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Audubon Sugar Institute Contact Information:

Audubon Sugar Institute, LSU AgCenter
3845 Highway 75
St. Gabriel LA, 70776
Office: (225) 642-0135
Fax: (225) 642-8790

TABLE OF CONTENTS

LARGE SCALE RIPENER EVALUATION

Albert Orgeron[1], Kenneth Gravois[2], Benjamin Legendre[3], and Jim Griffin[4]

[1]St. James Parish LCES, [2]LCES, [3]Audubon Sugar Institute,
[4]School of Plant, Environmental and Soil Sciences

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 large scale study (25 acres) was conducted on a second stubble field of HoCP 96-540 at Blackberry Farms in Vacherie. A randomized complete block (RCB) experimental design was used to evaluate the ripener treatments. Treatments were replicated 3 times. Aerial application of Moddus (19 oz/A) was applied on August 27, 2014, and Roundup PowerMax (5.3 oz/A) on September 24, 2014. Plots were harvested October 22, 2014, resulting in a ripener treatment duration of 57 days for Moddus and 29 days for Roundup PowerMax. Two tractor-trailer loads were harvested from each treatment by combine. The harvested area for tractor-trailer loads varied from 0.55 to 0.70 acres. Harvested area and scale weights obtained from Lafourche Sugar Factory were used to calculate sugarcane yield (tonnage). 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. Both Moddus and Roundup PowerMax increased TRS by 11.8 and 13.9% above the nontreated control, respectively (Table 1). Sugarcane yield was negatively impacted by Moddus which reduced sugarcane yield by 4.4 tons/A. Roundup PowerMax sugarcane yield was equivalent to the nontreated control. Sugar yield was not statically improved regardless of ripener treatment.

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 2014.

Ripener Treatment	TRS lb/ton	% TRS Increase	Sugarcane Yield Tons/A	% Sugarcane Yield Decrease	Sugar Yield lb/A	% Fiber
Nontreated	197 b		47.4 a		9307 a	17.3 a
Moddus (19 oz./ac)	220 a	11.8	42.0 b	11.4	9216 a	18.1 a
PowerMax (5.3 oz./ac)	224 a	13.9	44.3 ab	6.4	9917 a	17.6 a
P-value	0.0157		0.0407		0.1334	0.7707

THE MYSTERY OF LOWERED PURITIES AND SUGAR YIELDS DURING THE 2014-2015 HARVEST SEASON

B. Legendre, H. Birkett and D. Day

Audubon Sugar Institute, LSU Agricultural Center, St. Gabriel, LA.

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 freezes occurred in Louisiana on November 14-15 and November 18-19 where the temperatures at Ryan Airport in Baton Rouge were 29, 29 29 and 27°F, respectively. Then on November 16 and 17, 2.64 and 0.35 in, respectively, of rainfall were recorded. At this point in the harvest, approximately 50% of the 12.8-million ton Louisiana crop had been processed by the state's 11 factories.

Following the freezing conditions that occurred between November 14 and 19, there was a precipitous drop in brix and sucrose reported at most factories resulting in a significant drop in sugar recovery. In most instances, the drop in sucrose content was greater than the drop in brix resulting in a significant drop in syrup purity as well. Field conditions prior to November 16-17 were mostly dry with excellent cane and juice quality. The first freeze event on November 14-15 occurred with very dry field and soil conditions prior to the significant rainfall that occurred on November 16-17. It appeared that with the first freeze event, there was more damage to the canopy and stalk tissue than you would expect with a low temperature of 27-29°F (Table 1). Then with the significant rainfall event that occurred on November 16-17 there was, undoubtedly, a significant uptake of water which had the effect of diluting both brix and sucrose of juice. Prior to the rainfall, the sugarcane plant was possibly under limited water stress which added to the high brix and sucrose content of the juice. However, with the take up of water it is possible that the plant also absorbed various cations that would add to brix but not sucrose thus lowering purity.

Table 1. Effect of freezing temperatures on damage to the sugarcane stalk assuming freeze duration of 8-12 hr. hours.

Temperature	General effect	Comments
> -2.2 °C (> 28°F)	-Only slight damage to terminal buds and tender leaves. -Sucrose content continues to rise	-Can generally still plant the stalk with good germination
-2.8 to -3.9°C (25 to 27°F)	-Growing point and top third of the stalk is affected. -Most lateral (auxiliary) buds are killed. -Sucrose content and purity increase for a short time due to dehydration	-Minimal deterioration for 4-6 weeks assuming the top 6-12 in of stalk is removed.
-4.4 to -5.6°C (22 to 24°F)	-Most tissue is killed. -Lateral buds begin to weep and allow the entrance of bacteria into the tissue with associated mannitol and dextran.	-Expect some deterioration in 2-4 weeks depending on the sugarcane variety and post-freeze weather conditions. Removal of the top 12-18 in of the stalk is strongly recommended.
< -5.6°C (< 22°F)	-All tissue is killed. -The rind usually splits to produce freeze cracks that allow direct entrance of bacteria into tissue.	-Expect severe deterioration in 1-2 weeks. -Removal of the top 12-18 in of stalk is required; however, significant reduction in sugar yield will still occur.

Dr. Don Day theorized that the drop in purity might be the result of the action of a plant enzyme, namely invertase. He explained that sugarcane produces two types of invertase, an acid and an alkaline invertase, each characterized by the pH where they exhibit maximum activity. The enzyme is important to the plant for sucrose utilization. Given the low pH of juice, the acid invertase may be more important for this discussion. Acid invertase normally declines during maturation of the cane, and increases as growth starts, but it never completely disappears, as it has a role in maintaining cell turgor. It is normally found in vacuoles and apoplastic spaces of internodes. Slow maturing cane shows higher levels of this enzyme later in the cycle than earlier maturing varieties. This year we had both drought and freeze conditions. It is possible that drought slowed the decline in acid invertase levels in the plant as it would be required to help maintain osmotic pressure, which followed by freeze damage or harvest damage, would release the enzyme into the juice causing a decline in purity and an increase in invert levels.

This might explain some of the drop in sucrose content, purity and sugar recovery following what was thought as a minor freeze event. Every year is so different in Louisiana and the dry harvest conditions followed by a “dry” freeze and then followed by a significant rain event and subsequent freezing conditions triggered something in the cane not seen in recent years. Regardless, Louisiana still had record sugar yields of 232 lbs sugar/ton for the crop.

However, for the week ending 11/15/14, sugar yields exceeded 258 lbs/ton at Alma Plantation (Lakeland, LA) and 240 lbs/ton for the State as a whole (Table 2). After the first freeze and rainfall events, sugar yields dropped 15.3 lbs/ton at Alma Plantation and 5.9 lbs/ton for the State as a whole. But by the second week after the freeze sugar yields tumbled 23.8 lbs/ton at Alma Plantation but only 9.6 lbs/ton for the State when compared to the week ending 11/15/14. After the week ending 11/29/14, sugar recovery at Alma Plantation actually rebounded from the previous week while for the State as a whole sugar recoveries dropped another 1.7 lbs/ton. It is interesting to note that both absolute juice purities and syrup purities dropped significantly from week ending 11/15/14 through 11/29/14 (Table 2). However, after week ending 11/29/14 juice and syrup purities stabilized. One can’t also overlook the fact that, in many cases, farmers were nearing the end of their ripener- treated cane by mid-November which might have had a negative impact on juice and syrup purities towards the end of November that coincided with the freeze and rainfall events. At the same time, it appeared that many farmers had started harvesting a significant amount of the variety L 01-299 which is known to have a lower sucrose content, purity and sugar yield in plant cane and stubble crops when compared to most of the other commercial varieties grown in the State with the possible exception of L 99-233.

Table 2. Pol % Cane, absolute juice purity, syrup purity, sugar yield and fiber content for the weeks ending 11/15/14, 11/22/14, 11/29/14 and 12/06/14 taken from the Weekly Comparative Manufacturing Report prepared by Dr. Harold Birkett.

Parameter	Week ending 11/15/14 Alma	State	Week ending 11/22/14 Alma	State	Week ending 11/29/14 Alma	State	Week ending 12/06/14 Alma	State
Pol % Cane	14.30	13.35	13.53	13.00	12.97	12.81	13.13	12.72
Juice Purity	86.22	85.21	85.70	85.09	84.85	84.64	84.44	84.55
Syrup Purity	88.02	87.30	87.26	87.20	86.51	86.83	86.50	86.65
Sugar Yield	258.8	240.2	243.5	234.3	235.0	230.6	240.3	228.9
Fiber	12.91	13.58	13.25	14.11	13.53	13.98	13.44	13.99

Each year the USDA-ARS, Sugarcane Research Unit in cooperation with the LSU AgCenter conducts field testing of commercial and candidate varieties for stalk cold tolerance at the Ardoyne Farm, Chacahoula, LA. The results of these tests help in the harvest management of varieties especially once a freeze event has occurred. The current reaction of Louisiana varieties to freezing temperatures of 24-26°F is found in Table 3. Also included in the tests is an Argentine variety, TucCP 77-42, which is known to have little or no tolerance to freeze events at the above stated temperatures and used as a check in these tests. During the 2014-2015 harvest season the temperature at the Ardoyne Farm never reached the threshold where significant deterioration occurred in standing cane.

Table 3. Stalk cold tolerance of Louisiana sugarcane varieties following a freeze event of below 26°F based on post-freeze deterioration.

Sugarcane Variety Post-Freeze Deterioration		
Good	**Moderate**	**Poor**
LCP 85-384	Ho 95-988	L 99-226
HoCP 85-845	L 97-128	L 99-233
CP 89-2143	HoCP 00-950	L 03-371
HoCP 96-540	Ho 05-961	TucCP 77-42
L 01-283		
L 01-299		
HoCP 04-838		

In summary, there was not any definitive research that could prove what exactly happened to reduce juice and syrup purities and sugar yield during the 2014-2015 harvest season; however, there are several possible explanations for the decline which include: 1) freeze events; 2) rainfall events; 3) invertase activity; 4) end of ripened cane; and, 5) change in varieties. This is by no means the only possible scenarios as to what happened during the harvest season. However, history will show that the 2014-2015 harvest season set a new record for sugar recovery.

COMBINING PROCESS MODELING AND BOILING HOUSE ASSESSMENT DURING FACTORY OPERATIONS

Daira Aragon, Iryna Tishechkina, Gabriel Rivera and Frank Fincher

INTRODUCTION

Crystallization of sucrose in the boiling house is the second most important operation for the recovery of sugar from the cane in a raw sugar factory. Measurements of purities, temperatures and crystal size are taken from different points of the process to assess the performance of the C-strike and make the adjustments necessary to obtain maximum exhaustion. Simulation of the boiling house and, specifically, the C-strike, can complement the data measured in the factory to obtain values of process variables that are not routinely measured. Once the model is developed, obtaining results does not require experiments or laboratory analysis, so it does not consume valuable resources during the grinding season.

A preliminary study of the performance of the C-strike in Louisiana sugar mills was conducted in 2014. Nutsch purities along the C-strike for two factories and crystal size of C-massecuites after reheater and of C-sugar for five factories were measured. A model of the C-strike of one factory was developed and simulated to demonstrate the potential use of process models in complementing factory data.

PERFORMANCE OF C-STRIKE

The sampling points for this study are shown in Figure 1. Samples of final molasses and C-sugar were collected weekly from each centrifugal in operation at two of Louisiana's sugar mills for a total of 5-7 weeks. Nutsch molasses were obtained from massecuites samples collected at the factory from the following points of the C-strike:

- Receiver, as soon as the C-pan had dropped.
- After crystallizers. In some cases, more than one crystallizer was sampled.
- After the last reheater, right before the centrifugals.

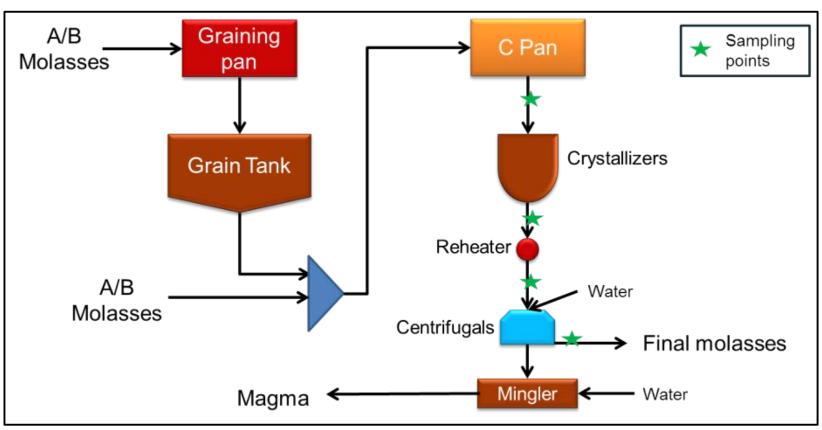


Figure 1. Sampling points in the C-strike station

Results obtained are summarized in Table 1. These values include data provided by a third factory that performs Nutsch analysis routinely.

Table 1. Summary of results for performance of the C-strike

	Molasses Purities (%)				C-mass reheater crystal size (microns)			C-sugar crystal size (microns)		
	Pan Nutsch	Crystal Nutsch	Reheater Nutsch	Final	D90	Mean	CV	D90	Mean	CV
Average	35.32	26.92	28.30	30.88	398	196	0.70	397	201	0.68
Minimum	30.15	24.52	24.84	25.99	342	133	0.60	371	160	0.58
Maximum	37.89	30.94	33.06	39.34	426	233	0.83	438	240	0.88
Stdev.	2.48	1.55	2.47	2.59	32	32	0.07	17	21	0.08
#Samples	17	16	17	29	11	11	11	30	30	30

Nutsch purities of the massecuites averaged 35.32% after the pan dropped, 26.92% after the crystallizers and 28.3% after the reheater. This gives a purity drop until after the crystallizer of 8.04% and a total purity drop of 7.02% until after the reheater. Purities of the material feeding the pan were not measured. Purity of final molasses averaged 30.88%, corresponding to a purity rise of 2.58%. Overall, values from the different factories presented differences between 10-12%. Difference between mean crystal size of C-massecuites and C-sugar is 5 microns, and on the D90 is only 1 micron, indicating that there is no much breakage of crystals in the centrifugals.

COMPLEMENTING PLANT DATA WITH SIMULATION

The model of the complete C-strike operation of one factory was modeled and simulated in the software SUGARS^{TM}. Once the layout of the station was determined, parameters within the software were adjusted to match previous factory data with the simulation results. Input data used for model development was:

- Temperature and pressure of steam
- Flow rate, purity and Brix of A and B molasses
- Brix of C grain
- Brix of C massecuites
- Temperature of massecuites
- Purity and Brix of final molasses
- Purity and Brix of C sugar
- Brix of C-Magma
- Water temperature

Some of these values, such as the Brix and purity of C-sugar and the purity final molasses, were used both as input data and for checking model deviations, because of the software internal iterations. Figure 2 shows the C-strike as created in the software SUGARS.

