

# **LSU AgCenter Audubon Sugar Institute Factory Operations Seminar**

Sugar Research Station  
5755 LSU Ag Road  
St. Gabriel, LA  
April 20, 2016





Visit our website: [www.LSUAgCenter.com](http://www.LSUAgCenter.com)

William B. Richardson, LSU Vice President for Agriculture  
**Louisiana State University Agricultural Center**  
**Louisiana Agricultural Experiment Station**  
**Louisiana Cooperative Extension Service**  
**LSU College of Agriculture**

The LSU AgCenter is a statewide campus of the LSU System and provides equal opportunities  
in programs and employment.

Louisiana State University is an equal opportunity/access university



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

## CANE SUPPLY

Sugarcane Ripener .....	3
<i>A. Orgeron, K. Gravois, B. Legendre, J. Griffin</i>	

## BOILING HOUSE

Sugar Crystal Size Analysis for Louisiana Sugar Mills.....	6
<i>F. Ehrenhauser, C. Gaudet, I. Tishechkina, D. Aragon</i>	

Boiling House Simulation Models in Louisiana .....	10
<i>D. Aragon</i>	

Off-Season Evaporator Cleaning with EDTA .....	14
<i>F. Ehrenhauser, C. Gaudet, R. Whitelaw, J. Bonin</i>	

The 2015 Molasses Survey .....	19
<i>C. Verret, C. Wall</i>	

Effect of Purity Drop and Crystal Size on Molasses Exhaustion in the C-Strike.....	25
<i>D. Aragon, I. Tishechkina, F. Ehrenhauser, C. Gaudet, T. Faucheux</i>	

## MISCELLANEOUS TOPICS

Color Changes through the Factory .....	30
<i>H. Birkett, J. Stein</i>	

Syrup Storage.....	44
<i>S. Linares, F. Ehrenhauser</i>	

Bagacillo in Raw Sugar .....	50
<i>C. Gaudet, F. Ehrenhauser</i>	

Sugar Cane Starch Reference Materials .....	54
<i>F. Ehrenhauser, C. Gaudet, J. Bonin</i>	

Deashing of Sugar Streams .....	56
<i>T. Lueangrujiwong, P. Gaston, F. Ehrenhauser</i>	

## Sugarcane Ripener

Albert J Orgeron  
Area Pest Management Specialist  
LSU AgCenter

Kenneth A Gravois, Ben L Legendre, James L Griffin

### **Early Season Variety Response to Glyphosate Ripener**

On August 31, 2015, 5.3 oz/a of Roundup PowerMax II was applied to the experimental plots in St. Gabriel. Varieties included in the experiment were HoCP 96-540, L 01-299, HoCP 04-838, HoCP 07-613, HoCP 09-804, and Ho 09-840. The experiment was hand harvested on September 28<sup>th</sup>, 29 days after glyphosate treatment. Samples were processed using Spectra Cane NIR to determine variety TRS, fiber, and purity. Environmental conditions promoted natural ripening, resulting in above average sucrose levels for all varieties as evident by purity levels above 84% (Table 1) for nontreated sugarcane. TRS was increased by 10.7% for HoCP 96-540, 29 days after treatment; however, other varieties evaluated did not respond to glyphosate.

### **Evaluating ATRIun as a Sugarcane Ripener**

On August 31, 2015, Atrium was applied at 20, 30, and 40 oz/a to plantcane HoCP 96-540 in St. Gabriel. Also included in this study were the ripeners Roundup PowerMax II and Moddus, applied at 5.3 and 19 oz/a, respectively. A nontreated check was also included for comparison. The experiment was hand sampled then harvested by combine on September 29<sup>th</sup>, 30 days after ripener treatment. Samples were processed using Spectra Cane NIR to determine variety TRS, fiber, and purity. Cane yield (TC/A) and sugar per acre (TRS/A) were also calculated. TRS, Cane Yield and Sugar Yield were similar to the check regardless of ATRIun rate (Table 2).

