of Matlab, ASPEN plus and Sugars™.

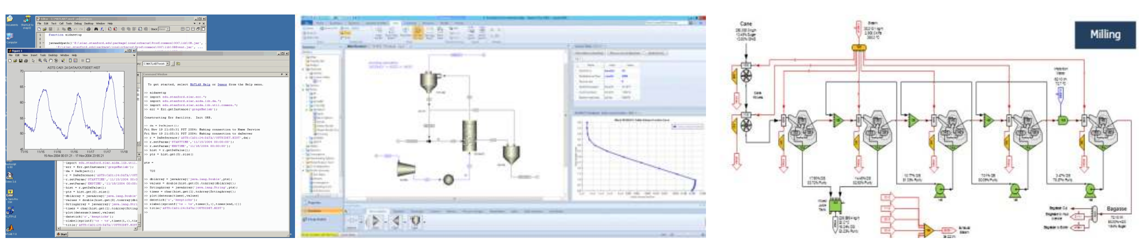

Figure 2. User interface of three modeling and simulation software: Matlab[1] (left), Aspen Plus[2] (center) and Sugars (right)[3]

In particular, Sugars™ is tailored to the beet and cane sugar industries. It includes common equipment such as mills, evaporators, clarifiers, boilers, etc. The units can be laid out as

[1] http://www.slac.stanford.edu/grp/cd/soft/aida/epicsChannelArchiverDpGuide.html

[2] http://www.aspentech.com/products/aspen-plus.aspx

[3] http://www.sugarsonline.com/

they appear in the real plant. Audubon Sugar Institute has been working towards the development of the Sugars model for all sugar factories in Louisiana. To date, complete factory models have been developed for Louisiana Sugar Cane Coop. (LASUCA) and for Lafourche Sugars. It is important to note that periodical maintenance of the model is required, which includes re-validation with current operational data such as new flow rates and updates to equipment.

APPLICATIONS IN THE SUGAR INDUSTRY USING SUGARS^{TM}

Factory balances.

The process model of a sugar mill can help in the quantification of steam and energy consumption, and stream flows that are not measured, as well as in the identification of possible losses throughout the factory. Figure 3 and 4 show the Sugars^{TM} model for the evaporation section of a factory grinding close to 10,000 tons of cane per day. Initially, vapor is bled only from first effect evaporator into juice heaters and pans. In this example, simulation is used to evaluate how the factory steam balance would change if vapor is bled from first effect evaporator into tertiary heater and pans, from second effect into secondary heater, and from third effect into primary heater.

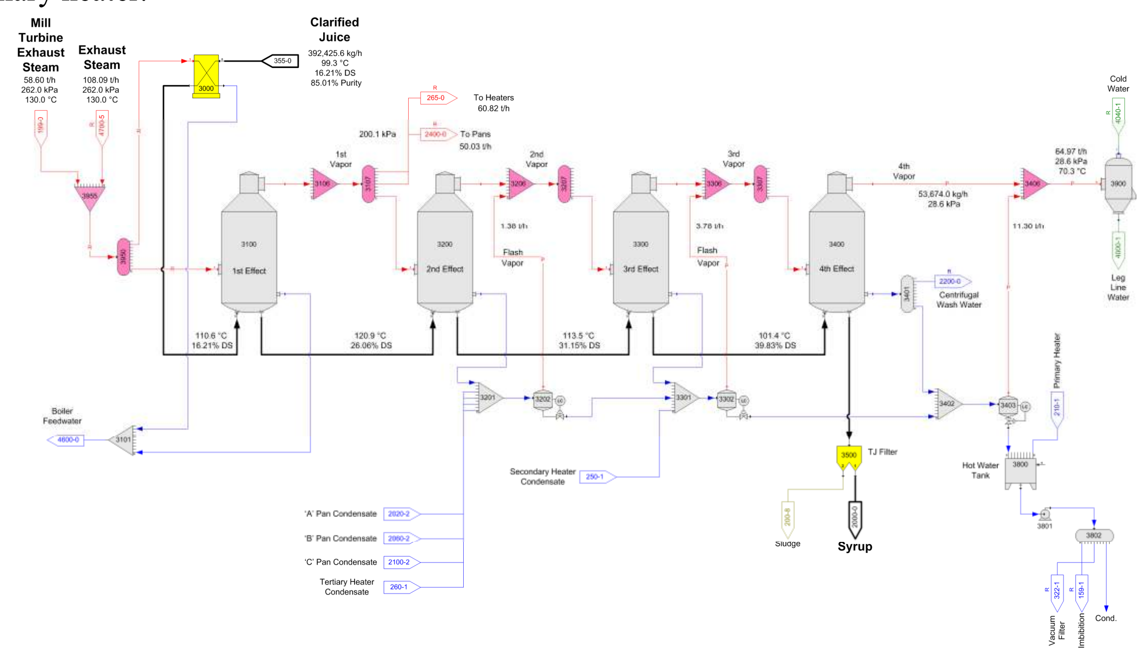

Figure 3. Sugars^{TM} model for evaporation section with vapor bleed from first effect evaporator only