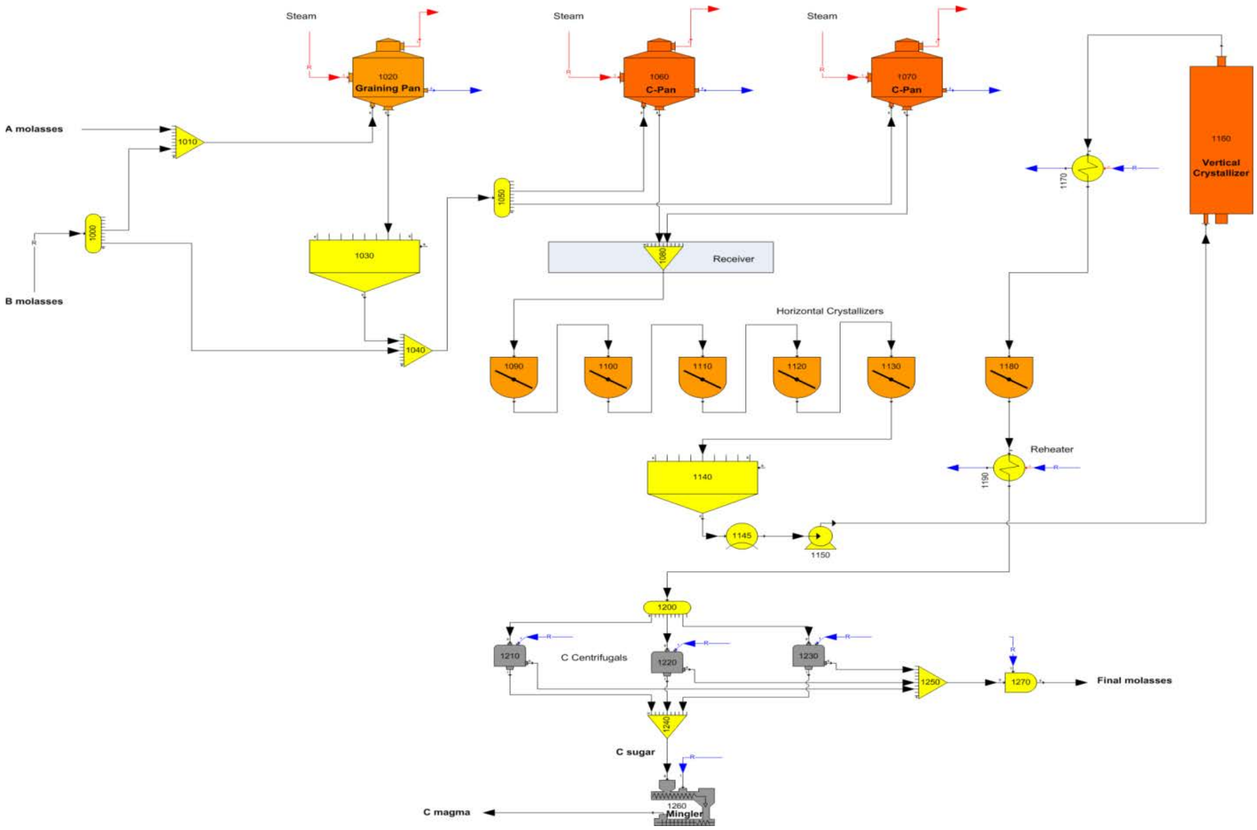


Figure 2. C-Strike station in software SUGARS

Results obtained in the simulation and their deviations from factory data during model development are shown in Table 2. Overall, the match of the model to the process was very good; simulation results had a maximum deviation of 6.24% from factory data, corresponding to the C-grain flowrate.

Table 2. Simulation results and their deviation from factory data during model development

Variable	Factory data	Simulation	Deviation from factory data (%)
C-Grain flow rate (lb/h)	5,346.4	5012.6	6.24
C-Grain purity (%)	58.40	58.14	0.45
C-Massecuite flow rate (lb/h)	46,207.7	45,921.8	0.62
C-Massecuite purity (%)	51.59	51.52	0.14
C-Sugar flow rate (lb/h)	18,497.6	18,333.8	0.89
C-Sugar purity (%)	84.72	83.71	1.20
C-Sugar Brix (%)	99.37	99.89	0.49
Final molasses flow rate (lb/h)	27,710.1	27,950.4	0.70
Final molasses purity (%)	28.97	29.80	2.88
C-Magma flow rate (lb/h)	19,829.2	19,557.3	1.37

Once the model was developed, simulation was used to obtained additional information, which was not measured routinely during operation, in this case, crystal content of massecuites and centrifugal performance. Simulation was also used to study the variations in purities and crystal content for changes A-molasses purity.

CRYSTAL CONTENT

Figure 3 shows the crystal content obtained during simulation. Values ranged from 16.42% to 62.04%. If these values are lower or higher than expected, factory personnel can pin-point trouble equipment and establish changes to the boiling procedure or operating conditions to bring the process back to the desired values. For example, very low crystal content at the end of the strike (shown in figure after the C-pan) may require increasing the boiling time or adding more seed into the graining.

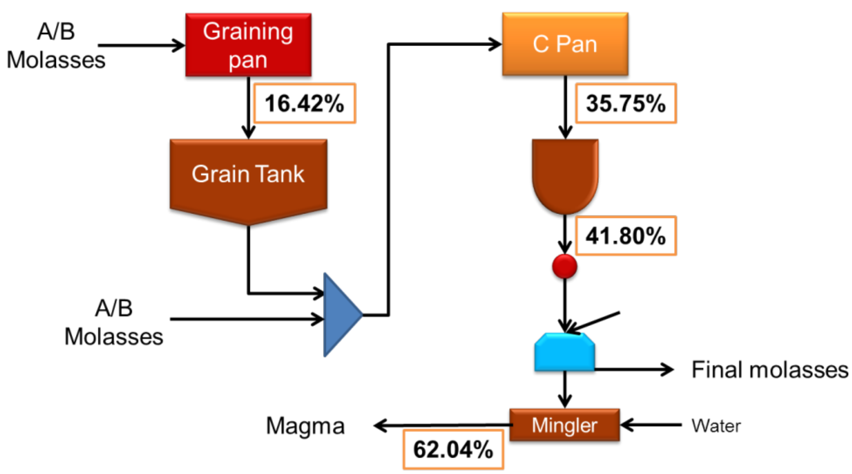


Figure 3. Crystal content along the C-Strike station

CENTRIFUGAL EVALUATION

Good performance of the centrifugal is important to minimize losses to the molasses. Simulation can be used to assess the performance of the centrifugals. It should be noted that initial experimentation is necessary to develop a reliable model. After this, experimentation is only necessary when there are changes to the equipment or for model maintenance. The software SUGARS provides information about the performance of the centrifugals as presented in Table 3.

Table 3. Centrifugal performance parameters

Parameter	Value
Crystal loss ratio	23.28%
Liquor purge ratio	93.75%
Wash purge ratio	92.95%
Non-sugars in sugar	6.25%

For this particular process, there is a loss of 23.28% of crystals into the final molasses. The liquor purge ratio is the percent of the mother liquor of the massecuite which goes into the molasses (wt. %). The wash purge ratio is the percent of the wash flow into the centrifugal that is purged out to the molasses (wt. %). The purge ratios are high with a wash/massecuite ratio of 28 wt.%. These values can be used to determine if there is a problem with the centrifugals; for example, a high crystal loss ratio may indicate a damaged screen or small C-sugar crystal size.

PURITY CHANGES

The model was also used to evaluate the variations in purities and crystal content when there are changes in A-molasses purities. Figures 4 and 5 show the results obtained in the simulation. Purity and crystal content of C-grain are affected to a higher degree than other variables. This is expected as A-molasses are used directly in the graining pan only. During operation, figures such as these ones can help plant personnel to determine the upper and lower limits of purities that can be tolerated.

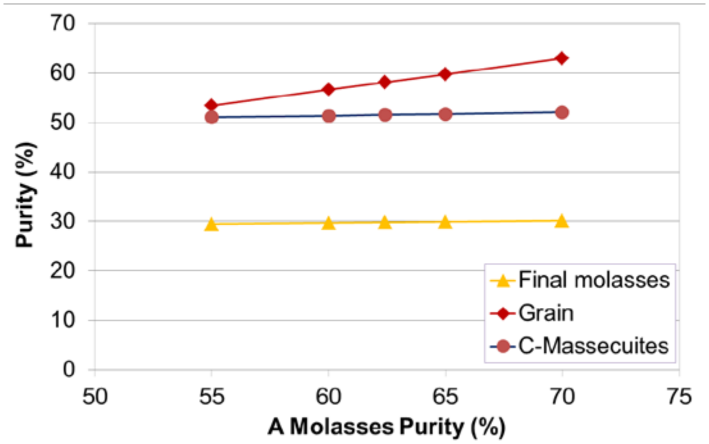


Figure 4. Purity of final molasses, grain and massecuites vs. A-molasses purity

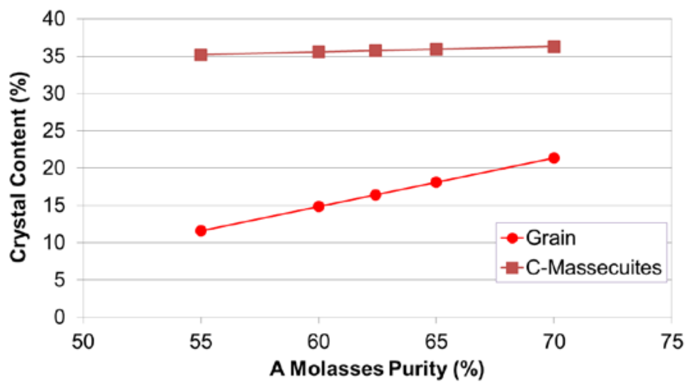


Figure 5. Crystal content of grain and massecuites vs. A-molasses purity

SUMMARY

Purity drop from C-pan to crystallizer averaged 8.4 points, purity drop until after reheater averaged 7.02% while purity rise across centrifugals averaged 2.58 points. Crystal size of C-Sugar agrees with C-Massecuites with mean crystal size 196-201 microns, indicating little breakage in the centrifugals.

Process simulation is useful to estimate parameters and process conditions that are not measured routinely during operation, and to study effects quality and operational parameters. Figures can be obtained to help plant personnel in assessing performance of the C-strike and determining the best course of action after a problem has been detected.

ACKNOWLEDGMENTS

American Sugar Cane League for providing funding for this project; the participating mills Lafourche Sugars, Cajun Coop., Lula-Westfield LLC., Cora Texas Manufacturing Co., M.A. Patout and Son Ltd. and their personnel for providing all information requested, and to Chardcie Verret and Franz Ehrenhauser from Audubon Sugar Institute.

SIZE MATTERS – CRYSTAL SIZE ANALYSIS FOR LOUISIANA SUGAR MILLS 2014/15 Season

Franz Ehrenhauser, Iryna Tishechkina, Daira Aragon

During the 2014/15 season more than 288 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.

During the 2014/2015 grinding season 208 samples of A, B, C sugars were evaluated. The size of the sugar crystals is governing their growth rate, their purity, and the ability to separate them from the molasses. Narrow, uniform distributions are desirable to minimize washing effort in the raw sugar mill and baking and dust issues in the refinery. Table 1 presents an overview of the measured parameters of C-sugars for the 2014 crop.

Table 1. CV-values, median and 90-percentile of C-sugars during the 2014 crop. (Average values given ± standard deviation)

Mill	# samples	Median Size [µm]	90-Percentile [µm]	CV
A	30	193 ±33	408 ±21	0.68 ±0.07
B	19	209 ±17	408 ±14	0.63 ±0.04
C	7	151 ±25	376 ±23	0.74 ±0.04
D	5	120 ±9	379 ±9	0.84 ±0.03
E	28	170 ±36	370 ±36	0.71 ±0.08
F	18	165 ±24	387 ±22	0.73 ±0.05
G	4	145 ±5	398 ±4	0.80 ±0.01
H	1	105	363	0.81
I	6	180 ±26	398 ±12	0.76 ±0.06
J	6	172 ±63	402 ±37	0.75 ±0.13
K	13	167 ±20	375 ±10	0.68 ±0.07
All Mills	137	176 ±36	391 ±27	0.70 ±0.08

The CV values range from 0.63 to 0.81. A low CV value is desirable to improve the purging performance of the sugar. The average median sizes range from 105 to 220 µm and the 90-percentiles range from 363 to 429 µm. The CV values correlate inversely with the size of the crystals, i.e. larger crystals exhibit lower CV. As such larger C-sugar is desirable to facilitate operations.

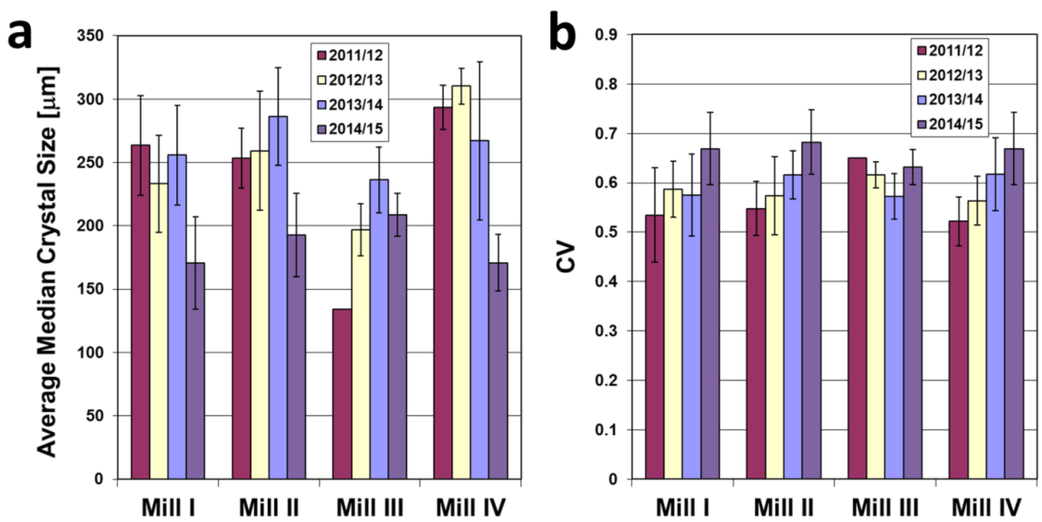


Figure 1. (a) Average median crystal size and (b) CV of C-sugar for mills I-IV during the 2011/14 seasons.

Figure 1 displays the trends of median crystal size and CV values for C-sugar for four mills. These mills have provided over the last four seasons enough samples (weekly) to allow an evaluation of trends in their C-sugar size. Clearly visible is in Figure 1a the decrease in the crystal size. As this is consistent for all mills it reflects most likely unique processing conditions due to the cane quality and weather conditions. CV values have been increasing for all mills compared to the previous season. This increase is due to the dependence of the CV value to the crystals size, i.e. a certain distribution width (e.g. with a fixed range of 100 microns) will give a higher CV value for smaller crystals than for larger crystals. The reason behind the crystal size decrease is unclear. Current speculations involve either modifications to the boiling scheme and/or procedure, limitations in the boiling house residence time, or impurities limiting crystal growth, among other possibilities.

In addition to the measurements of C-sugars during the 2014 crop, a research project surveying the seed slurry preparation methods was conducted. The seed slurry is a mixture of very fine sugar and isopropyl alcohol which is used to initiate the crystallization of C-sugar. The sugar serves as seed for the growing sugar crystals in the pan. Three seed slurry preparation methods are used in the Louisiana industry. Either powdered sugar is dispersed in isopropyl alcohol or granulated or powdered sugars are ball milled in isopropyl alcohol to yield a suspension of micrometer-fine sugar particles.

Powdered sugar of different refineries/beet sugar factories differ in their size and as such in their slurry properties, therefore consistency of the selected sugar can be of importance for the sugar boilers. Table 2 displays the size distribution parameters of several powdered sugars available in Louisiana and the US. While the size of the powdered sugar falls for all measured sugars between 3 and 50 microns, there are subtle differences, which should be taken into consideration. Western sugar provides the most narrow size distribution with a CV of 0.62, while Milliana sugar has the smallest average size resulting in the most number of seed crystals per gram of powdered sugar. Clearly, if the powdered sugar is changed the change in the number of added crystals should be considered. Generally slurry preparation via mixing powdered sugar and isopropyl alcohol is a convenient method, though several points should be taken care of: To allow for good consistency the powdered sugar should be stored dry, ideally in a climate controlled environment. Alternatively keeping powdered sugar in sealed containers also allows for good storage (e.g. plastic bag inside a tightly closed pail or drum). Some mills provide pre-weighed aliquots of powdered sugar in individual plastic bags to the pan floor. This seems to be a recommendable practice to ensure the control and the consistency of the slurry preparation.