### **Evaluating NAI 1360 as a Sugarcane Ripener**

On August 26, 2015, NAI 1360 was applied at 2.1oz/a to plantcane HoCP 96-540 and HoCP 04-838 in St. Gabriel. Also included in these studies was the ripener Roundup PowerMax II applied at 5.5 oz/a, and a nontreated check. The experiments were hand sampled at 4, 6, and 8 weeks after treatment (WAT) and were harvested by combine on October, 20, 2015. Samples were processed using Spectra Cane NIR to determine variety TRS, fiber, and purity. Cane yield (TC/A) and sugar per acre (TRS/A) were also calculated. TRS, Cane Yield and Sugar Yield were similar to the check regardless for NAI 1360 (Table 3 and 4). Roundup PowerMax II increased TRS compared to the nontreated check, 4 and 8 WAT for HoCP 96-540; however, TRS was not improved in HoCP 04-838 with Roundup PowerMax II.

Table 1. Effect of Roundup PowerMax II on TRS, Fiber, and Purity for 4 commercial and 2 experimental varieties at the Sugar Research Station in St. Gabriel, LA in 2015 plantcane.

Variety	Glyphosate	TRS <sup>1</sup> (lb/ton)		Fiber (%)	Purity (%)	
HoCP 96-540	No	252		13.8	84.6	
	Yes <sup>2</sup>	279	+	13.7	86.9	+
L 01-299	No	259		12.6	85.3	
	Yes	270		12.0	86.0	
HoCP 04-838	No	261		11.9	86.1	
	Yes	250		13.4	84.0	-
Ho 07-613	No	271		12.6	86.5	
	Yes	263		12.5	85.8	
HoCP 09-804	No	253		12.7	84.1	
	Yes	259		12.5	85.3	
HoCP 09-840	No	269		13.6	86.3	
	Yes	272		13.4	86.2	

<sup>1</sup> Mean separation used least square means probability differences where P=0.05. Varieties that are significantly higher or lower than nontreated are denoted by a plus (+) or minus (-), found next to the mean value for each trait.

<sup>2</sup> Plots treated with 5.3 oz/a of Roundup PowerMax II on August 31, 2015 and harvested September 28, 2015.

Table 2. Effect of ATRiun on Cane Yield, TRS, and Sugar Yield on plantcane HoCP 96-540 at the Sugar Research Station in St. Gabriel, LA in 2015.

Treatment <sup>1</sup>	Rate/a	Cane Yield (tons/a)	TRS (lb/ton)	Sugar Yield (lb/a)
ATRiun	20 oz	43.6	228 abc <sup>2</sup>	9,936
ATRiun	30 oz	44.5	220 bc	9,780
ATRiun	40 oz	47.2	211 c	9,954
Roundup PowerMax	5.3 oz	43.6	241 a	10,532
Moddus	19 oz	44.7	236 ab	10,531
Check		44.2	225 abc	9,909

<sup>1</sup> Plots treated with on August 31, 2015 and harvested September 29, 2015.

<sup>2</sup> Means followed by the same letter are not significantly different from each other.

Table 3. Effect of NAI 1360 on TRS, Cane Yield, and Sugar Yield on plantcane HoCP 96-540 at the Sugar Research Station in St. Gabriel, LA in 2015.

Treatment	Rate/a	TRS	TRS	TRS	Cane Yield (tons/a)	Sugar Yield (lb/a)
		4WAT (lb/ton)	6WAT (lb/ton)	8WAT (lb/ton)		
NAI 1360	2.1 oz	189 b	222 a	243 b	46.4 a	11238 a
Roundup PowerMax	5.5 oz	200 a	229 a	254 a	45.8 a	11623 a
Check		183 b	220 a	241 b	45.9 a	11104 a

<sup>1</sup> Plots treated with on August 26, 2015 and harvested by combine on October 20, 2015.

<sup>2</sup> Means followed by the same letter are not significantly different from each other.

Table 4. Effect of NAI 1360 on TRS, Cane Yield, and Sugar Yield on plantcane HoCP 04-838 at the Sugar Research Station in St. Gabriel, LA in 2015.

Treatment	Rate/a	TRS	TRS	TRS	Cane Yield (tons/a)	Sugar Yield (lb/a)
		4WAT (lb/ton)	6WAT (lb/ton)	8WAT (lb/ton)		
NAI 1360	2.1 oz	223 a	244 a	278 a	40.3 a	11186 a
Roundup PowerMax	5.5 oz	226 a	248 a	286 a	39.5 a	11302 a
Check		217 a	238 a	273 a	42.8 a	11685 a

<sup>1</sup> Plots treated with on August 26, 2015 and harvested by combine on October 20, 2015.