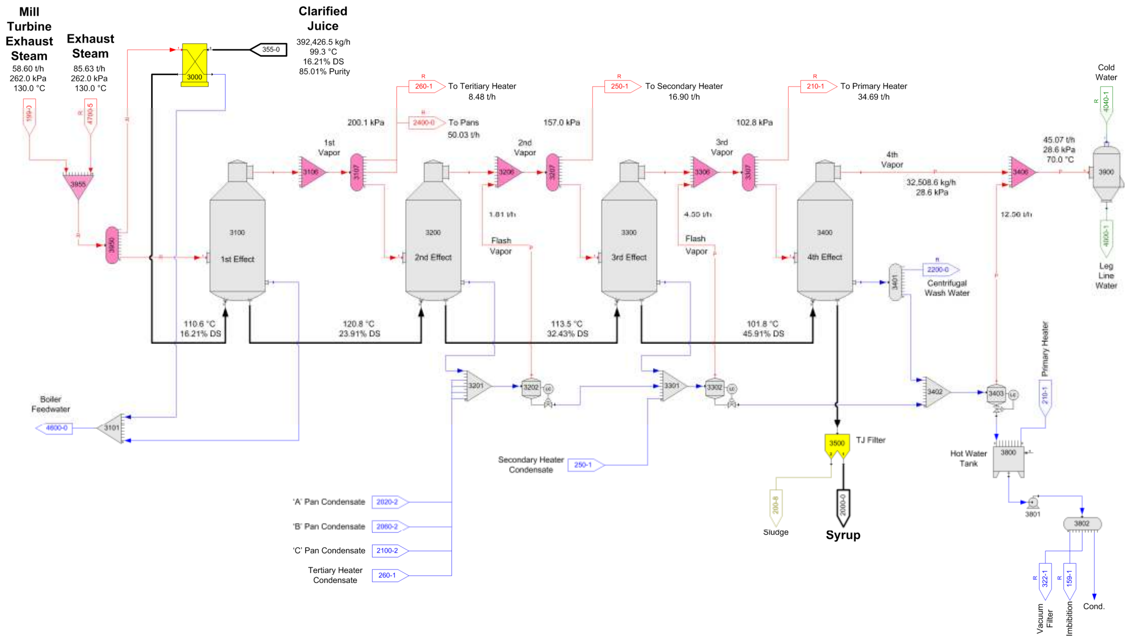

Figure 4. Sugars™ model for evaporation section with vapor bleed from first, second and third effect evaporators

For simplicity, Table 1 shows the changes in bagasse left and make-up steam for evaporation. Using this information, plant managers and engineers might decide on which is the best configuration to meet their goals. For example, if it is not possible to burn all bagasse because its moisture content is too high, it would be possible to determine whether changing the vapor bleeds could keep the factory in balance. In a different situation, if the factory report shows that make-up steam used is higher than what the model predicted, information from the simulation could also be used to determine possible locations of the losses and to quantify them, allowing plant personnel to devise strategies for correction.

Table 1. Leftover bagasse and make-up steam for two vapor bleed configurations

	Leftover Bagasse (ton/h)	Make-up steam for evaporation (ton/h)
One bleed	89	119
Three bleeds	94	95

Modifications to equipment and plant layout.

Throughout the years, sugar factories undergo changes in equipment, expansions and, possibly, changes in boiling schemes. Before the installation of new equipment, engineers might use process models to assist in the determination of design parameters such as required flow rates and equipment capacities, and in evaluation of expected product quality based on the overall production goal. As an example, let's consider that managers from the plant with three vapor bleeds from the previous example plan to install a new higher pressure boiler, to go from steam at 225 psia and 395°F to steam at 500 psia and 700°F. (Figure 5).