Table 2. Powdered sugar crystal sizes

Powdered Sugar	**D10% [µm]**	**D50% [µm]**	**D90% [µm]**	**CV**	**# Crystals/gram sugar**	**Cane Sugar Y/N**
Domino (bag)	3.8	18	46	0.75	51×10^6	Y
Domino (box)	3.6	19	50	0.77	51×10^6	Y
Western Sugar	4.9	19	41	0.62	66×10^6	N
C&H	3.3	17	47	0.79	63×10^6	Y
Milliana	3.1	16.4	41	0.74	86×10^6	N

The alternative to using powdered sugar and IPA directly is to either mill powdered or granulated sugar in a ball mill. While the ball mills used in Louisiana are all of the same type (Protech), the recipes, i.e. the quantity of sugar and IPA for ball milling the slurry vary widely. Table 3 presents all recipes as they were reported prior to the 2014/15 season. The ball milling time is the most uniform parameter with 4 hours. The ratio of sugar to alcohol varies greatly from 83.3 g/L to 737.1 g/L. This ratio affects the efficiency of the milling in the ball mill. Figure 2 shows the milling progress of the granulated sugar samples, depicted as crystals per gram of sugar versus the milling time. Based on the conducted experiments there are several observations possible: Granulated sugar takes some time to break down, as at the beginning of the milling process the number of crystals changes only slowly. After approximately one hour the number of crystals increases linearly with time. The ball milling process is very reproducible as the number of crystals does not vary significantly between experiments on the same ball mill. The milling efficiency, i.e. the increase of number of crystals per gram over time (the slope) is proportional

to the richness of the sugar/IPA mixture – mixtures with high sugar content mill faster. The number of crystals per gram of sugar exceeds the number of crystals found in powdered sugar.

Similar observations are possible for ball milling powdered sugar. Figure 3 shows the progress of ball milling for powdered sugars. The main difference to the granulated sugar milling is here the absence of a lag phase, i.e. the number of crystals increases immediately linearly with time. The sugar quality affects the ball milling results, i.e. an old, clumped powdered sugar will produce less number of seeds; however as the number is 20 times larger than when compared to the virgin powdered sugar the impact is less than when using the clumped sugar without ball milling.

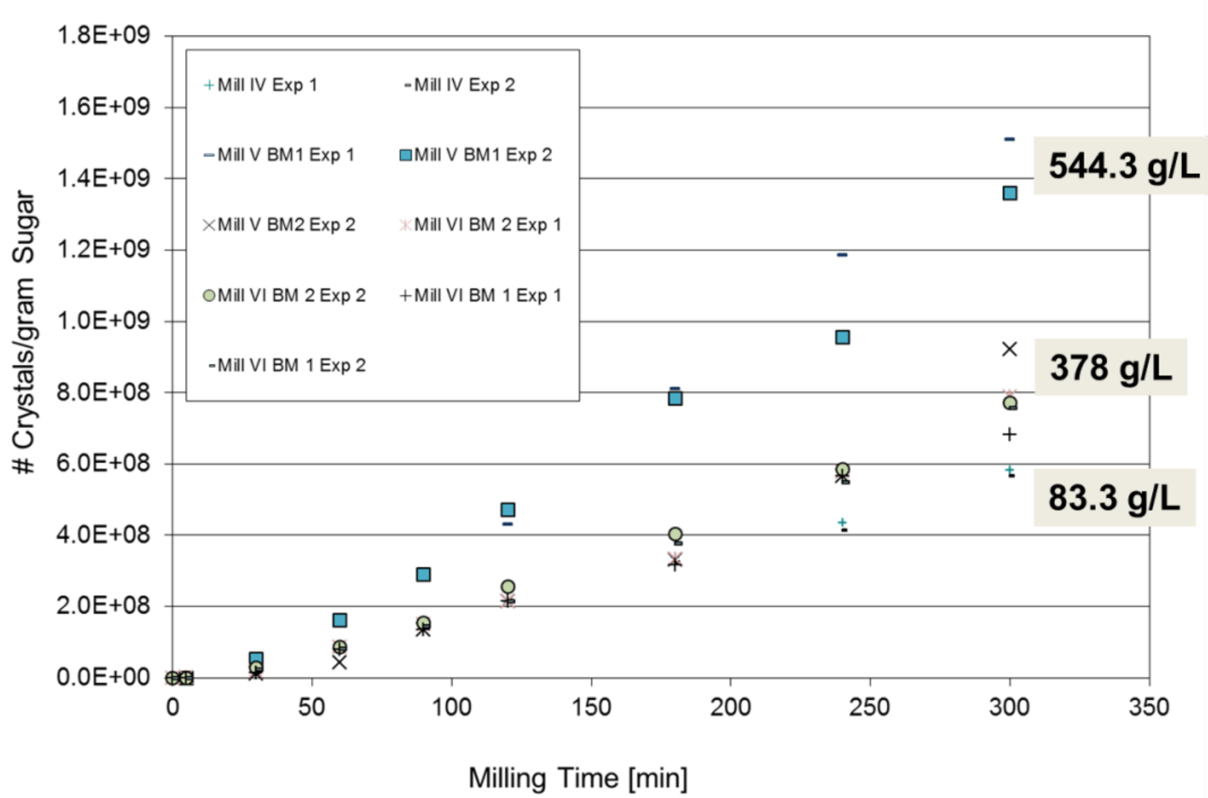


Figure 2. Milling progress of granulated sugar in ball mills in Louisiana. Sugar/IPA ratio in grey boxes.

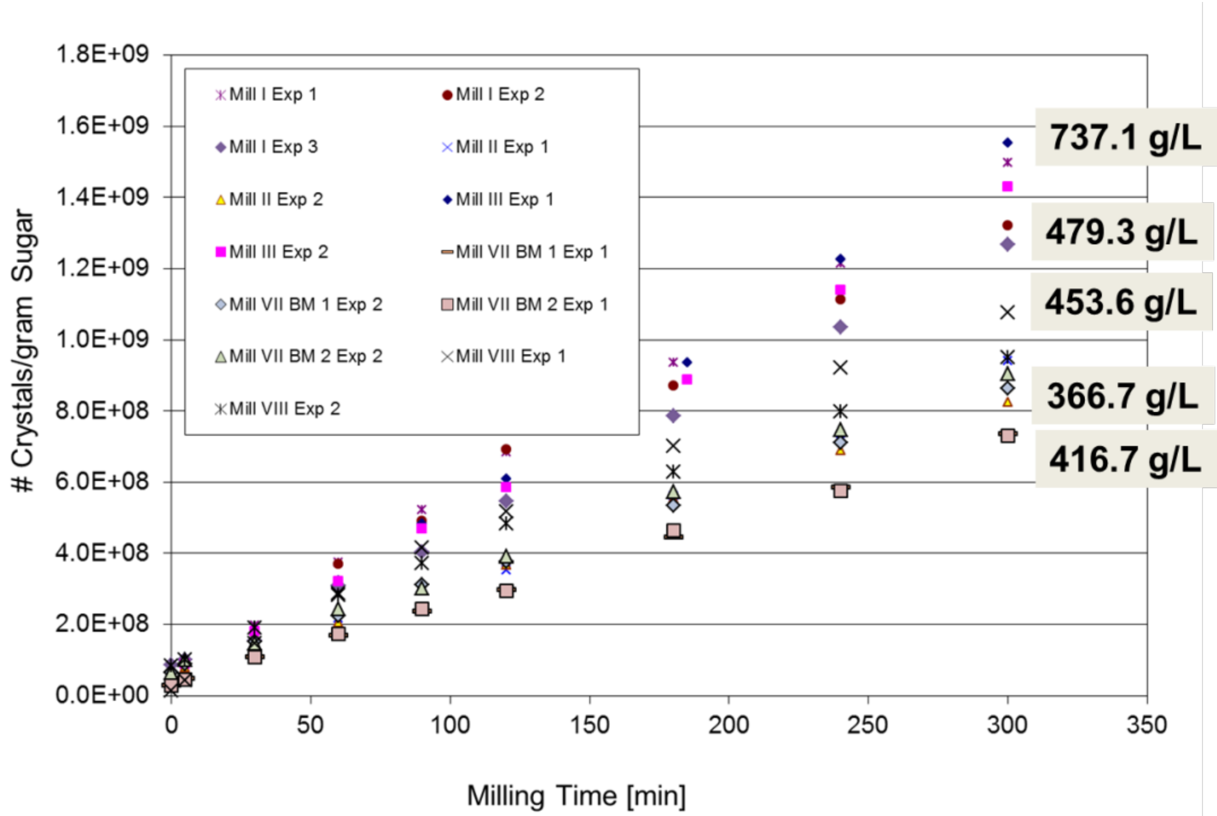


Figure 3. Milling progress of powdered sugar in ball mills in Louisiana. Sugar/IPA ratio in grey boxes

Table 3. Ball-milled seed slurry preparations (annotation I to VIII is independent of Figure 1.)

Factory	Sugar	IPA	Concentration	Comment Milling Time
Mill I	4 lbs powdered Domino 2 lbs bag	1 gal	479.3 g/L	4 hrs
Mill II	2.2 kg powdered Western Sugar	6 L	366.7 g/L	4 hrs
Mill III	6.5 lbs powdered Western Sugar	3 L+1 L	737.1 g/L	Diluted before use 4 hrs
Mill IV	500 g granulated Domino's extra fine	6 L	83.3 g/L	4 hrs
Mill V	6 lbs granulated Dixie Crystals	4 L +1 L	544.3 g/L	4 hrs
Mill VI	5 lbs granulated Dixie Crystals	6 L	378 g/L	4 hrs
Mill VII	2.5 kg powdered Western Sugar	6 L	416.7 g/L	4 hrs
Mill VIII	4 lbs powdered Milliana	4 L	453.6 g/L	4 hrs

Other observations were (results not shown here) that while ball milling is very reproducible, every ball mill is unique. The ball milling time should be at least 3-4 hours, which is current practice in Louisiana. It is possible to achieve with ball-milling granulated sugar the same slurry quality (seed crystal number) as with powdered sugar; however the milling time is 1-2 hours longer. The CV value of the seed slurry does not change much after 3 hours and typical values are between 0.6 to 0.7. Granulated sugar is easier to handle and to store than powdered sugar.

Several recommendable practices were observed and are summarized in the following: Inspecting the inside of the mill at the end and the begin of the season, including the weighing of the balls can aid in consistency. The mill should be cleaned with IPA after use and if not used for some time should be stored filled with mineral oil to minimize rust. An electronic timer control as was found in one factory is highly recommendable as the milling time affects the seed crystal number. Utilizing pre-weighed sugar aliquots in sealed plastic bags can also aid the process. The bottom valve contains a small amount of lesser-milled material. This material should be collected separately, and either be discarded or added the following slurry preparation.

ACKNOWLEDGEMENTS

We would like to thank all Louisiana sugar mills and their personnel for their support and participation in this work and the American Sugar Cane League for providing funding. We would also like to thank Mr. Praneet Karki and Dr. Amit Gautam for their help.

MULTIPLE RESPONSE OPTIMIZATION STRATEGY FOR A DOUBLE PURGE OF C-MAGMA SYSTEM INTEGRATED TO A THREE BOILING CRYSTALLIZATION SCHEME

Polanco, L.S., Day, D., Legendre, B. and Hall, S.

ABSTRACT

Raw sugar color, sugar recovery, heating requirements and pans utilization rates are operational criteria or process responses for an effective integration of a double purge of C-magma system to the traditional three-crystallization scheme. The optimization strategy involves several stages from data collection; modeling; design of experiments; simulation; surrogate modeling and finally optimization. The simpler linear surrogate models were used to approximate the sensitivity of each response to the variation of random and controlled parameters at different syrup quality scenarios. A model specific for Lula Sugar Factory created using Sugars™ and the statistical software JMP® were the tools that made possible the application of this strategy. This approach allows the evaluation of the importance of random and controlled parameters and assigning a weight to each response it gives optimal double purge system settings depending on the syrup quality input.

INTRODUCTION

Clarke (1999) stated that on the process integration of a new technology, productivity improvement has to be paired with quality improvement and, this has to be approached as a multi-factor problem considering the efficient use of resources like equipment and energy, costs and adverse conditions. The goals for the integration of a second centrifugation of C-magma (or double purge) are to estimate the product parameter combinations that reduces raw sugar whole color, maximize sugar recovery, maximize availability of pans and minimize energy consumption in relation to changes that can occur during a harvest season. Sugar crystallization is a multistage complex process where multiple factors are changing simultaneously with a long delay time (3-5 days) from syrup to final molasses. The solution is to simulate the changes on controllable factors combining actual correlations and a double purge integration model (Sugars^{TM} 2014) . The output responses of each simulation is used to evaluate major effects and to create surrogate models based on the most significant factors for each response (JMP$^{(R)}$ 2014). A singular objective maximization function is created by the application of desirability functions connecting the surrogate models and given individual importance to each response (Polanco 2015). Figure 1 summarizes the strategy approach for optimization.

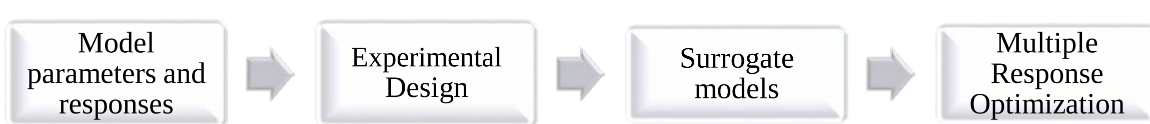


Figure 1 Strategy approach to optimize the integration of a double purge system to improve whole raw sugar color on a traditional three boiling scheme

MODEL, PARAMETERS AND RESPONSES

Historical data from Lula Sugar Factory was used to create correlations that were used together with a model (Sugars™ 2014) on simulations also, the information was used to determine fixed values and variation range for each random and controlled parameters.

The boiling house responses for the multiple-response optimization problem were:

Raw Sugar Whole Color (RSWC) = It is desirable to meet a Target ~ 1,200 CU

Sugar Recovery (RSREC) = It is desirable to maximize sugar recovery

$$RSREC=\frac{w_{sugar}*B_{sugar}*P_{sugar}}{w_{syrup}*B_{syrup}*P_{syrup}}*100 \quad \text{(Equation 1)}$$

- w : weight or mass-rate (kg/hour)
- B : Brix/100 (solids fraction)
- P : apparent purity= pol % Brix /100 (kg sugar per kg soluble solids)

Giga Joules per metric ton of syrup solids (GJ) = It is desirable to minimize heating (steam consumption)

$$GJ=\left(\frac{w_{vapor}\cdot L_P}{w_{syrup}*B_{syrup}}\right)*10^{-3} \quad \text{(Equation 2)}$$

- w : weight or mass-rate (kg/hour)
- B : Brix/100 (solids fraction)
- L_P: latent heat of vapor (P=177.2 kPa – evaporators vapor I) = 2,212.27 kJ/kg

High Grade Volume Ratio (HGV) = Installed/Required capacity ratio [I/R] for A, B and magma development strikes. It is desirable to maximize the I/R ratio

Low Grade Volume Ratio (LGV) = Installed/Required capacity ratio [I/R] for C, grain development and grain strikes. It is desirable to maximize the I/R ratio

$$HGV\ OR\ LGV=\frac{I}{R}=\frac{I}{Q_A\cdot t_A+Q_B\cdot t_B+Q_C\cdot t_C+Q_{Seed}\cdot t_{Seed}} \quad \text{(Equation 3)}$$

- Q : Volume rate out of massecuite or seed (m^3/hour)
- t : strike cycle time (hours) (Birkett 2011)

EXPERIMENTAL DESIGN

A custom experimental design (I-optimal) was used to define the number of simulations and the inputs to each simulation (JMP(R) 2014).