<sup>2</sup> Means followed by the same letter are not significantly different from each other.

## SUGAR CRYSTAL SIZE ANALYSIS FOR LOUISIANA SUGAR MILLS

2015/16 Season

*Franz Ehrenhauser, Cy Gaudet, Iryna Tischechkina, Daira Aragon*

Crystal size analysis had been provided as a service to Louisiana sugar mill since 2007. During the 2015/16 season more than 682 different samples were analyzed and requested from Louisiana sugar mills. The Cilas 1800 particle size analyzer provides the ability to assess the entire size spectrum of crystals ranging from 0.04  $\mu\text{m}$  to 2,500  $\mu\text{m}$ . 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.

### C-SUGAR SAMPLES

This year particular emphasis was placed on collecting sufficient samples to provide a comprehensive C-sugar crystal size overview for the Louisiana industry. From every factory samples were collected weekly. Table 1 shows the summary of the analyzed C-sugars. For each mill at least 11 samples were obtained. The average mean C-sugar crystal size was 203 microns, with the 10 and 90-percentile being 32 and 405 microns respectively. Median crystal size was on average 184 microns. Average CV values range from 0.61 to 0.78. The value for Mill V of 0.92 is exceptional due to a sampling issue. As the C-machines at this factory employ water inside the machine to reduce breakage, early samples collected with a solid stick at the top of the machine were not very representative. On a solid stick the larger crystals bounce off and are not properly measured. Halfway during the season the solid-stick sampling was substituted with a fish-net sampling method, which improved sampling accuracy dramatically. This change in sampling procedure is reflected in the high variability of the CV value.

Figure 2 shows the average 90-percentile for the 2015 crop. There is little variability in the crystal size between the mills indicating that the average value of 405 micron is a common upper boundary for growth of sugar crystals in C-strikes. High variability of the 90-percentile indicates either variable crystallization or breakage in the C-machines, both of which should be avoided.

Figure 3 shows the average median for all mills during the 2015 crop and for four mills the values for the previous four crops. Sizes vary here more greatly than for the 90-percentile. A large difference between the median and the 90-percentile is indicative of a broad distribution and should be avoided, as it raises the CV value. A typical upper value for the Louisiana industry is close to 200 microns; however, in previous years higher values have been observed.

Figure 4 shows the average 10-percentile of the 2015 crop. The large variability here is due to breakage in the centrifugal machine and potential false grain formation. The best value was achieved by mill III with an average value of 47 micron, indicating low breakage and low false grain formation.

**Table 1.** 10-percentile, median and 90-percentile, Mean and CV-values of C-sugars during the 2015 crop. (Average values  $\pm$  standard deviation)