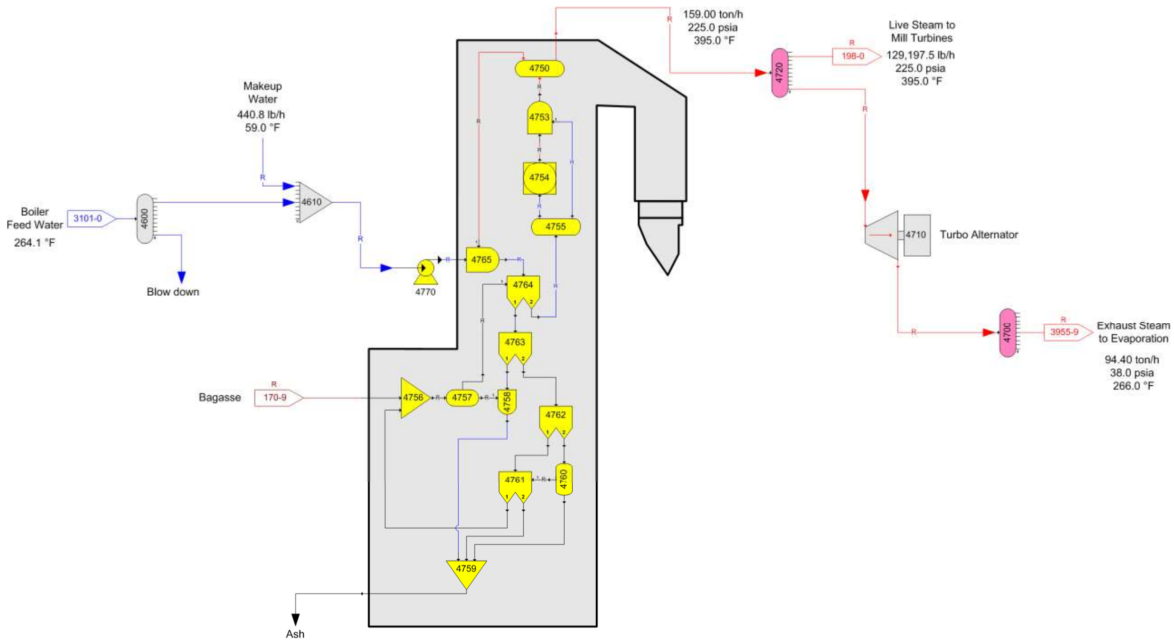

Figure 5. Sugars^{TM} model of bagasse boiler

With the lower pressure boiler, the turbo alternator produces 3 kilowatt-hour per ton of cane. In contrast, after making the changes in pressure and temperature of live steam produced, the turbo alternator produces 17 kilowatt-hour per ton of cane, representing an increase of more than 450%. Plant personnel might use these results to determine if the new boiler would be able to produce the electricity required by the plant. With this in mind, plant managers could also calculate the return of investment and decide whether to install the new boiler or not.

Evaluation of possible outcomes for changes in process conditions.

Plant personnel might use simulation to assist them in determining the course of action when process conditions change. An example of this application is to determine the amount of molasses required recycled back to A pans when syrup purity is too high. Also, simulation of the boiling house might be used to evaluate the changes in production of final molasses, production of raw sugar, purity of final molasses and pol of raw sugar, among others when different percentages molasses recycles are used. Ultimately, it is the engineers and superintendent's decision which settings are best to meet factory goals. Figure 6 shows the Sugars model of a boiling house using a three-boiling scheme and Table 2 shows simulation results for three percentages of B molasses recycles.

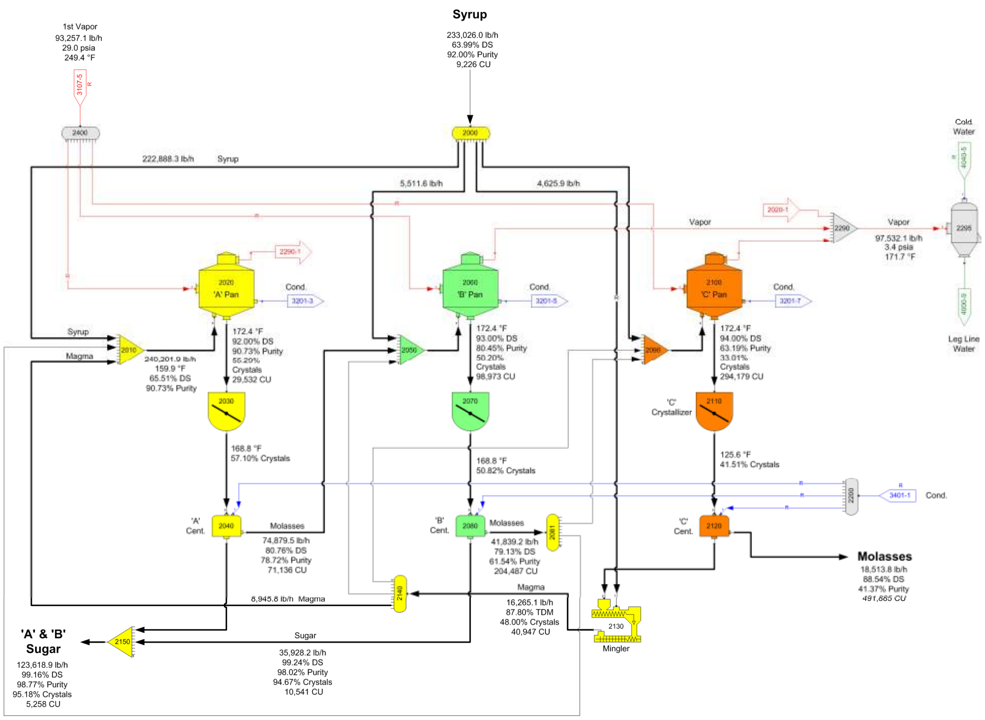

Figure 6. Sugars™ model of boiling house using three-boiling scheme.

When syrup purity is 92%, recycling 20% of B molasses into A pans would lower the final molasses purity from 44% to 41%, which is a significant reduction. Variations in internal massecuite flows could also be evaluated.