I-Optimal Design
Response Prediction

Low-Syrup Purity — **SP:** 81 – 82 – 83

Medium-Syrup Purity — **SP:** 84 – 85 – 86

High-Syrup Purity — **SP:** 87 – 88 – 89

RANDOM PARAMETERS	LEVELS	
SYRUP COLOR (CU):	18,000 - 23,000 - 28,000	SYCOL
SOLIDS RATE (TONS/H):	56 – 62 – 68	MTSH
% COLOR RISE A STRIKE:	1 – 2 – 3	CRA
% COLOR RISE B STRIKE:	1 – 2 – 3	CRB
% COLOR RISE C STRIKE:	15 – 20 – 25	CRC
FIRST MAGMA BRIX :	92 – 93 – 94	MB

CONTROLLED PARAMETERS	LEVELS	
FIRST MAGMA PURITY:	78 – 82 – 84	MP
DOUBLE PURGE MAGMA PURITY :	88 – 92 – 94	DPMP
DOUBLE PURGE MOLASSES RECYCLE:	A – B (0, +1)	DPRC

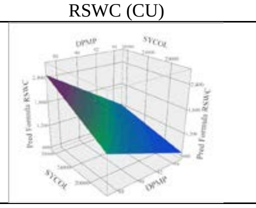
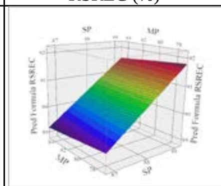
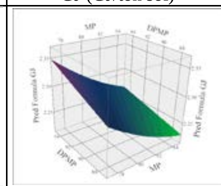
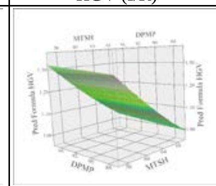
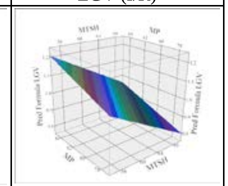
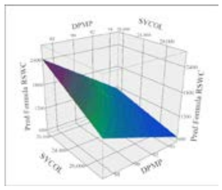
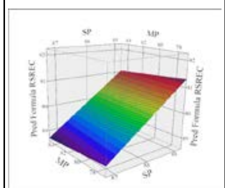
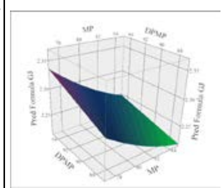
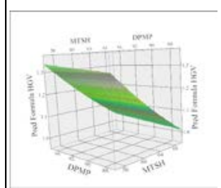
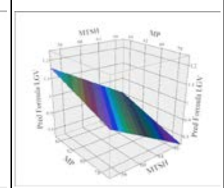
OPTIMIZATION RESPONSES	OBJECTIVE	
RAW SUGAR WHOLE COLOR:	~1,200 CU Target	RSWC
SUGAR RECOVERY:	Maximize	RSREC
HEAT REQUIREMENTS :	Minimize	GJ
HIGH GRADE VOLUME RATIO:	Maximize	HGV
LOW GRADE VOLUME RATIO:	Maximize	LGV

SURROGATE MODELS

Simulation results were evaluated using JMP multivariate analysis and model screening (SAS 2014). A first order polynomial (Equation 4) for low curvature and a second order polynomial for greater curvature are the most popular models for response surface methodology – RSM (Simpson, Peplinski et al. 2001). Table 1 shows the surface response plots at the high syrup purity scenario and the single optimization response results – individual maximum desirability.

$$y(x) = \beta_0 + \sum_{i=1}^{p} \beta_i x_i + \sum_{i=1}^{p} \beta_{i,i} x_i^2 + \sum_{i=1}^{p-1} \sum_{j=i+1}^{p} \beta_{i,j} x_i \, x_j + \varepsilon \qquad \text{(Equation 4)}$$

Table 1 Surface response plots at the high syrup purity scenario depending on the recycle of double purge molasses (DPRC). Controlled product parameters and responses at high and low syrup purity optimizing for each response (single response optimization) fixing SYCOL=20,000CU, MTSH=62 and MB=93

DPRC	RSWC (CU)	RSREC (%)	GJ (GJ/ton sol)	HGV (I/R)	LGV (I/R)
A					
B					
	DOUBLE PURGE SYSTEM – SINGLE OPTIMIZATION PARAMETERS AND RESPONSES				
LOW SYRUP PURITY (82)	DPRC: A DPMP: 88	DPRC: A MP: 78 DPMP: 88	DPRC: B MP: 85 DPMP: 87	DPRC: B MP: 84 DPMP: 94	DPRC: B MP: 84 DPMP: 94
	RSWC: 1,200 CU	RSREC: 84.9%	GJ: 2.30 (1.04 steam/solids)	HGV: 1.19	LGV: 0.72
HIGH SYRUP PURITY (88)	DPRC: A DPMP: 92	DPRC: A MP: 78 DPMP: 94	DPRC: B MP: 85 DPMP: 88	DPRC: B MP: 84 DPMP: 94	DPRC: B MP: 84 DPMP: 94
	RSWC: 1,200 CU	RSREC: 90.6%	GJ: 2.22 (1.00 steam/solids)	HGV: 1.18	LGV: 1.06

MULTIPLE-RESPONSE OPTIMIZATION

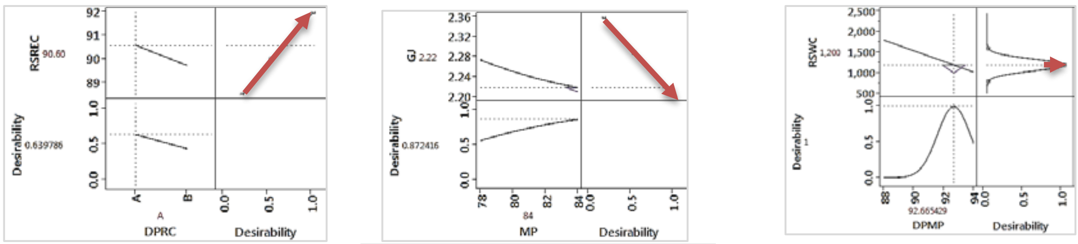
By definition, optimization is a methodology applied to find the values of a set of variables (x_1, x_2...,x_n) that makes a mathematical function – $f(x_1,x_2\ldots,x_n)$ the objective function, to reach a minimum or maximum value subject to some constraints – $g(x_1,x_2\ldots,x_n)$ (Deb 2010).

$$\min_{x_i}/\max f(x_i) \quad \text{(Equation 5)}$$

Subject to $g(x_i)\geq 0$

The approach of response surface methodologies (RSM) to multiple response optimization problems is to combine the models for selected responses into a single scalar value or cost function and solve the problem as a single objective optimization (Park and Kwang-Jae 2005; Baş, Arslan et al. 2010). The desirability function is an optimization approach which transforms a multiple response optimization problem into a single objective optimization. An individual desirability function relates the model of the response $Y_i(x)$ to a particular function $d_i(Y_i(x))$, whose solution values range between 0 and 1 (0 ≡ undesirable and 1≡ ideal response) depending on the response value of $Y_i(x)$ and the 'desired' objective of the response (Harrington 1965; Derringer and Suich 1980). The objective function (overall desirability) is obtained taking the geometric mean of all the individual desirability's and assigning a weight (w_i) according to the importance of each response. The optimization is achieved by the maximization of the overall desirability function – equation 6 (Ramsey, Stephens et al. 2005). Figure 2 shows the shape of the desirability function depending on the objective.

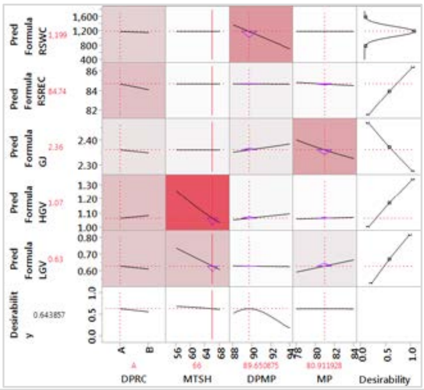
$$\max_x D^* = \max_x \exp[w_1\ln(d_1)+w_2\ln(d_2)+\cdots+w_k\ln(d_k)] \quad \text{(Equation 6)}$$



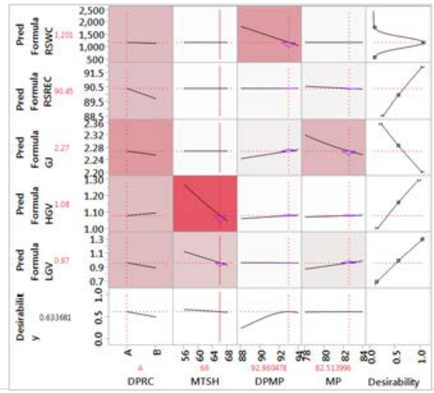
a) Desirability: Maximize | b) Desirability: Minimize | c) Desirability: Match target

Figure 2 Desirability plots a) maximize sugar recovery, b) minimize heat requirements and c) match a target value on raw sugar whole color

The number of responses used and the weight assigned to each response influenced the solution for the optimal settings of the double purge magma system. By trial and error looking for a high sugar recovery the weigh assigned to each response was: w_{RSWC} =0.20, w_{RSREC}=0.65, w_{GJ}=0.05, w_{HGV}=0.05 and w_{LGV}=0.05. Assessing parameter importance for the overall system, the recycle of double purge molasses (DPRC) has the highest total effect mainly at high syrup purity. The purity of the double purge magma (DPMP) has the higher effect for color of raw sugar while, the effect of the first magma purity (MP) is higher for heat requirements (GJ) and also for the low grade installed/required pan capacity (LGV). The random parameters syrup purity (SP) and rate of syrup solids (MTSH) have the highest overall effect on the system. The total effect of SP is very high for sugar recovery (RSREC), heat requirements (GJ) and low grade (LGV=I/R) volume ratio. MTSH is very important for the high grade and low grade (I/R) pan volume ratios. The profiles combining the five responses denotes the importance of the parameters by the intensity of the color and by the slope of the line, the triangle indicates the sensitivity of the response to the parameter and what it is the direction of the effect. Figure 3 compares optimization plots for low syrup purity and for high syrup purity. It can be seen that the importance of DPRC is higher at high syrup purity for all responses and mainly for heat requirements. The double purge system optimal parameters (maximum desirability) at low syrup purity are DPRC=A, MP=80.9 and DPMP=89.6 while that for high syrup purity DPRC=A, MP=82.5 and DPMP~93 considering same color input based on soluble solids (SYCOL=23,000 CU)



a) Low syrup purity (82) and high syrup solids rate (MTSH = 66 tons syrup solids/hour)



b) High syrup purity (88) and high syrup solids rate (MTSH = 66 tons syrup solids/hour)

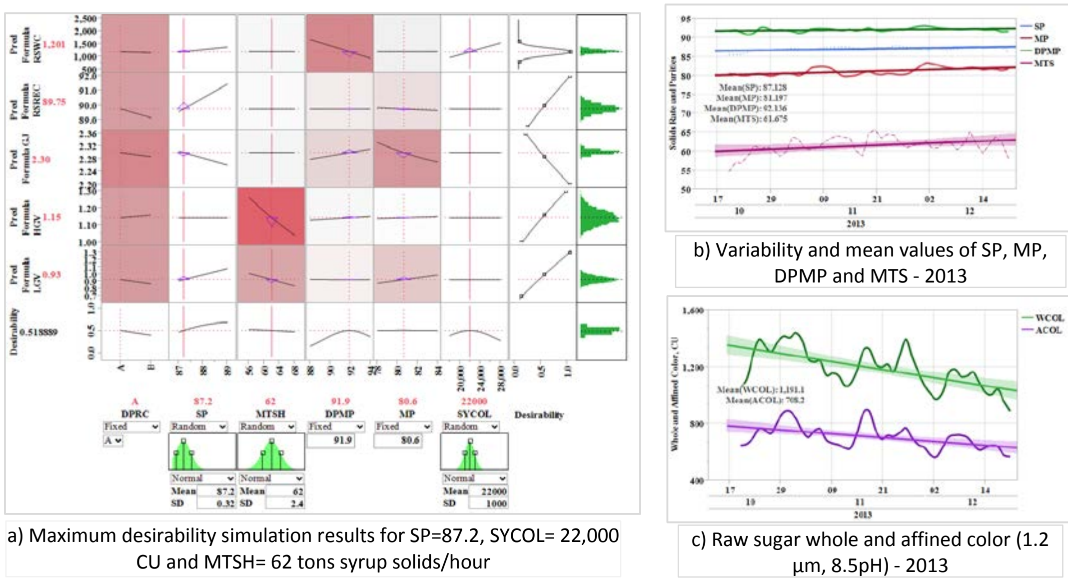
Figure 3 multiple response optimization profile plots for maximum desirability at a) SP=82 and MTSH=66 tons syrup solids/hour and b) SP=88 and MTSH=66 tons syrup solids/hour. Optimization objectives are targeting RSWC(w=0.2)=1,200 CU, maximizing RSREC(w=0.65), minimizing GJ(w=0.05), maximizing HGV (w=0.05) and maximizing LGV(w=0.05) (fixed SYCOL=23,000 CU, MB=93)

Table 2 illustrates the optimal double purge of C-magma product parameters for different syrup quality scenarios respect to purity and solids rate. The same color input based on soluble solids has more effect at high syrup purity because the ratio of the concentration of color compounds respect to the total amount of impurities (non-sucrose) is higher.

Table 2 Multiple response optimization with five responses for double purge product parameters for low syrup purity (LSP=82), medium syrup purity (MSP=85) and high syrup purity (HSP=88). Color input with syrup SYCOL=23,000 CU (fixed)

Response	**Scenario**	**Response**							
		MTSH=58				**MTSH=66**			
RSWC, CU	**LSP**	1,202				1,199			
	MSP	1,209				1,207			
	HSP	1,201				1,201			
RSREC, %	**LSP**	84.84				84.74			
	MSP	87.69				87.59			
	HSP	90.48				90.45			
GJ, gigajoules/ton solids	**LSP**	2.39				2.36			
	MSP	2.30				2.28			
	HSP	2.28				2.27			
HGV ratio	**LSP**	1.21				1.07			
	MSP	1.23				1.09			
	HSP	1.23				1.08			
LGV ratio	**LSP**	0.70				0.63			
	MSP	0.86				0.80			
	HSP	1.08				0.97			
Overall		**DPRC**	**DPMP**	**MP**	**D**	**DPRC**	**DPMP**	**MP**	**D**
	LSP	A	90	79	0.69	A	90	81	0.68
	MSP	A	91	82	0.71	A	91	84	0.66
	HSP	A	93	81	0.67	A	93	84	0.63

Figure 4 compares maximum desirability simulation results for the integration of a double purge of C-magma system to a three-boiling crystallization scheme for Lula Sugar Factory assuming same boiling house inputs according to the 2013 harvesting season.



a) Maximum desirability simulation results, b) Variability and mean values of syrup purity-SP, first magma purity-MP, double purge magma purity-DPMP and tons of syrup solids per hour-MTS, and c) Variability and mean values of whole color and affined color. Daily averages, Lula 2013

CONCLUSIONS

The primary goal of this research was to find the optimal settings to integrate a double purge of C-magma system into a three-boiling crystallization scheme in producing raw sugar, for reducing whole color over a range close to a target value. The random parameter SYCOL had a high effect on RSWC and its effect changed with the syrup purity. Low values on SYCOL will require lower purities of DPMP to produce the same color target. The multiple response optimization approach to the integration of the double purge system gave the approximate best product parameters (DPMP and MP) and the best location for the recycle of double purge molasses DPRC. The experience gained from analysis of samples, analysis of factory reports and modeling of the boiling house with actual implementation data gave the required information to define the design space and the multiple optimization objectives. The surrogate models were used to assess the importance of the selected parameters on each response. Desirability functions were used to transform the prediction (surrogate) models into uniform scale objective functions to maximize, minimize and match a target. The desirability approach offers an easy and flexible way to adjust the objective of a particular response (changing the shape of the desirability function) and to adjust the weight of the responses. This specific approach through surrogate models and application of desirability functions to perform a multiple-response optimization is novel for the sugar industry and can be improved and adapted to optimize other process stages.