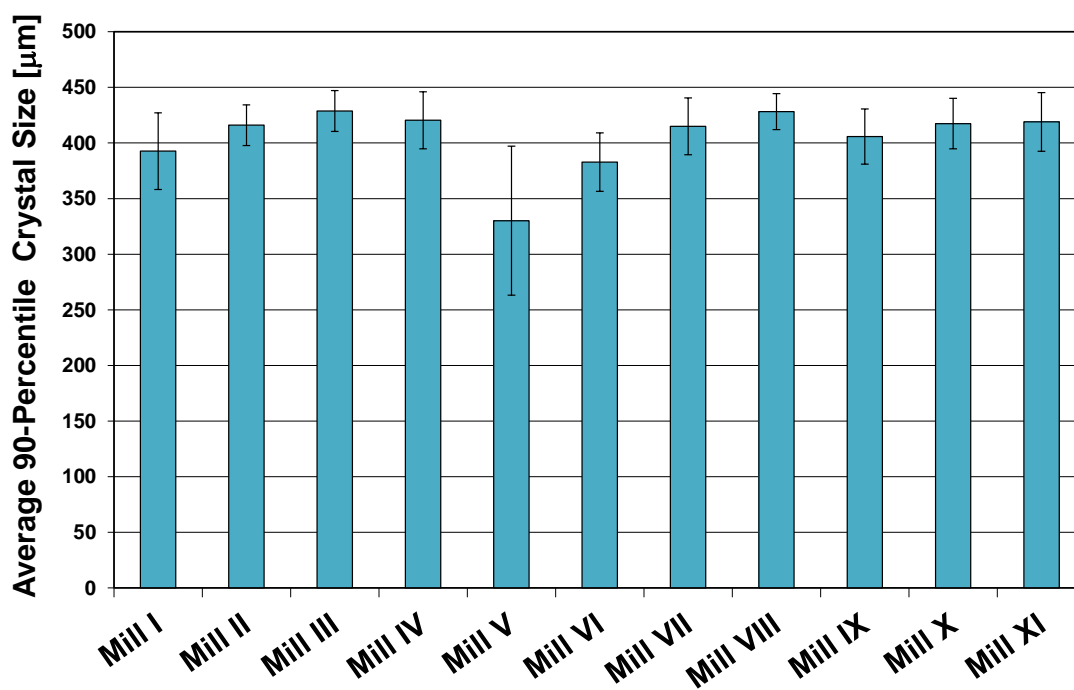
Mill	# of samples	D 10% [ $\mu\text{m}$ ]	D50% [ $\mu\text{m}$ ]	D90% [ $\mu\text{m}$ ]	Mean [ $\mu\text{m}$ ]	CV
I	49	37 $\pm$ 17	186 $\pm$ 36	393 $\pm$ 34	203 $\pm$ 29	0.66 $\pm$ 0.07
II	61	35 $\pm$ 10	188 $\pm$ 32	416 $\pm$ 18	208 $\pm$ 20	0.69 $\pm$ 0.06
III	22	47 $\pm$ 14	228 $\pm$ 38	429 $\pm$ 18	233 $\pm$ 23	0.61 $\pm$ 0.07
IV	18	33 $\pm$ 15	206 $\pm$ 52	420 $\pm$ 26	217 $\pm$ 32	0.68 $\pm$ 0.09
V	36	14 $\pm$ 5	100 $\pm$ 36	330 $\pm$ 67	140 $\pm$ 38	0.92 $\pm$ 0.11
VI	11	20 $\pm$ 6	147 $\pm$ 34	383 $\pm$ 26	174 $\pm$ 28	0.77 $\pm$ 0.08
VII	61	38 $\pm$ 9	196 $\pm$ 28	415 $\pm$ 26	213 $\pm$ 20	0.67 $\pm$ 0.05
VIII	20	22 $\pm$ 8	201 $\pm$ 45	428 $\pm$ 16	213 $\pm$ 22	0.72 $\pm$ 0.07
IX	16	22 $\pm$ 9	159 $\pm$ 49	406 $\pm$ 25	191 $\pm$ 27	0.78 $\pm$ 0.08
X	58	29 $\pm$ 15	190 $\pm$ 49	417 $\pm$ 23	209 $\pm$ 30	0.71 $\pm$ 0.10
XI	25	40 $\pm$ 22	208 $\pm$ 44	419 $\pm$ 26	220 $\pm$ 31	0.65 $\pm$ 0.10
Overall	377	32 $\pm$ 15	184 $\pm$ 50	405 $\pm$ 41	203 $\pm$ 35	0.71 $\pm$ 0.11

Figure 5 shows the CV value for the 2015/16 season as well as historic data for 4 mills. A typical CV value is approximately 0.7 (mill V is exceptional due to sampling changes). Most factories fall within this category; however, in the past better values have been achieved. The low variability of the 90-percentile, indicates that growing crystals bigger is not likely, instead the effort should be focused on avoiding false grain and breakage.