Table 2. Simulation results for different recycle of B molasses into A pans

B molasses Recycle (%)	Final molasses purity (%)	Final molasses (ton/h)	Raw sugar (ton/h)
10	42.7	9.59	61.55
20	41.4	9.26	61.81
30	40.0	8.92	62.07

Conclusion

Process modeling and simulation has the potential of assisting Louisiana sugar mills in maintaining and/or improving process operations to meet each factory's goals. If not the same person, the modeler and plant personnel must work hand-in-hand to obtain reliable and accurate models.

THE PRODUCTION OF MULTIPLE AGRICULTURAL FEEDSTOCKS AND PROCESSING TO BIOFUELS AND BIOBASED CHEMICALS

D. F. Day

The rationale for this project is to drive the expansion of biofuel crops throughout the Southeast United States by development of two crops suitable for this region. This is a region of great agricultural productivity but much in desperate need of a new economic base. By the same token, the biofuel industry has been limited by a shortage of readily convertible feedstocks (other than corn). This program targets both these problems. The Southern Regional Agricultural Sector can be expanded through production of sweet sorghum and energy cane, for the manufacturing of bio-based fuels and by-products. These crops are capable of being produced in this region. However, significant knowledge gap exists regarding the economic feasibility and sustainability of growing and processing these crops. A regional multidisciplinary consortium of agricultural scientists, biotechnologists, technology and engineering providers, economists and educators with the help of biofuel companies trying to secure sufficient carbohydrate and fiber feedstock to meet production goals was formed to facilitate production and conversion of these crops. This program includes: improving agronomic production of energy crops through breeding for selected parameters, screening using NIR; harvesting trials for energy cane and sweet sorghum using modified sugarcane harvesters. Utilizing a pilot facility to process selected feedstocks into fermentable sugar syrups, for industrial partners to test production scenarios. Supplying input for economic models addressing agricultural and processing and cost structures for feedstock - bioproducts possibilities. Initiating training programs for future biofuels workers and extending information to extension units in multi-state areas on the potential for agricultural development using these target crops.

INTRODUCTION

The Sustainable Bioproducts Initiative (SUBI) at the LSU AgCenter is working to expand the utilization of sweet sorghum and energy cane crops for the manufacture of bio-based fuels and by-products in order to grow the Southern Regional Agricultural Sector. The recent USDA Roadmap on Biofuels (USDA 2010) calls for production of 13.4 billion gallons of advanced biofuels (butanol or drop-in gasoline substitutes) from grassy crops. The Southern region is expected to supply 50% of these crops, as it has the most robust growing season. Energy cane and sweet sorghum are considered the appropriate fuel crops for this area. Any industrial facility can only run economically if it produces year round. Agricultural crops are inherently available only at harvest time. To solve this and several others problems we developed a model where small processing plants produce storable syrups of fermentable sugars, from crops with staggered harvests. These syrups can be shipped to centralized conversion facilities which produce and other products. Some highlights of the first two years of this program are presented.

CROPS

Developments

Seedlings and clones derived from cross hybridization of distinct species of grasses, (Saccharum spp hybrid) and two of its wild relative species, Saccharum spontaneum and Miscanthus were established in experimental plots in several climatically diverse locations across Louisiana. Two of these locations (St. Joseph; 31^o 55'07"N and Winnsboro; 32^o 9'48"N) are outside of the traditional sugarcane belt. Miscanes (derived from Saccharum x Miscantus cross) clones are being evaluated at Houma, LA (29^o 35' 15"N) and St. Joseph while seedlings (derived from true seed of cross hybridization between sugarcane and Saccharum spontaneum) are being evaluated at Houma and Winnsboro. Populations and individual clones performing favorably for combinations of these traits were selected for further testing and selection in subsequent trial stages. Markers are being developed to enhance selection for traits such as cold tolerance, biomass yield and biomass composition.

Energy cane plots, especially those planted outside of the traditional sugarcane belt are being closely monitored for the incidence of pests and diseases with a view to compel a plan of action to mitigate any adverse effects that growing this crop out of its range might have. Previously established energy cane trials in southern Louisiana were scouted for common pests and diseases, but none was at a level that called for concern.

Biomass composition is defined by a cumbersome to measure set of traits. To enable rapid screening of entries in breeding programs where large numbers of samples are usually processed, Near Infrared (NIR) Spectroscopy methods are being developed to improve accuracy and throughput in determining feedstock quality (fiber/biomass composition). NIR could also become the basis for developing a payment or pricing structure for a biomass feedstock as it is delivered to be processed to products. Calibrations are being developed for both sweet sorghum and energy cane parameters and then being extended to include biomass components.