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THE 2014 MOLASSES SURVEY

C. Verret

INTRODUCTION

The loss of sugar in molasses is generally the largest loss suffered by a sugar mill. It is important that reliable data on molasses exhaustion be obtained. The Audubon Sugar Institute (ASI) undertook analyses of molasses samples for the mills in Louisiana from 1980-1997. It was briefly discontinued after the 1997 season and reintroduced in 2000 and is currently providing this service to the Louisiana sugar industry.

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 2014 survey season stretched from 10-05-14 until 12-28-14. A total of 148 molasses and syrup samples and 10 juice samples were analyzed in duplicate for the 2014 season. Including standards, this totaled 525 samples for 2014. Analyses included:

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

*Because we measure sugar using HPLC, we perform a direct polarization of molasses, syrup and juice samples are clarified using Octapol™ (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 2014 season operated for 13 weeks. The 2014 season maximum TPD weekly average was 13.4 and the minimum was 7.6. Throughout the season, the TPDs demonstrated the usual trend of decreasing TPD. The Industry average TPD for 2013 was 8.6. (Figure 1)

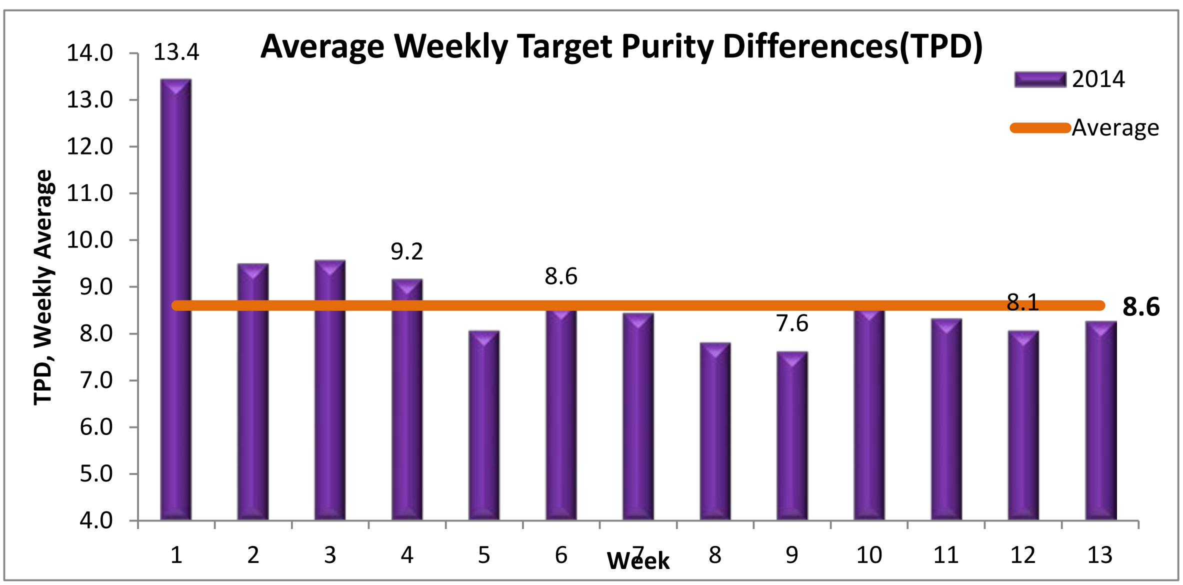

Fig. 1 – 2014 Average Weekly Target Purity Difference

The conductivity ash component for the 2014 season started at the minimum value of 13.4. As the season continued the ash increased to the maximum value of 17.0 and then decreased towards the end of the season. The conductivity ash average was 14.7. (Figure 2)

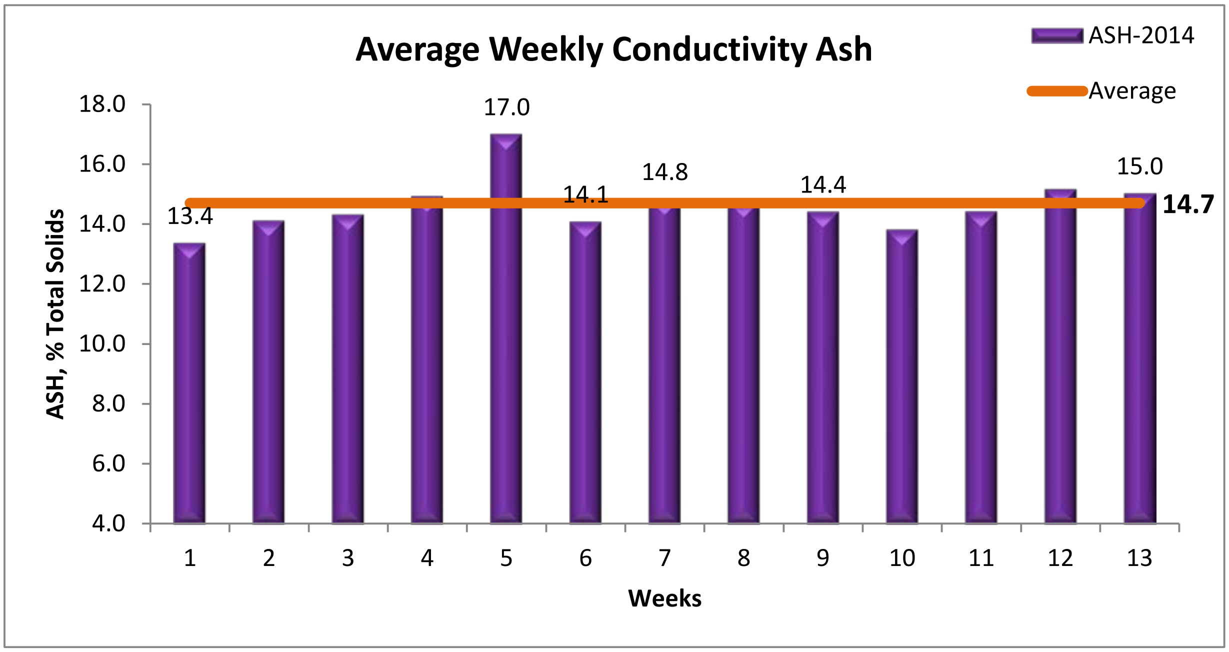

Fig. 2 – 2014 Average Weekly Conductivity Ash

In general, there has been a significant downward trend, relative to time, in the amount of reducing sugar in final molasses. In the 2014 season, the reducing sugars was up and down throughout the season. The maximum was 16.1 and the minimum occurred at the end of 13.5. (Figure 3)

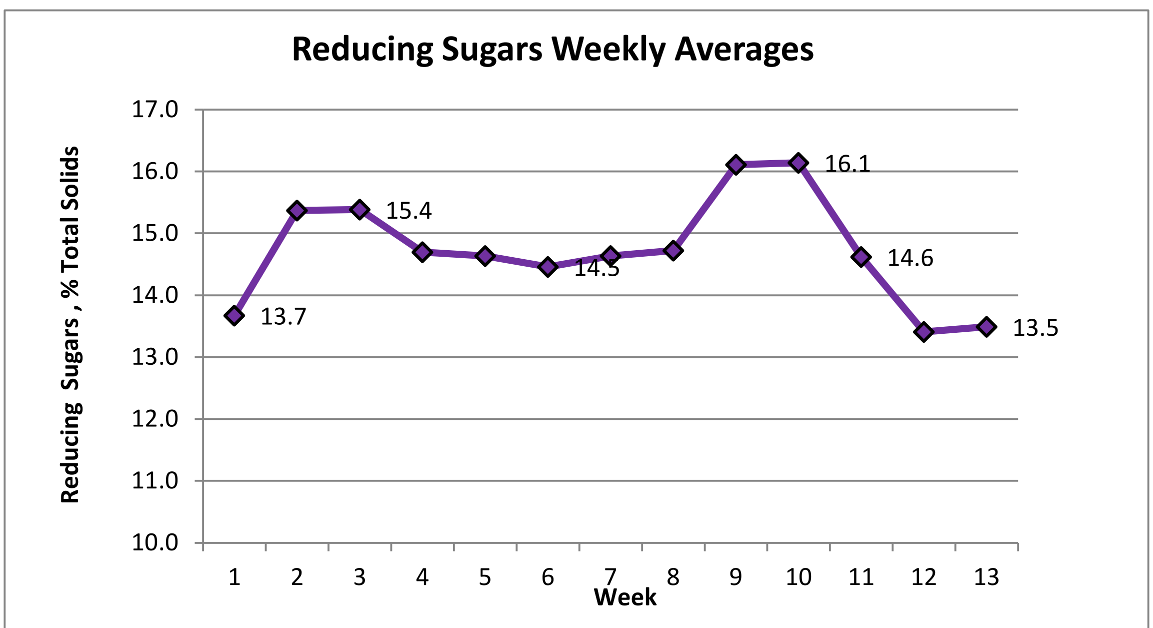

Fig. 3 –2014 Reducing Sugars Weekly Averages

Comparing the results from the 2014 season to the results from the 2011, 2012 and 3013 seasons showed the yearly average TPD increased. This is demonstrated in **Figure 4.** The 2014 season maximum TPD was 10.1. The minimum TPD for 2014 was 6.6. (Table 1)

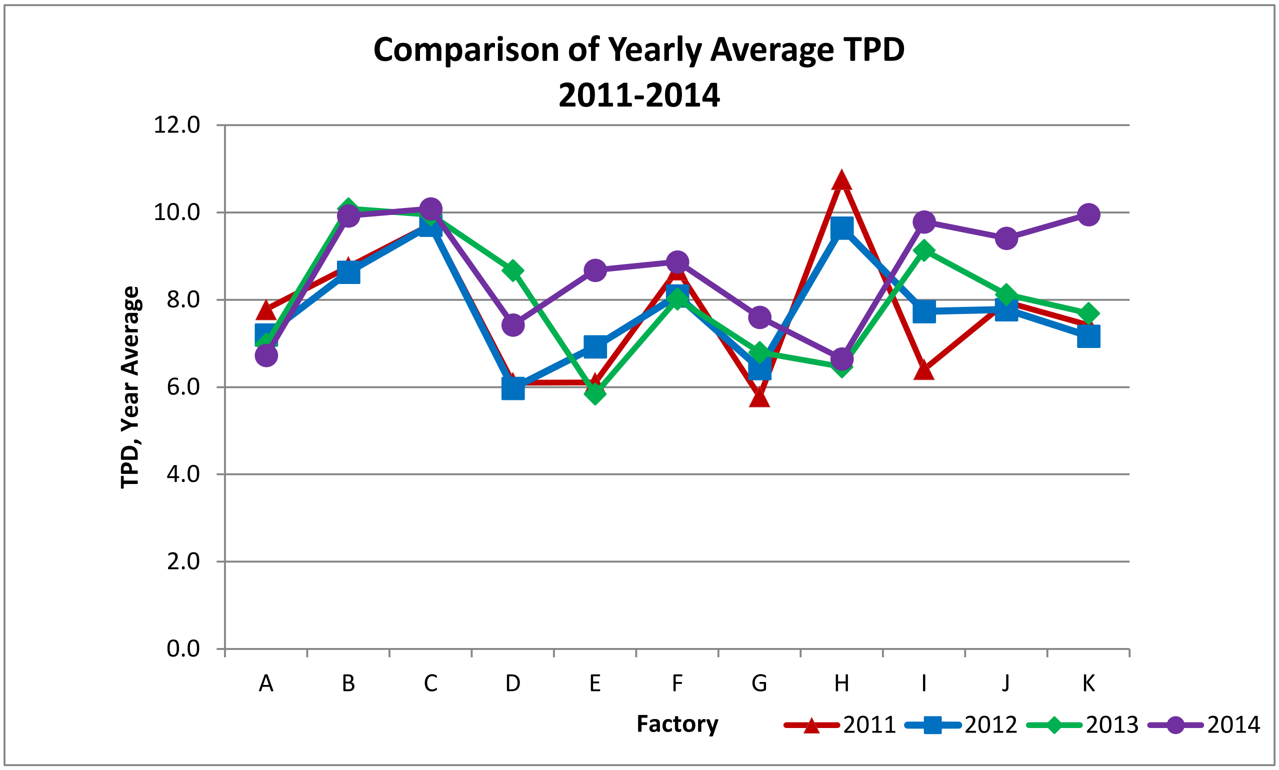

Fig. 4 – Comparison of Yearly Averages TPD 2011-2014

TPD Data Summary for 2011-2014			
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
2014	6.6	10.1	8.6

Table 1 – Summary of Yearly TPD 2011-2014

The sugar cane juice from the sugar mill has been analyzed for the past 5 years. In **Table 2**, shows the summary from the analysis. Over the last five seasons, the average brix is 14.6, a true purity average of 88.9% and reducing sugars 5.4%.

Juice Survey Summary for 2010-2014			
Year	**Ref. Brix (%Juice)**	**True Purity (% Juice)**	**Reducing Sugars (%Juice)**
2010	14.5	89.3	3.8
2011	15.1	87.2	5.0
2012	14.6	88.1	3.6
2013	14.1	91.1	10.8
2014	14.8	88.7	4.1
Average	**14.6**	**88.9**	**5.4**

Table 2 – Summary of Juice Survey 2010-2014

CONCLUSIONS

The seasonal average TPD was 8.6 for the 2014 season. The ash decrease slightly for the 2014 season to 14.7%. The reducing sugars increased for the 2014 season to 12.5% from 14.9% 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.

For the 2014 season, the juice had an average brix of 14.8%, a true purity of 88.7% and reducing sugars of 4.1%.

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COLOR CHANGES THROUGH THE FACTORY

Harold Birkett and Jeanie Stein

In recent years the sugar refineries have been demanding higher pol sugar of low color. Most of the factories have modified their sugar boiling schemes to produce sugar of the required 99.2 pol. Several factories have had difficulty in producing sugar of sufficiently low color to obtain the attractive color premiums after an extended storage period.

The usual way of reporting color is on a solids basis. However, as sucrose has no color, a more meaningful way to investigate color increases in the process is to report the color of the impurities (i.e. non-pol). The color on a non-pol basis can be calculated from the usual color on a solids basis by dividing this color by the purity of the material. Figure 1 shows the state average analysis for mixed juice and sugar in 2014 for pol, impurities and water content.

During the 2014 crop, a limited number of samples were collected at the factories to investigate the level of color in the juice entering the factories and to determine how much color was formed through the processing operations. Initially, samples of mixed juice, clarified juice, syrup, raw sugar and final molasses were obtained for analysis. These preliminary analyses indicated that most of the color was generated after the syrup stage. Figure 2 shows these colors for one factory on a solids basis while Figure 3 presents the same data on a non-pol solids basis.

Since the above indicated that most of the color is formed after the syrup stage, additional samples of the intermediate process products after the syrup stage were collected for analysis including the A molasses, B molasses, B sugar, and C sugar as well as filtrate juice from the filters. For a typical factory, the color of all major streams are shown on a solids basis in Figure 4 and on a non-pol solids basis in Figure 5. The average data for all factories is shown in Figure 6 on a solids basis and in Figure 7 on a non-pol solids basis.

Mixed juice color at all factories tested is shown in Figure 8 on a solids basis and in Figure 9 on a non-pol basis. Similarly, sugar color is shown in Figure 10 (solids basis) and in Figure 11 (non-pol solids basis). Figure 12 shows both mixed juice and sugar color on a non-pol solids basis.

Figures 13, 14 and 15 show the color of the A, B and C sugars together with the corresponding molasses on a non-pol solids basis.