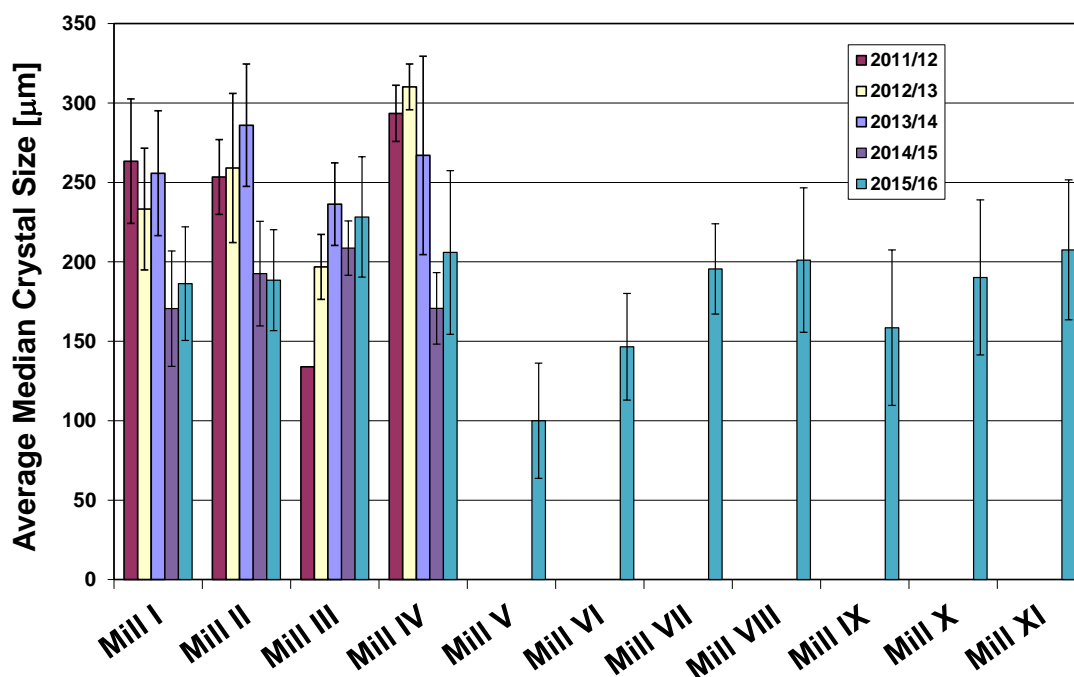
#### ACKNOWLEDGEMENTS

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

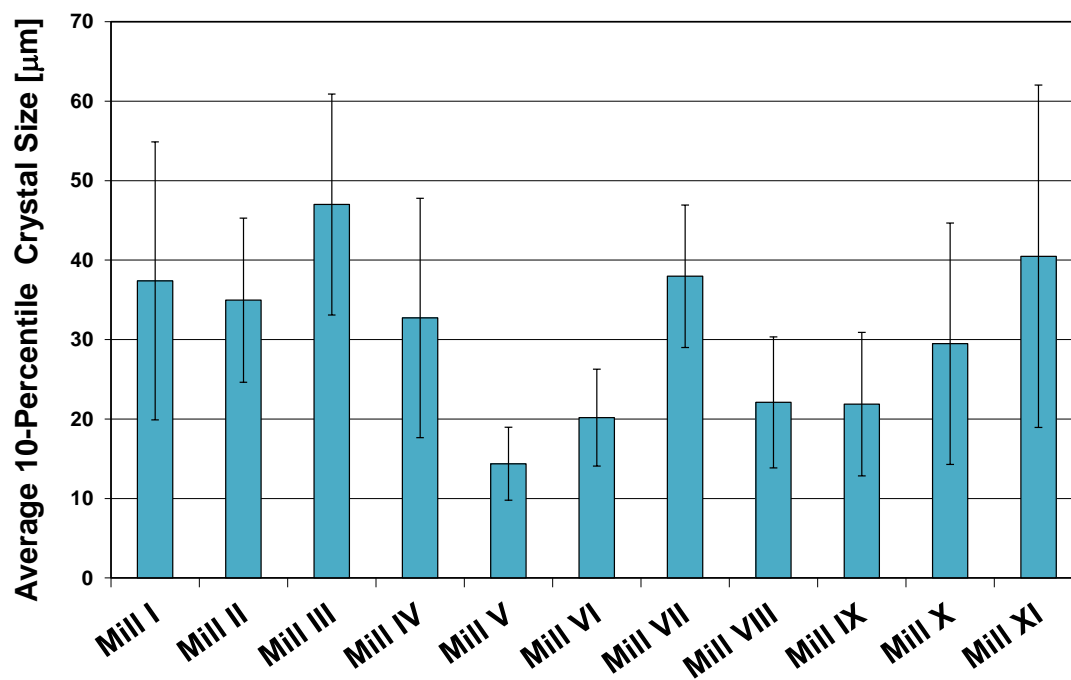




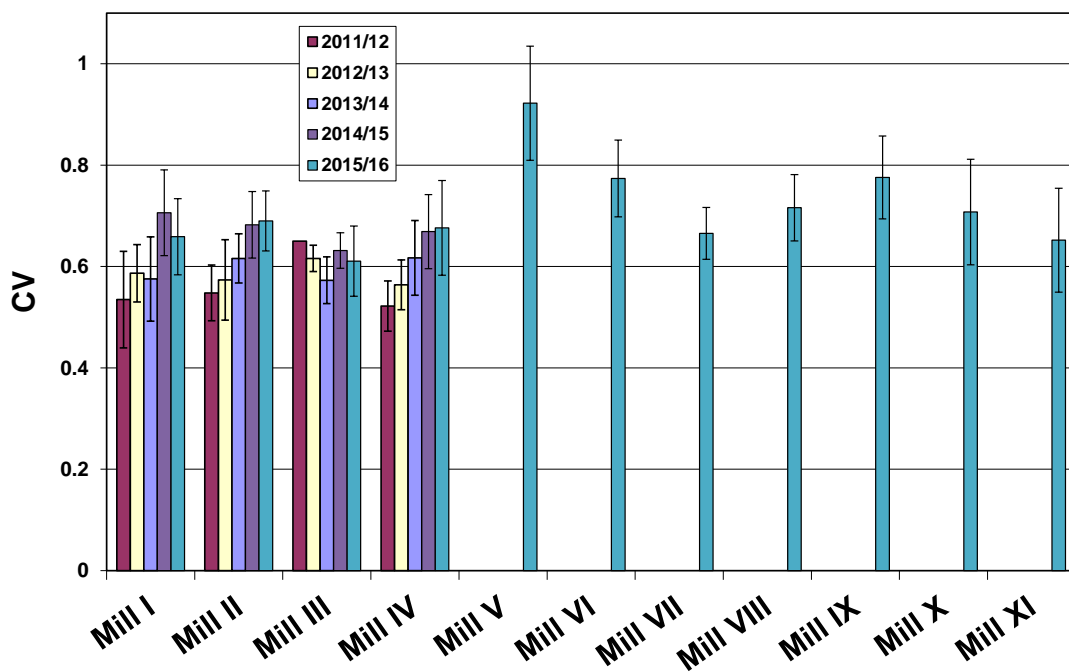
**Figure 1.** Average 90-percentile of C-sugar during the 2015 crop



**Figure 2.** Average Median of C-sugar during the 2011-2015 crop



**Figure 3.** Average 10-percentile of C-sugar during the 2011-2015 crop



**Figure 4.** CV of C-sugar during the 2011/14 seasons.

## Boiling House Simulation Models in Louisiana

Daira Aragon  
Audubon Sugar Institute, LSU AgCenter

### Summary

Process simulation models are used to evaluate the effects of changes in process conditions or equipment on sugar quality and exhaustion, to balance the factory and to obtain process conditions for good sugar quality and exhaustion. The Audubon Sugar Institute initiated an effort to develop simulation models for all sugar factories in Louisiana, and to use such models to assist during factory modifications and operation. This paper summarizes the models that have been developed, presents the deviation of simulation results from factory data, and gives the common challenges faced to develop and implement such simulation models.

### Introduction

In a raw sugar factory, simulation models can be used to assist during the design, operation and optimization of the process, for example:

- to find additional pan capacity and steam requirements when switching to a different boiling scheme,
- to find massecuite and magma purities, wash water ratio at centrifugals and to use to obtain lower final molasses purity, lower color sugar color and higher sugar pol,
- to find evaporator bleeding locations to lower steam consumption,
- to evaluate effects on steam production in bagasse boilers and
- to obtain settings to improve control of ph in clarifiers, among others.

The following simulation models have been developed for Louisiana sugar mills, using the software Sugars™:

#### 2013-2016

- M.A. Patout's diffuser section (diffuser, clarifier and evaporators)
- Cajun's C strike
- Cajun's boiling house (including updates)
- Westfield's boiling house (including updates)
- Westfield complete factory
- Raceland's boiling house
- Lula's boiling house model

#### 2011-2012

- Lafourche's complete factory
- Sterling's boiling house
- Lula's complete model
- Alma's boiling house

This paper presents only the models developed or updated after 2013, with the exception of the complete model for Westfield.