Sustainable Production

Experimental plots were planted at Winnsboro, LA in July 2012 to determine energy cane feedstock production systems in northern Louisiana. The soils in and around the experimental area were sampled in April 2012 to determine baseline soil nutrient levels and for Life Cycle Analysis studies. To determine optimum harvest dates for energy cane 6 varieties were planted at Houma, LA in September of 2012. Six varieties, 4 energy cane varieties and 2 sugarcane varieties as reference were planted using billets and whole stalks to determine the best type of material for planting energy cane

A supply of sweet sorghum feedstock, based on staggered planting dates and hybrid maturity, was produced at the LSU AgCenter Sugar Research Station (St. Gabriel, La) from late July through the fall of 2012. Averaged over planting dates the early, medium and late maturing

hybrids yielded 1.74, 3.08 and 2.44 tons/A (3.90, 6.91 and 5.48 Mg ha-1) of fermentable sugar, respectively. An evaluation of the treatment effects on soil C bulk density analyses for multiple depths was conducted. Microbial biomass C, activity, and metabolic rate have been determined from subsamples of soil taken in the 0-8 cm depth for the energy cane and sweet sorghum plots for the 2012 samples.

Logistics and Storage

A transportation model (Leboreiro J. and A. Hilaly, 2011) to estimate the energy required and the emissions of greenhouse gases generated to supply biomass to the milling facility was incorporated along, with a milling model, into the LCA inventory to determine the inputs and output of this processing phase (based on crop amount needed). The milling model is based on the process layout of the LSU pilot plant to estimate energy demand (electricity and heat), energy surplus, bagasse generation, juice production, and syrup output, was developed. It was developed using the equations from E. Hugot 1986.

Harvesting trials were conducted for both sweet sorghum and energy cane in collaboration with John Deere. Collaborative research with Sugar Cane Growers Cooperative of Florida (SCGC) on improving the fuel value of bagasse has shown that bagasse can be fluidized, opening the possibility of utilizing a superheated steam dryer with 85% to 95% low pressure steam energy recovery. An engineering study comparing various bagasse driers has been conducted. A mass and heat balance model incorporating the superheated steam dryer is under development.

FEEDSTOCK CONVERSION/REFINING

A 5 ton per day syrup processing plant was installed at the Audubon Sugar Institute (Figure 1). This facility was operated in 2013 to produce syrups from both sweet sorghum and energy cane for industrial supporters so they could test their processes for producing fuels and products from these materials.

Figure 1. Syrup production facility at the Audubon Sugar Institute

One of our industrial supporters has contracted research on the development of butanol production from the feedstock syrups. An immobilized cell column using a strain of C. beijerinkii was constructed and has operated on a synthetic media with a variety of glucose containing carbon sources. The concentration of butanol was low, as is common with this fermentation. In order to increase solvent levels in solution to that which is economically feasible for distillation liquid-liquid extraction was employed

Pretreatment studies have focused on treating energy cane and sorghum bagasse with water or ammonia as catalysts, and determining finding the optimum parameters (temperature, retention time, catalyst:biomass ratio) for increased yields in xylan (C5 sugars), glucan (C6 sugars) and/or combined sugars (C5 and C6 sugars)after enzyme hydrolysis. Ammonium hydroxide as a pretreatment chemical enhances the surface area of cellulose, disrupts crystalline structures, extracts lignin, while the ammonia can be recovered due to its high volatility. At total of 32 pretreatments of sorghum bagasse were carried out at different temperatures (130°C, 160°C, 190°C, 220°C), retention times (30 and 60 min) and biomass to water loading ratios (1:8, 1:20).

ECONOMICS, MARKETS AND DISTRIBUTION

Economic research during the first two years of this project focused on the development of reliable estimates of production cost for energy cane and sweet sorghum. Primary outcomes from economic research conducted during Year 2 involve the relationship between the estimated costs of producing feedstock crops, primarily energy cane and sweet sorghum, and what market prices would be required for production of those feedstock crops to be able to compete economically for available cropland with existing crops in various regions.

EDUCATION

Biofuel utilization in the United States will ultimately depend on support of the general population. Our education program has several goals, including training programs at the professional level and training programs for potential workers in the nascent biofuel industry, education Professional level training focuses on developing educational programs in the broad areas of sustainable, renewable energy production at two campuses, one at the Louisiana State University and the other at the Southern University. Training programs for potential biofuel workers are being made available on social media as a series of video course. The first one completed is short course on “Essentials of Chemical Engineering for Non-Chemical Engineers”.

The CEES sustainability camp, run by Southern University put biofuels in as part of their program. Approximately 100 students were registered for the summer camp in 2013. The camp engaged 6 teachers from the EBR School system with the sole responsibility of developing the appropriate curriculum that will focus on bioenergy and its significance on the country's energy future. One of the highlights of the camp was a visit to the LSU Audubon Sugar Institute (Figure 2) to expose the students to the processing pilot facility that will use sugar cane grow from Louisiana crops to generate biofuels.

Figure 2. Students at CREES summer camp at the Audubon Sugar institute (2013)

EXTENSION

The extension team has been active, with Field day tours and events across Louisiana and surrounding areas. A highlight of the second grant year was a National Energycane/Sweet Sorghum field day at the USDA-ARS Sugarcane Research Unit in Houma, LA as a pre-conference tour for a 2012 National Sun Grant Conference entitled, "Science for Biomass Feedstock Production and Utilization", which was held in New Orleans, LA. As part of the information stream, a sugarcane/energycane variety identification guide was completed and posted on the Sustainable Bioproducts Initiative (SUBI) web site. An energycane/sweet sorghum production handbook, which will leverage existing cultural practices is in preparation.