Preliminary results indicate:

1. The color of the non-pol solids (impurities) in the mixed juice entering the factory varied from 120,000 to 240,000 and averaged 170,000.
2. The color of the non-pol solids in the sugar varied from 110,000 to 500,000 and averaged 290,000. This indicates that color increased on average about 70% through the processing operations.
3. The color of the non-pol solids in the A sugar and A molasses were similar. Similarly, the color of the non-pol solids in the B sugar and B molasses were comparable, as was the color of the non-pol solids in the C sugar and final molasses.
4. The filtrate juice typically had the lowest color non-pol solids.

FIG. 1 TYPICAL SOLIDS AND NON-POL SOLIDS

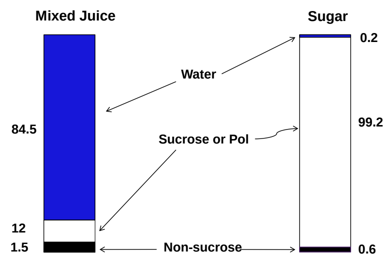


FIG. 2 COLOR ON SOLIDS

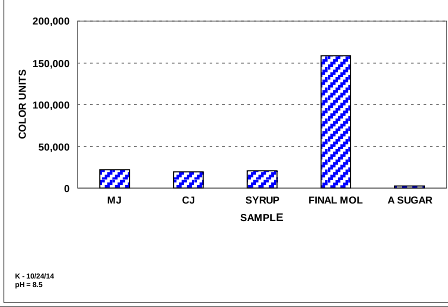


FIG. 3 COLOR ON NON-POL SOLIDS

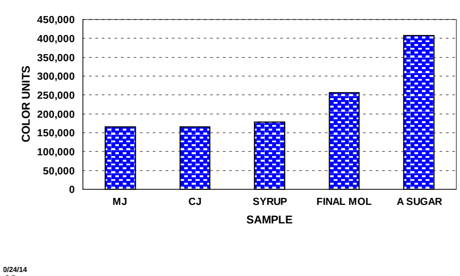


FIG. 4 COLOR ON SOLIDS

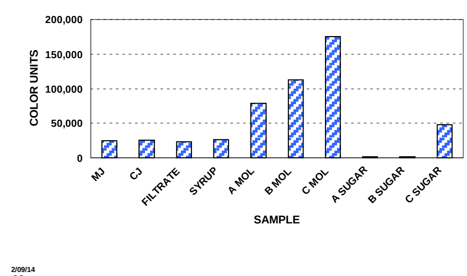
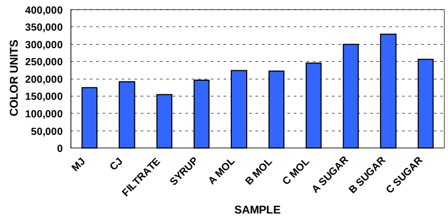
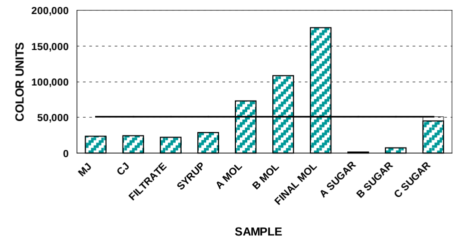


FIG. 5 COLOR ON NON-POL SOLIDS



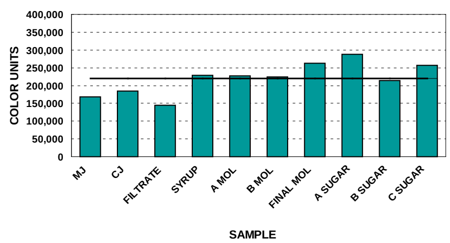
2/09/14
8.5

FIG. 6 AVERAGE COLOR ON SOLIDS



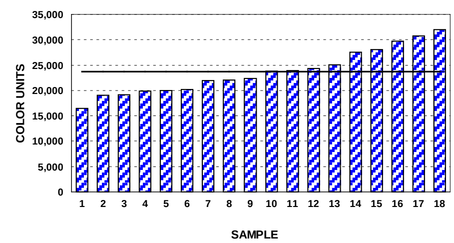
erage = 51,105

FIG. 7 AVERAGE COLOR ON NON-POL SOLIDS



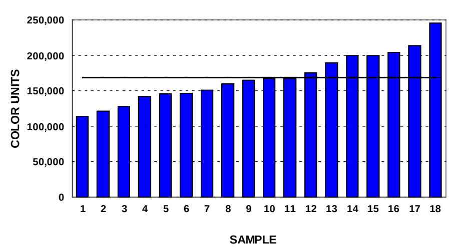
verage = 219,548

FIG. 8 COLOR ON MIXED JUICE SOLIDS



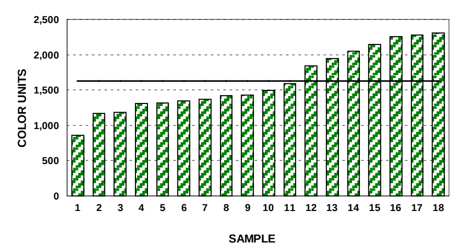
verage = 23,679

FIG. 9 COLOR ON MIXED JUICE NON-POL SOLIDS



verage = 168,646

FIG. 10 COLOR ON SUGAR SOLIDS



verage = 1,629

FIG. 11 COLOR ON SUGAR NON-POL SOLIDS

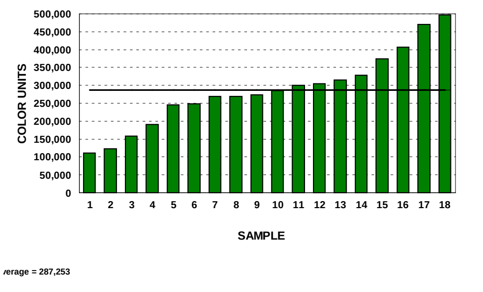


FIG. 12 COLOR ON NON-POL SOLIDS: MIXED JUICE AND SUGAR

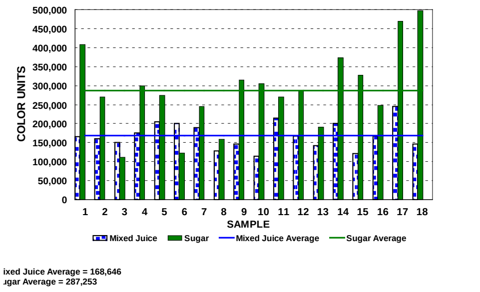


FIG. 13 COLOR ON NON-POL SOLIDS: A MOLASSES AND A SUGAR

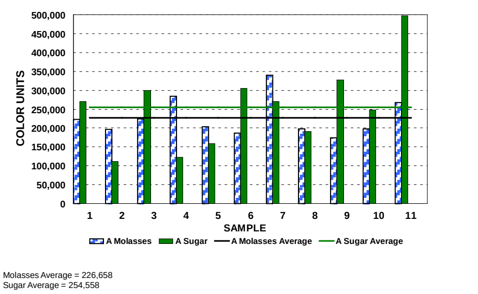


FIG. 14 COLOR ON NON-POL SOLIDS: B MOLASSES AND B SUGAR

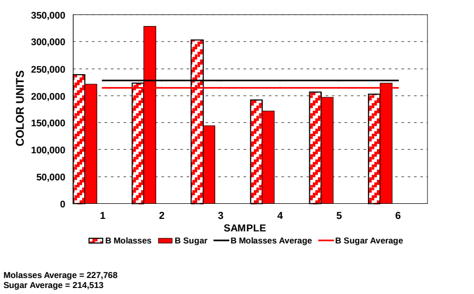
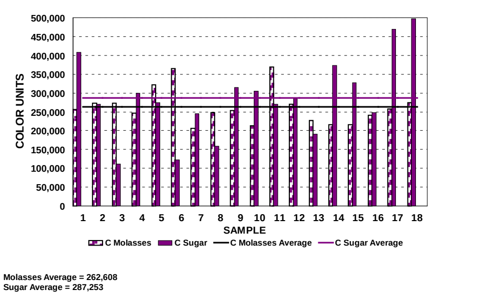


FIG. 15 COLOR ON NON-POL SOLIDS: FINAL MOLASSES AND C SUGAR



DEASHING OF SUGAR SOLUTIONS USING ELECTRODIALYSIS

*Amit K. Gautam, Stephanie Linares, Franz S. Ehrenhauser**

INTRODUCTION

Biomass feedstocks such as energy cane and sweet sorghum contain high concentrations of inorganic ions (ash). It is essential to remove these inorganic ions prior to catalytic bioforming processes to minimize catalyst deactivation. The goal of this work is to achieve a 0.05% ash level in energy cane and sweet sorghum syrups to meet ash levels as they are found in corn-derived glucose syrups.

Ion exchange is commonly used for deashing of syrups. Ions are adsorbed on solid resins which are periodically regenerated with caustic soda and hydrochloric acid. Chemical requirements and waste generation due to intermittent washing are fairly high. Electrodialysis is an alternative technology for deashing which has fewer chemical requirements compared to ion exchange.

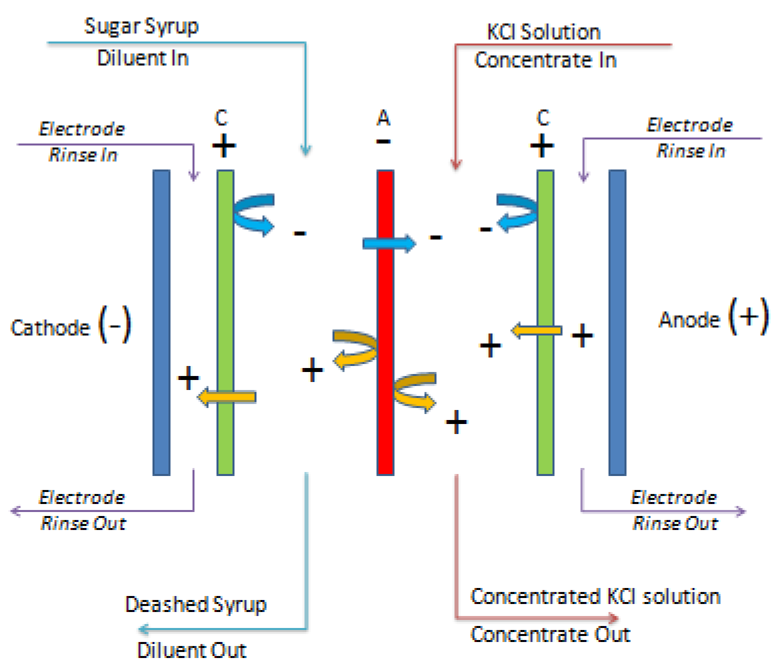


Figure 1. Electrodialysis cell schematically

Electrodialysis has found applications for desalination in the energy, food and sugar industry. Mitsui sugar industry (Japan) and Elmidaoui et al. (Morocco) have previously reported on the demineralization of cane sugar juice/ syrup using electrodialysis [1, 2].

As shown in Figure 1, an electrodialysis cell contains a positive (anode) and a negative electrode (cathode). Between the electrodes an alternate arrangement of cation (C) and anion (A) exchange membranes with spacers separates three individual interconnected chambers and flows – diluent, concentrate and electrode rinse. Under the influence of an electric field, positive ions (cations) move to the negative and negative ions (anions) move to the positive electrode. As the C and A membranes allow the passage of only positive and negative ions respectively, one chamber depletes in ions (diluent), whereas the other concentrates the ions (concentrate). The electrode rinse is a solution of high conductivity which provides for high efficient current transfer and minimizes undesired electrode reactions. For the practical application sugar syrup would be pumped through "diluent in" into the diluent chamber of the electrodialysis cell, and due to the applied electric field ions move from the syrup through membranes to the concentrate chamber. As a result, deashed syrup is recovered from diluent chamber. Similarly, the dissolved ash (predominantly potassium chloride) is recovered from the concentrate chamber.

In this work, three ion exchange membrane pairs from different manufacturers were evaluated for their potential of removing potassium chloride from pure salt and salt/sugar solutions. The best membrane pair was evaluated for its application for the deashing of energy cane syrup.

MATERIAL & METHODS

Electrodialysis Cell (ED Cell):

A lab-scale electrodialysis cell FT-EDR-40-2-x was purchased from Fumatech GmbH (Bietigheim, Germany). The three-chamber cell consists of two titanium-iridium plasma coated stainless steel electrodes, PP endplates and 22 PVC spacers providing a housing for up to ten membrane pairs. The membrane effective area per cell is 36 cm^2. Electric current was supplied by an Extech DCP60 600 W, 120 V adjustable DC power supply. Three different ion exchange membrane pairs (anion/cation) AMI-7001/CMI-7000, FAS/FKS and AMX/CMX were purchased from Membrane International Inc., Fumatech and Ameridia, respectively.

Flow to the individual chambers were provided by peristaltic pumps (Coleparmer MODEL77250-62 with L/S 35 silicone tubing), whereas the concentrate and the diluent chamber were operated at 1 L/min and the electrode rinse at 0.8 L/min. The diluent and the concentrate solutions were kept at 40°C by keeping their reservoir on a temperature controlled hot plate. 0.2 M K_2SO_4 was used as electrode rinse solution. All experiments were conducted in batch mode, i.e. the solutions were recycled through their reservoirs.

pH and conductivity were monitored with an Oakton PC700 pH/conductivity meter in the respective reservoirs. A Buck Scientific flame photometer (East Norwalk, Connecticut, USA) was used for the analysis of K^+, Na^+, Ca^{2+} in all three streams. Concentrations were determined by comparing sample measured emission against a linear calibration curve prepared from standards of 2 to 24 mg/L of K^+, Na^+ and Ca^{2+} respectively. Sucrose, glucose and fructose concentrations were determined by HPLC Analysis on an Agilent 1200 system with a Biorad AMINEX HX-87K column and an RI detector. Deionized water was used as mobile phase at 0.6 mL/min and 85°C column temperature with a sample injection volume of 20 µl.

Potassium chloride (99%, ACS grade) and potassium sulfate (99%, ACS grade) were purchased from Alfa Aesar and used without further purification. Sucrose was purchased as granulated cane sugar from Walmart.

The tested energy cane syrup was obtained from the pilot facilities at Audubon Sugar Institute. Energy Cane Ho 02-113 was single-pass milled in a three-roll Farrell mill, screened to 300 µm, cold-limed with milk-of-lime to pH 7.5 and clarified with 5 ppm anionic flocculant (Garrat Callahan 7622) in a LLT short-residence time clarifier. The clarified juice was concentrated in a single effect evaporater to 64.8°Bx.

EXPERIMENTS

All electrodialysis experiments were carried out at 1 A of constant current. Two sets of experiments were conducted for the evaluation of the individual membrane pair performance. The first set of experiments utilized a 10% potassium chloride solution in both the dilute and concentrate streams. The second set used a solution which contained 10% potassium chloride and 20% sucrose in the dilute stream and 10% potassium chloride solution in the concentrate stream.

For the deashing of the energy cane syrup a 10% potassium chloride solution was used as concentrate while the syrup was diluted to 30 °Bx prior to use as diluent.

RESULTS AND DISCUSSION

Membrane pair evaluation

Table 1. Performance evaluation of ion exchange membrane pairs

Membrane Pair	Ash removal [equ/hr]	Cumulative energy requirement [kWh/equ KCl]	Water loss in diluent chamber [Vol%]	Sugar loss [g fermentable sugars / equ KCl]
10% KCl				
FAS/FKS	0.40	0.012	36.7	n.a.
AMX/CMX	0.36	0.013	20.0	n.a.
AMI/CMI	0.31	0.033	23.3	n.a.
10% KCl/20% sucrose				
FAS/FKS	0.44	0.012	69.2	154.31
AMX/CMX	0.38	0.013	13.8	1.82
AMI/CMI	0.26	0.045	19.2	1.75

Table 1 shows the performance parameters for the tested membrane pairs. While high ash removal is desirable, low energy requirement and low water loss (due to water splitting) are also desirable. For the first set the AMC/CMX and the FAS/FKS pair perform comparably well, with FAS/FKS having both the lowest energy requirement and highest ash removal rate. The rather high water loss of the FAS/FKS membrane is here not a major issue as the removal of water out of the syrup due to electrodialysis is potential positive side effect (though the evaporation with steam is certainly cost-preferred).