CONCLUSIONS

The Southeast region of the United States is scheduled to produce a significant portion of the Nation's biofuel feedstocks in the next 10 years. Much of the region is agricultural in nature and has been plagued with low prices for its relevant crops, mainly cotton and timber. The region is populated with rural family farms in desperate need of a new economic base. By the same token, the biofuel industry has been limited by a shortage of readily convertible feedstock (other than

corn). This program offers the opportunity to improve farm incomes, create local manufacturing jobs and supply significant quantities of feedstock for the next generation biofuel industries. A successful program will provide new crops that can be produced sustainably in this region and the technology for converted locally them to a simple feedstock (fermentable sugar) for centralized biofuel production facilities. Energycane and sweet sorghum both contain fermentable sugars that can be deployed in biorefineries. The simple sugars present in these crops could support early development of production of sugars for biofuels, with LC sugars phasing in as the technology develops. The crops appear to be productive on marginal land, such that they don't impact current crops. We are now in a position to answer some key questions as to the utility of the sugars from these crops as biofuel feedstocks.

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CHARACTERIZATION OF ENZYME HYDROLYSATES DERIVED FROM DILUTE AMMONIA PRETREATED SORGHUM BAGASSE

P. J. Pham-Bugayong and G. Aita

INTRODUCTION

Sorghum is considered as an energy crop in almost all temperate, subtropical and tropical climates. The pretreatment and conversion of sorghum bagasse to ethanol has been extensively studied in Dr. Aita's laboratory[1-9]. Dilute ammonia pretreatment removed 44% of the original lignin and 35% of the original xylan (hemicellulose) while retaining 90% of the glucan (cellulose) [9]. Prior to conversion of the pretreated sorghum bagasse (PSB) into ethanol, enzymatic hydrolysis is employed. Enzymatic hydrolysis (saccharification) of lignocellulose has long been studied as a method to depolymerize biomass into fermentable sugars for conversion into biofuels and chemicals. A detailed characterization leading to an effective detoxification strategy of sorghum bagasse enzyme hydrolysate has not yet been reported. In most of the cases, only monomeric sugars, some organic acids and furaldehydes are detected. These are the ones monitored because of their known importance and inhibitory effect during the fermentation process. Detailed characterization is essential for the analysis of possible value-added product/s in the enzyme hydrolysate stream other than sugar monomers or inhibitory compounds. The enzyme hydrolysate derived from ammonia pretreated biomass is characterized in this study to aid in the design of a detoxification strategy with maximum sugar recovery yields and detection of possible value-added components (Figure 1).

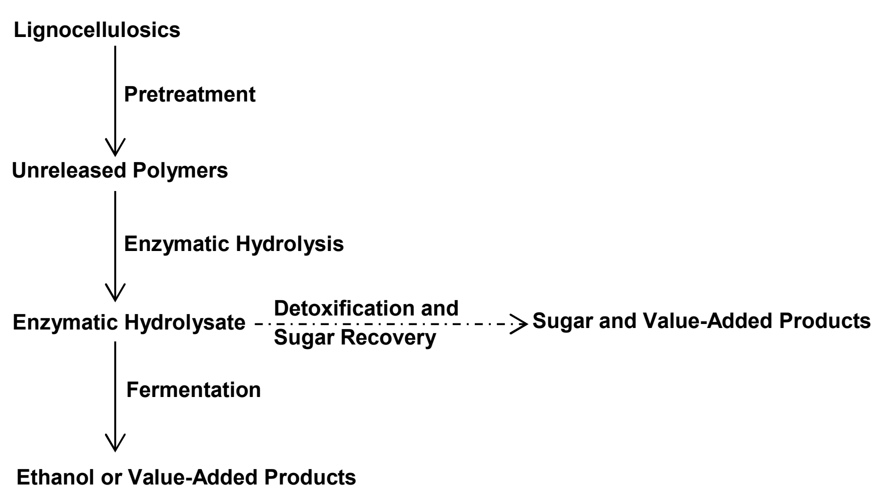

Figure 1. Detoxification and sugar recovery of enzyme hydrolysates.

EXPERIMENTAL PROCEDURE

Sorghum bagasse (head, stalk and leaves; juice extracted, dried and milled) was pretreated with dilute ammonia (28% NH_4OH solution) for 1h at 160°C in a 300-mL pressure reactor. The ratio of biomass to dilute ammonia and water was 1:0.5:8. The pretreated sorghum bagasse was enzymatically hydrolyzed using a combination of Spezyme®CP and Novozyme188, a cellulase and β-glucosidase, respectively. Also, combinations of Accelerase XY and Accelerase 1500 were used. Enzymatic hydrolysis was allowed to proceed for 72h. After which, the reaction was stopped by deactivating the enzymes. The resulting enzymatic hydrolysate (EH) was then rapidly cooled to about 25°C in an ice bath, centrifuged, and the mass and liquor (EHL) stored separately in the freezer until analyzed. Enzyme loading was based on the glucan (glucose) content and mass of dry biomass added (g glucan/g dry biomass). Biomass loading at 5% was used.

RESULTS

A High Performance Liquid Chromatography (HPLC) separation method with Diode Array Detection (DAD) was developed for the simultaneous determination of organic/aliphatic acids (1), phenolic acids (by-products of lignin degradation (2), and degradation products, 5-hydroxymethylfurfural and furfural (3) in enzymatic hydrolysate liquors (EHL) collected from 0h-72h. After establishing the EHL-HPLC method, relevant *markers* (or groups) of interest in the PSB-EHL were further characterized. The identified *markers* (or groups) were used to monitor the composition of all EHL sampled at different times (0-72h). Figure 2 shows the EHL composition of enzyme combination, 30 FPU Spezyme CP and 30 CBU Novozyme 188 obtained after 24h. Multiple components in the EHL were detected within the organic acid, furaldehydes and phenolic acid groups. Monomeric sugars were also monitored. Glucose, xylose and arabinose were detected. HPLC results also revealed the presence of other unidentified sugar components. The method was used for all EHL samples collected and provided detail into the optimum combination/concentration of enzymes generating maximum sugar with the least amount of non-sugar components. An EHL purification process was also developed. The method involved a C_{18}-SPE (**S**olid **P**hase **E**xtraction) cartridge (Empore Universal Resin-Standard density; 7mm/3mL) followed by elution of the concentrated sample with solvents of increasing polarity [ethyl acetate (EtOAc), methanol (MeOH), and H_2SO_4-H_2O mixture) to remove components adsorbed on the cartridge as fractionated groups (Figure 3). A modified glass column packed with silica gel (Chromatographic, 100-200 mesh) was also tested for stepwise solvent fractionation. The solvent fractions obtained from the elution were analyzed using the EHL-HPLC method developed.

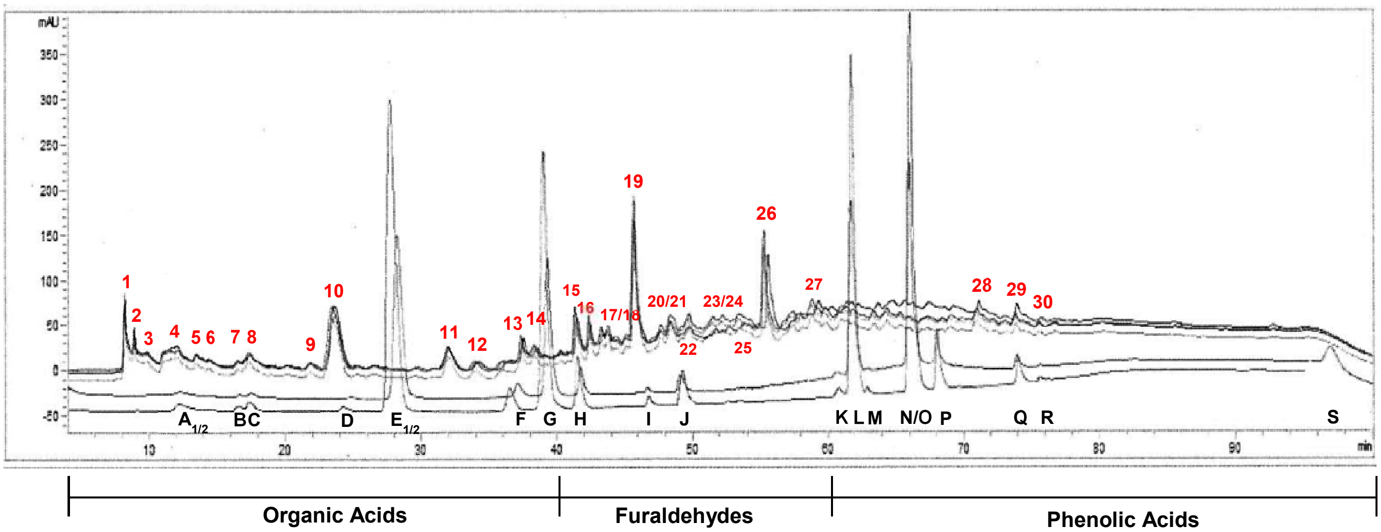

Figure 2. EHL composition of 30 FPU Spezyme CP and 30 CBU Novozyme 188 obtained after 24h.