The addition of sucrose to the concentrate does not change the ash removal or the energy requirement significantly. However, the FAS/FKS membrane pair exhibits unacceptable sugar losses and it seems the addition of sucrose increases the movement of water out of the diluent chamber. As such the utilization of the AMX/CMX was chose for further experimentation as sugar losses and energy requirements are low.

Deashing could be employed either at the begin of processing, where it would reduce scaling in heat exchange equipment and reduce steam requirement for evaporation or after evaporation where temperature, ash concentration and the volumetric flow are favorable for electrodialysis. As such unclarified raw energy cane juice of 11.3 ^{o}Bx was subjected to electrodialysis. Unfortunately, no deashing was possible as the charged colloidal and suspended solids fouled the membranes immediately. Therefore only clarified juice and syrup are suitable for electrodialysis.

Energy cane syrup of 30^{o}Bx was subjected to electrodialysis with 10% potassium chloride as initial concentrate solution. Figure 2 shows the deashing progress and the energy requirement over time. Clearly electrodialysis works well to remove ash (72% removal) from clarified juices/syrups; however, there is an energy limit due to the limited conductivity in the diluent due to ash removal. Furthermore, a deposit of aconitate rich scale was found limiting continuous use of electrodialysis. As the deposit was easily acid soluble and did not impact the membrane permanently this limitation can be handled with proper operation (occasional acid wash). The pH level stayed constant during operation, which is a significant advantage over ion exchange, where large pH changes are common.

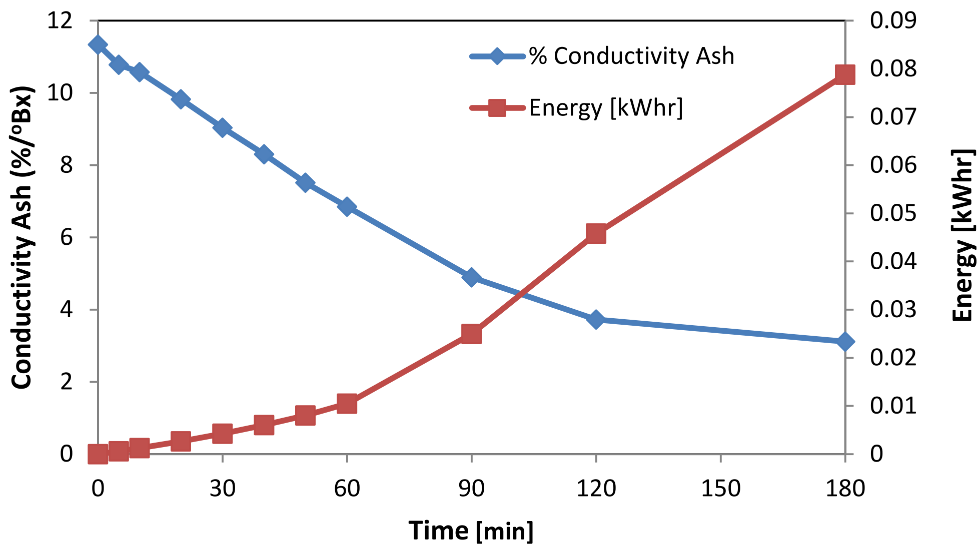


Figure 2: Ash level and energy consumption during deashing of energy cane syrup.
As there is no processing technology without sugar loss, this is neither the case forelectrodialysis. However, the fermentable sugar losses of only 0.16% during the electrodialysis are excellent and compare very favorably with losses encountered during ion exchange. The energy requirement of 0.0417 kWhr/equ (4.74E-06 $/ equ increasing to 0.01 $/ equ) is very small and represents a fraction of the cost required for the chemicals used in ion exchange (0.0259$/ equ). As such electrodialysis presents itself as a superior technology for deashing at high levels of ash. For deashing to the aforementioned low levels of ash a combination of electrodialysis and ion exchange is suggested due to the exponential increase of energy requirement of electrodialysis at low levels of ash.

CONCLUSION AND OUTLOOK

Electrodialysis was successfully tested for the deashing of energy cane syrup. Electrodialysis promises vastly superior operating cost than ion exchange for the deashing of syrups with high level of ash due to reduced operating cost and as there is no pretreatment necessary. While scale formation has been observed, its impact on operation is anticipated to be manageable. Further research addressing these potential shortcomings is ongoing. Electrodialysis is not expected to be able to lower the ash level to the desired 0.05% due the conductivity limitations at this low ash level. Nonetheless, electrodialysis seems to be the ideal complementary methodology to reduce the ash level to reasonable levels for the application of ion exchange to achieve the desired low-ash syrups for biorefineries. Electrodialysis also holds potential for the raw sugar factory as the operation was accomplished without pH change, allowing for inversion-free deashing.

ACKNOWLEDGEMENTS

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FERMENTABLE SUGAR FEEDSTOCKS FROM LOUISIANA

Donal F. Day

Audubon Sugar Institute

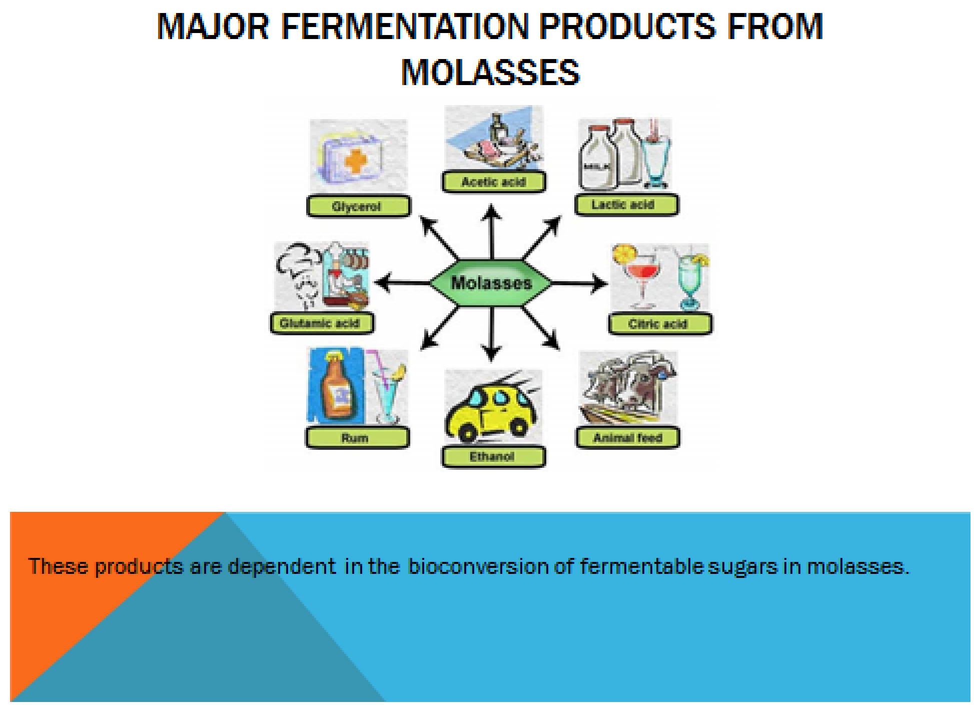
Fermentable sugars are defined as those simple sugars that are readily utilized by microorganisms for producing fermented products. Generally, the term is restricted to the sugars glucose, fructose and sucrose.

SOURCES OF FERMENTABLE SUGARS

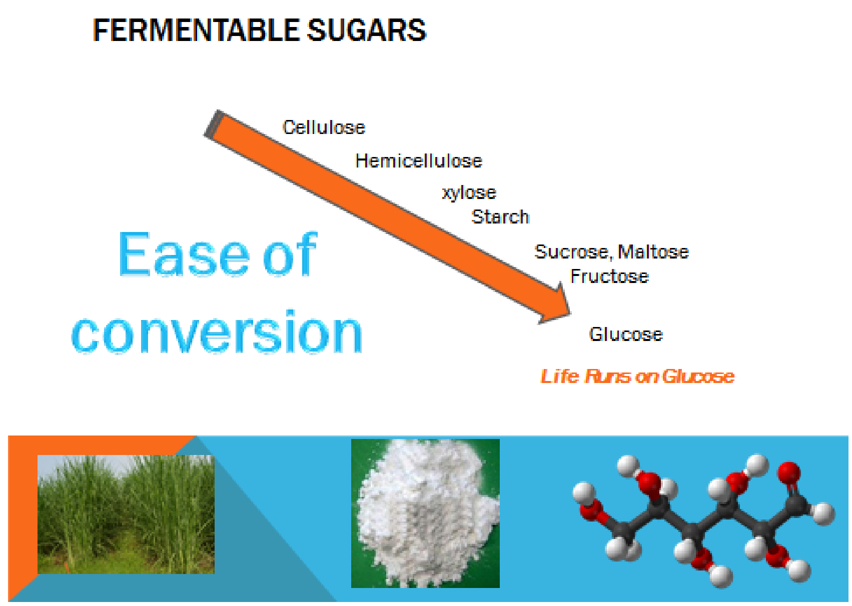
Sugar containing crops	Industrial sources
Sugarcane	Molasses (cane)
Sugar beet	Molasses (beet)
energycane	syrups
sweet sorghum	syrups
Starch converted to sugars	
Corn	DE syrups (corn)
Future sources?	
Lignocellulose	

The ultimate source for all sugars is plant photosynthesis. Expansion in this area has led to Fermentable sugars are commonly produced from plant sources. The largest industrial source of fermentable sugars is hydrolyzed corn starch, followed by molasses from either cane or beet. Like other sugar products, the value of these sugars is a function of the market price (as reflected in molasses prices) and the "cleanliness of the product".

There are a large number of major commodities that are produced by fermentation from molasses. The products range from animal feeds to fine chemicals. More recently, the growth market has been ethanol fuels. Expansion of the ethanol market raised the price of corn, but also tied it the fluctuations in the fuel market. The push by the government for greater levels of "green fuel" increased the need for greater sources of fermentable sugars and has driven much of the effort to derive fermentable sugars from lignocellulose.

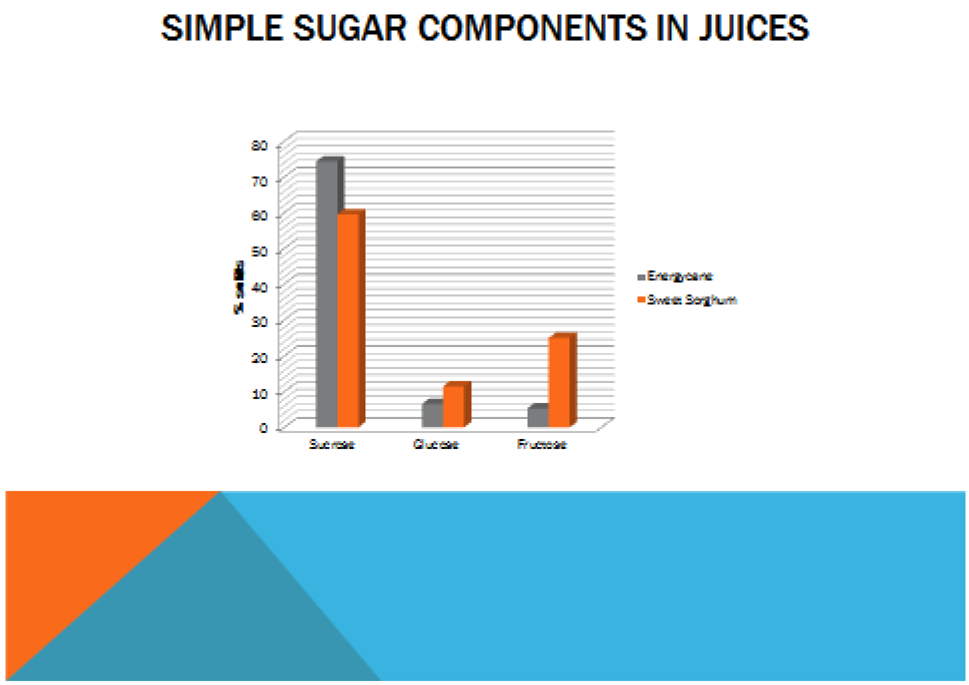


There has been a lot work conducted on the use of lignocellulosic sugars, but the primary value of any complex sugar lies in its ease of conversion to a monosaccharide, and the most favored monosaccharide is glucose.



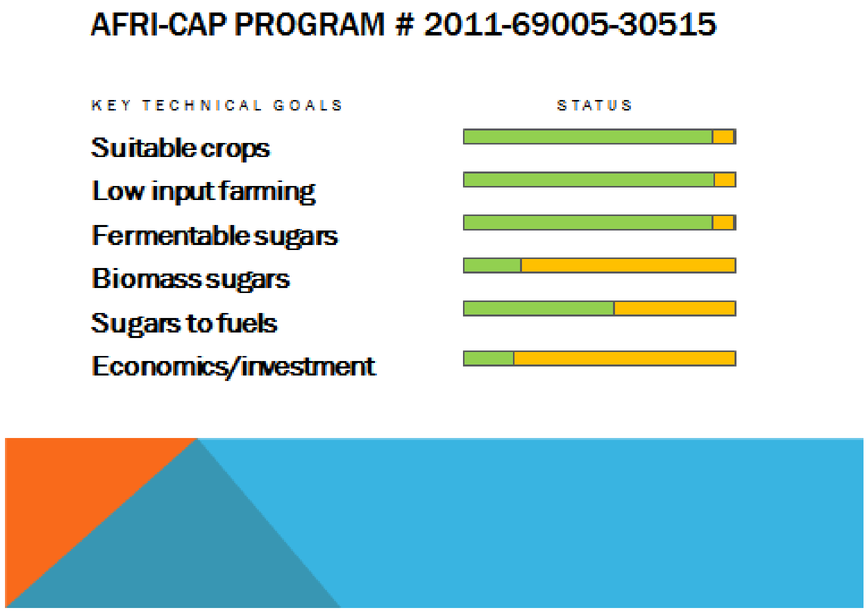
Glucose is in fact the primary fuel for all life.

As you are aware we have been working for 4 years on the potential for two crops to be producers of fermentable sugars. These crops are energycane and sweet sorghum. Besides being biomass sources each of these crops produce a sugar containing juice, much as does sugarcane.



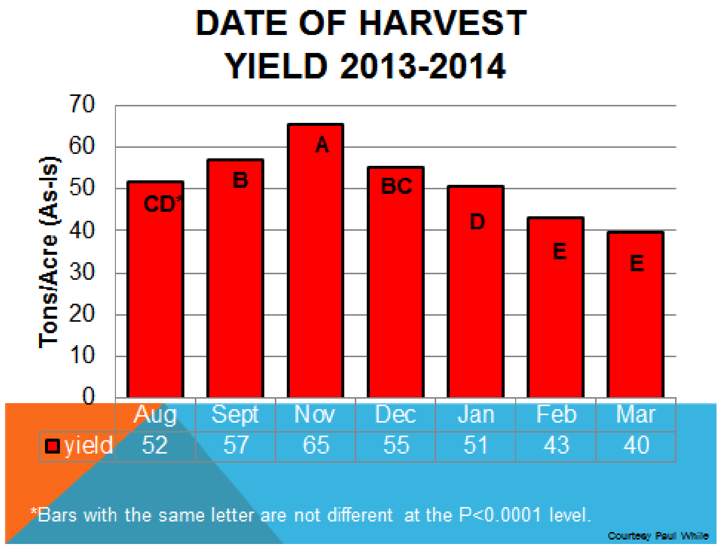
In composition, energycane juice looks like commercial sugarcane juice (but on a tonnage basis there is much less of it than in commercial sugarcane) while the sugars in sweet sorghum juices show higher invert levels that the juices of energycane.

The AFRI-Cap program called for technical developments in several major areas, one of which is production of fermentable sugars. We have made significant progress in several of these areas shown below, but I will focus today only on the production of fermentable sugars.