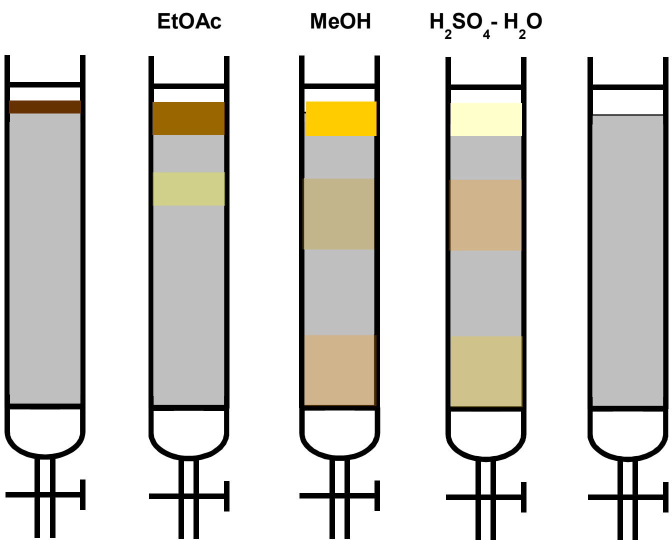

Figure 3. Step-wise solvent fractionation of EHL.

ACKNOWLEDGEMENTS

We would like to express our gratitude to the ASI Analytical Laboratory for the instrumentation. USDA-NIFA Award No.: 2011-69005-30515.

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SUGAR AND POLYMERS: NOVEL MATERIALS TO ADD VALUE TO LOUISIANA SUGARCANE

Derek Dorman

INTRODUCTION

Plastic is ubiquitous; yet, many neglect to recognize the abundance of products that use plastic components or packaging for preserving freshness and visual appeal. The variety in available plastics is not reflected in the types offered in most markets. There are merely 7 plastic recycling identification symbols including 2 for polyethylene. Although other types do exist, these 7 categories make up the majority of plastic found in homes. Electronics and appliances contain plastic that do not include a recycling symbol. They contribute less to landfill waste and litter since most of them are used for many years. About 251 million tons of municipal solid waste (MSW)[1] was generated in 2012 including 31.9 million tons of plastic[1]. Thanks to nation-wide recycling programs, 2.78M tons of plastic[1] was diverted from landfills; but, the remainder (28.9M tons)[1] was left to slowly degrade. Biodegradable plastic can reduce the accumulation of trash by persisting in the environment for years rather than decades. Byproducts of raw sugar and biodiesel refining can be used to produce biodegradable polymers and supply companies with alternative sources of plastic tailored to suit specific applications.

Aconitic acid (trans-prop-1-ene-1,2,3-tricarboxylic acid) occurs naturally in sugarcane and can be acquired without impacting recoverable sugar. It is a versatile acid which ranges in concentration from 0.5-3.0 % in sugarcane molasses[2,3]. It can be isolated from molasses or vinasse to yield 62-68 % free-acid at 99.9 % purity[4]. Polymers formulated using aconitic acid in combination with various polyols and capping compounds demonstrated a wide range of interesting physical properties ranging from hard and brittle to rubbery (table 1). Coupled with tunable biodegradability, these polymers show promise toward the production of food-packaging materials that are functional, non-toxic, and superior in terms of overall balance of energy. Because it was demonstrated that aconitic acid can be removed from molasses without disrupting the total sugar content[4], from which molasses derives its value for use as cattle feed, it is also true added-value product.

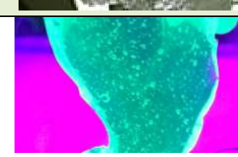
RESULTS AND DISCUSSION

Biodegradable polyesters are synthesized from aconitic acid or citric acid, glycerol, and cinnamic acid. A prepolymer is formed by bulk polymerization of glycerol and citric acid and heated with a drop or 2 of sulfuric acid to catalyze the reaction. Upon heating, citric acid dehydrates to aconitic acid. The prepolymer is poured on to a piece of aluminum foil and placed in a 90°C oven to cure overnight to form the highly crosslinked polymer matrix (figure 1). The amount of crosslinks can be controlled by the amount of time the polyester is allowed to cure. Citric acid is used as a precursor to aconitic acid due to accessibility, but aconitic acid derived from sugar

processing can be used directly in the reaction. The properties of the polymer can be adjusted by changing the stoichiometry of the monomers. The polymer made with citric acid or aconitic acid and glycerol produces a translucent, hard, and brittle plastic (figure 1 and table 1). Substituting some of the acid with sebacic acid produces a flexible polymer easily bent without breaking (table 1).

Figure 1 Biodegradable polyester after thermal curing at 90°C from the bulk polymerization of citric acid, glycerol and cinnamic acid.

Table 1 Properties of various polyester formulations including composition and followed by a photograph of the resulting polymer.

Properties	Monomers Used	Additives	Picture
Hard, Brittle, Translucent	Citric Acid, Cinnamic Acid, Glycerol		
Hard, Brittle, Translucent, Fluorescent	Citric Acid, Cinnamic Acid, Glycerol	Fluorescein	
Hard, Tough, Opaque	Citric Acid, Cinnamic Acid, Glycerol	Plaster	
Rubbery, Tough, Translucent	Citric Acid, Sebacic Acid, Glycerol		

CONCLUSION

Aconitic acid can be purified during raw sugar processing and used as a monomer for plastic. A wide array of plastics can be formulated from it, increasing the variety of specialty polymers available.

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