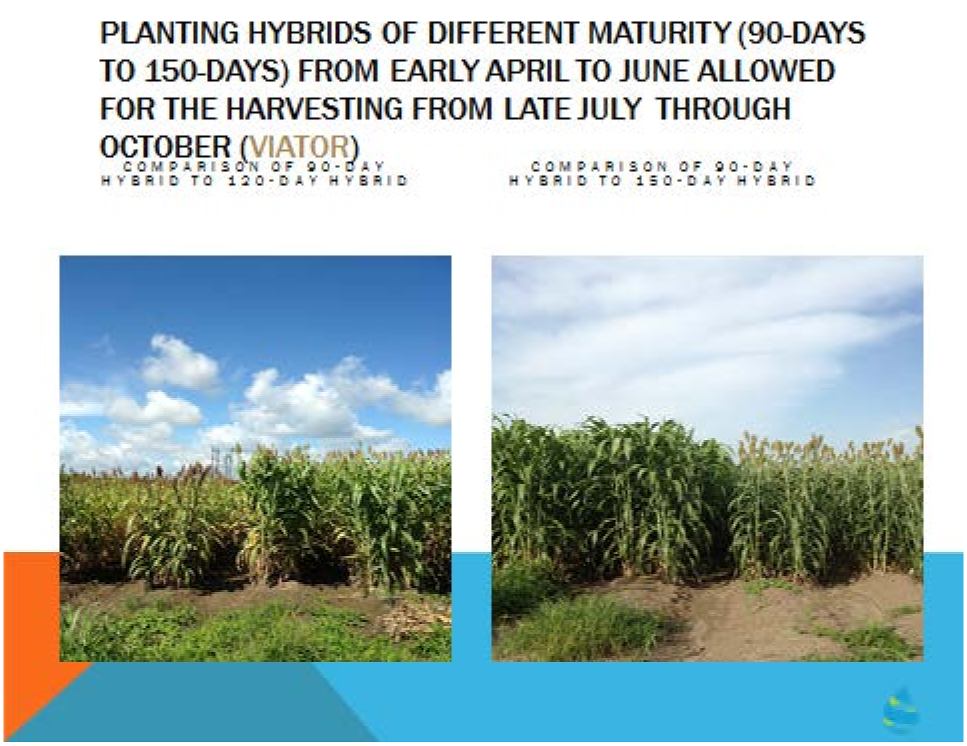
We chose to develop two crops, energycane and sweet sorghum, because they could be grown, harvested and processed in a manner similar to sugarcane. A constraint was that these crops should interfere with existing sugar production. Energycane was targeted for production in the Northern part of the State. An area that is not traditional for sugarcane and sweet sorghum ican be grown across a wide swath of the United States.

The picture above shows a stand of energycane in North Louisiana. The determination that energycane can be grown well north of the traditional sugarcane belt in Louisiana was critical to establishing its potential as a biomass/fermentable sugar crop. The figure below shows the yields achieved in North Louisiana, for four different energy cane varieties, harvested at different times of the year.



The bottom line for the use of this crop is that significant tonnage can be harvested over the designated time period. It is important to note that significant tonnage per acre was still available through February.

The second crop under consideration is sweet sorghum. This is an annual, short rotation crop, that also produces fermentable sugar containing juices. A series of sweet sorghum hybrids that mature at different rates were tested to determine how much the harvest time could be extended.



The tonnage yields of sweet sorghum were significantly less that for that of energycane. Between 1 and 9 dry tons of biomass per acre could be produced, with the highest levels found with the longer growing hybrids.

HARVESTING INITIATED AT HARD-DOUGH STAGE

Dry-weight biomass yield ranged from approximately 1.0 ton/A to > 9.0 tons/A and fermentable sugar yields have ranged from < 1.0 tons/A to > 3 tons/A.... Considerably less yield in years 2 and 3, which may be due to N leaching and cool/wet springs.

A simple comparison of the two crops (shown below) shows somewhat more fermentable (simple) sugars are produced per ton with sweet sorghum but the tonnage is considerably higher with energycane.

CROP CHOICES (APPROXIMATE SUGARS YIELDS)

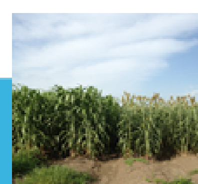
Energycane

Wet ton/acre	51
lbs simple Sugar/ton	123.6
lbs complex Sugar/ton	362.3
Total lbs Sugar/acre	12,633

Sweet Sorghum

Wet ton/acre	20
lbs simple Sugar/ton	184.1
lbs complex Sugar/ton	186.4
Total lbs Sugar/acre	7,410

Meets EPA requirement for RFS



Does not meet EPA requirement

Applying the approximate value of sugars in molasses ($0.08/lb) to these numbers gives a estimate of the value of the crop per acre. As biomass also has a value, an estimate of $65/dry tone was applied.

ESTIMATED VALUE OF FERMENTABLE SUGARS FROM CROPS

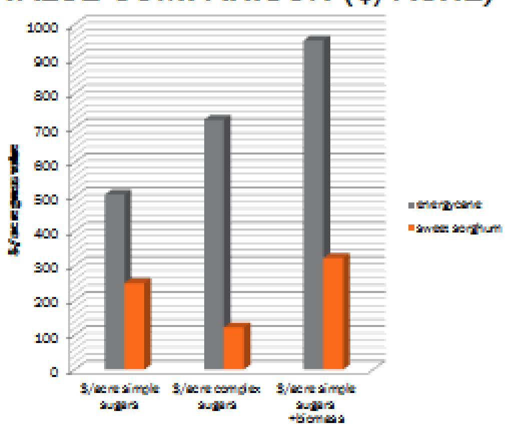
Crop	Lbs FS/wet ton	$ @ 0.08/lb	Lbs dry biomass/ton	$@$65/ton
Energycane	124	9.92	270	8.77
Sweet sorghum	184	14.72	130	4.23
	tons/acre	FS $/acre	Biomass $/acre	Gross value/acre est.
Energycane	51	505.92	447.27 922.64*	953.19 1427.91*
Sweet sorghum	17	250.24	71.91 121.92*	322.15 372.18*

*value of complex sugars in biomass

The approximate value that might be achieved if complex sugars could be economically converted to simple sugars is given in red. It is very obvious that the ultimate success of these crops as fermentable sugar sources will be dependent on the selling price for the sugars, which in turn depends on an increasing demand.

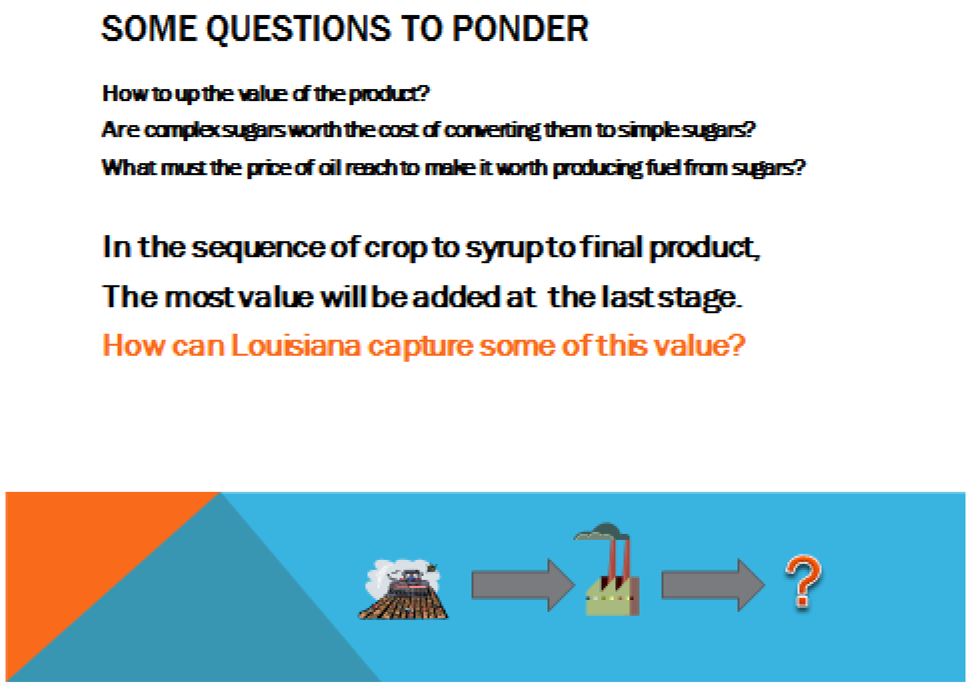
As a comparison, energycane probably holds of more value per acre than sorghum.

GROSS VALUE COMPARISON ($/ACRE)



Value calculated at $0.08/lb fermentable sugars)

This work leaves a number of questions which potential producers of such products must answer.



Special thanks to Paul White (USDA-Houma) and Sonny Viator (LSU AgCenter) for the use of their slides.

SYRUP PRODUCTION FROM ENZYME HYDROLYSATES DERIVED FROM DILUTE AMMONIA PRETREATED SWEET SORGHUM AND ENERGYCANE BAGASSE

Patrisha J. Pham-Bugayong and Giovanna M. Aita

INTRODUCTION

Pretreatment and conversion of sweet sorghum and energycane bagasse into green fuels and chemicals have been extensively studied in Dr. Aita's laboratory[1-10]. Dilute ammonia (DA) pretreatment removed 44% of the original lignin and 35% of the original hemicellulose while retaining 90% of the cellulose for sweet sorghum [9], meanwhile, 55% lignin, 30% hemicellulose and 9% cellulose were removed during the DA pretreatment of energycane bagasse[5]. Pretreated sweet sorghum bagasse (PSSB) and energycane bagasse (PECB) were then enzymatically hydrolyzed. 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 sweet sorghum and energycane bagasse enzymatic hydrolysate was initially accomplished. We have reported the presence of monomeric sugars (glucose, xylose, arabinose, and mannose), organic acids, furaldehydes, and phenolic acids in these enzyme hydrolysates. These groups are monitored because of their known importance and inhibitory effects. In this work, the characterized enzymatic hydrolysates derived from DA pretreated biomass were detoxified using various detoxification methods (i.e. solid phase extraction, ionic liquid, ion exchange resin, and activated charcoal) prior to syrup conversion (Figure 1). The mixed sugars syrup (non-food grade) that can be generated from sorghum and energycane enzymatic hydrolysates represents a potential feedstock for the production of green fuels and chemicals.

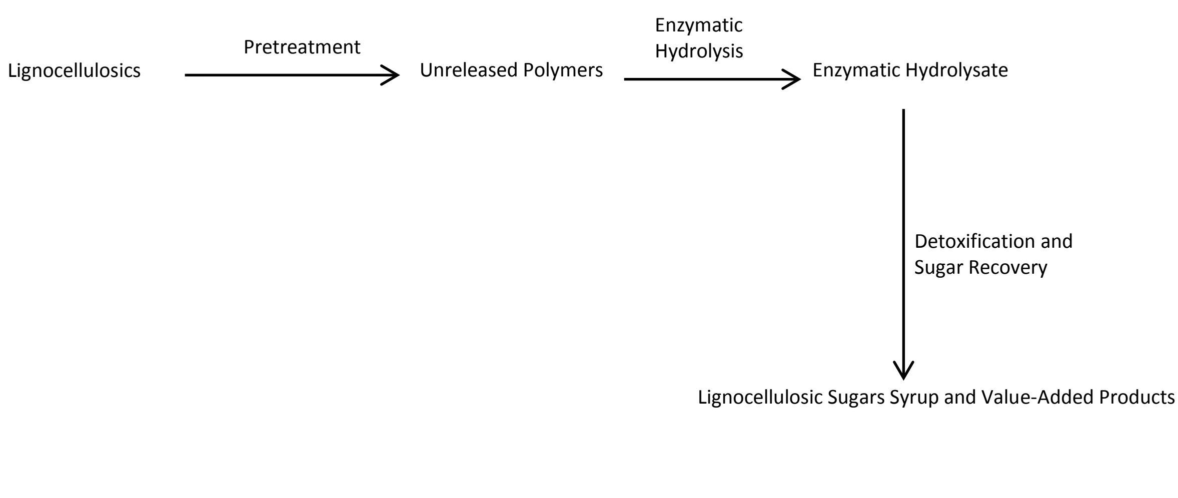


Figure 1. Detoxification and Sugar Recovery of Enzymatic Hydrolysates.

EXPERIMENTAL PROCEDURE

Sorghum bagasse and energycane bagasse were pretreated with dilute ammonia (28% NH_4OH solution) for 1h at 160ºC in a 300-mL pressure reactor. The ratio of biomass to dilute ammonia and water was 1:0.5:8. Biomass loading at 5% was used. The pretreated sorghum and energycane bagasse were enzymatically hydrolyzed using a combination of Spezyme®CP and Novozyme188, a cellulase and β-glucosidase, respectively. The resulting enzymatic hydrolysate (EH) was then rapidly cooled to about 25ºC in an ice bath, centrifuged, and the mass and liquor (EHL) stored separately at -20ºC until analyzed and detoxified. For the detoxification procedures, a fixed amount of PSSB or PECB EHL was combined with ionic liquid (IL LLE), ion exchange resin (IER), activated charcoal (AC), molecular sieve (MS), or calcium salts (Ca^2+). Sugars (sugar recovery %) in the EHL and the non-sugar components were measured before and after each detoxification procedure. Sugars and non-sugar components were analyzed by High Performance Liquid Chromatography (HPLC). Detoxification parameters were optimized.

RESULTS AND DISCUSSION

High Performance Liquid Chromatography separation with Diode Array Detection (DAD) method provided detail into the optimum combination and/or concentration of enzymes generating maximum sugars with the least amount of non-sugar components. An initial detoxification process was also developed. The method involved a C_{18}-SPE (Solid Phase Extraction) cartridge followed by elution of the concentrated sample with solvents of increasing polarity (ethyl acetate, methanol, and sulfuric acid-water mixture) to remove components adsorbed on the cartridge as fractionated groups. Although successful in separating the major groups such as organic acids and furaldehydes from phenolic acids, the strategy needs to be evaluated and developed further since sugars were not fully recovered and separated. Some of the detoxification strategies were affected by the nature of the EHL bagasse such as the case of activated charcoal and molecular sieves. Although, IER and AC had good sugar recoveries using their respective optimized conditions (both for sweet sorghum and energycane), recovery of non-sugar components is still problematic. MS, on the other hand had moderate sugar recovery; however, it could not sufficiently remove the non-sugar components. The ionic liquid LLE detoxification strategy proved to have good sugar recoveries under optimized conditions of IL1 (87% for PSSB and 60% for PECB) while removing and/or lowering the non-sugar components regardless of the nature of the EHL bagasse. The best detoxification strategy is one where sugar concentrations are kept high after detoxification while non-sugar components are low or completely removed. Also, recovery of these non-sugar components can be accomplished easily. Ionic liquid LLE and possibly ion-exchange resin proved to be routes we want to explore further for the detoxification of lignocellulosic enzyme hydrolysates (Table 1). After the detoxification step, the hydrolysate can be concentrated into lignocellulosic sugar syrup.

Table1. Summary of non-sugar components from pretreated sorghum and energycane enzyme hydrolysates removed by various detoxification strategies.

Detoxification Strategy	Sugar Recovery (%)		Inhibitors*	
	Sweet sorghum	Energycane	Sweet sorghum	Energycane
Solid Phase Extraction SPE	Low	Low	-	-
Ionic Liquid IL LLE	Good	Moderate	-	-
Ion-exchange resin IER	Good	Moderate	-	-
Activated Charcoal AC	Good	Low	-	-
Molecular sieve MS	Moderate	Low	+	+
Calcium salts Ca^{2+}	Very low	Very low	-	-

*Inhibitors: Organic acids, Furaldehydes and Phenolic acids; +/-: present/absent

ACKNOWLEDGEMENTS

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