## Procedure

Boling schemes used in Louisiana include 3-boiling, 3-boiling plus double purge and double magma. One of the factories has a diffuser to process cane. Factory simulation models were developed in the software Sugars™ (Sugars International, LLC). The following information was used as input for the models:

### Boiling house

- Temperature and pressure of steam
- Brix of A and B molasses
- Brix of C grain
- Brix of A, B and C massecuites
- Purity and brix of final molasses
- Purity and brix of magmas
- Sugar pol

### Diffuser section

- Cane throughput
- Steam temperature and pressure
- Profile of juice purity and brix
- Brix and purity of syrup from evaporators
- Bagasse moisture and pol

Purity of molasses, massecuites and grain was estimated by the software. Syrup flowrate was estimated by either factory balances or as sum of all syrup needed in pans. Most of the information initially used for model development was taken from factory reports and personal communication with engineers and other plant personnel. Once the initial model was developed, some samples were taken and analyzed in Audubon's lab (if possible), while other data was taken for reports and compared to simulation results. Models were modified accordingly to match as close as possible the available data.

It is important to note that simulation models need to be regularly checked and validated against factory data. This is especially true after any change whether in process conditions or in equipment.

## Results

Table 1 presents the percentage that simulation results deviated from factory data for each mill, in the case of boiling house models. Results in this table suggest that the quantities of massecuite and final molasses produced have the highest deviations. Flowrates from simulation are harder to match to factory data because values presented in the factory reports are usually estimated. Purities given by simulation are in very good agreement with factory data, with very few deviations higher than 3.0%.

Simulation models of Westfield, Lula and Raceland are being used to evaluate effects on final molasses purity and sugar quality at different magma purities. These results can be used to determine the purities that give lower molasses purity and color.

Table 1. Deviation (%) of simulation from factory data for boiling house models

		Cajun C strike (2014)	Cajun (10/19/2015)	Westfield (10/20/2015)	Lula (10/22/15)	Raceland (11/02/2015)
A massecuite	Quantity	--	0.8	10.0	4.2	1.7
	Purity	--	0.8	0.3	0.0	0.1
B massecuite	Quantity	--	15.5	41.2	33.8	3.5
	Purity	--	2.7	1.1	0.3	0.4
C massecuite	Quantity	0.6	3.2	20.4	29.3	0.05
	Purity	0.1	12.5	3.7	1.2	0.4
C grain	Quantity	6.2	--	--	--	--
	Purity	0.5	1.7	0.7	0.6	0.05
A molasses	Brix	--	0.4	0.4	0.1	0.1
	Purity	--	0.6	0.3	0.0	0.03
B molasses	Brix	--	1.4	0.2	0.3	0.00
	Purity	--	9.8	1.3	0.1	0.04
Final molasses	Quantity	0.7	10.3	7.6	13.6	1.4
	Brix	0.9	1.0	2.0	1.4	0.0
	Purity	2.9	4.6	3.3	0.7	0.1
C magma	Brix	0.5	0.3	0.0	3.1	--
	Purity	1.2	1.9	0.1	0.2	0.1
B magma	Brix	--	0.0	--	--	--
	Purity	--	0.8	--	--	--
Raw sugar	Quantity	--	3.0	5.3	4.9	4.8
	pol	--	0.2	0.0	0.0	0.04
	Color	--	--	1.6	0.9	--

Table 2 shows the differences for the diffuser model of M.A. Patout. Simulation purities deviate not higher than 6% from factory data.

Table 2. Deviation (%) of simulation purities from factory data for diffuser model (11-14-2014)

Variable	%Deviation
Draft juice	2.8
Scalding juice 1	2.3
Scalding juice 2	2.1
Cell 3	2.3
Cell 4	2.6
Cell 5	4.8
Cell 6	7.4
Cell 7	6.0
Cell 8	3.9
Cell 9	2.3
Cell 10	1.3
Cell 11	1.2
Cell 12	1.2
Pre/Quad	0.4

## **Challenges in Simulation Models for Raw Sugar Factories**

The use of the simulation model determines the accuracy (i.e. deviation of simulation results from actual data) that can be tolerated. In general, fundamental studies require the lowest deviation possible, while higher variations are acceptable for comparison studies. Very variable processes, such as the case of a sugar mill, will generally have high deviations of simulation values from factory data, at least in some of the quantities. The same is true for larger models.

In particular, a sugar factory is a complex process. Uncontrolled changes in impurity levels, viscosities, and pH, among others depend on the cane crushed. Additionally, engineers and other personnel make changes in the operation itself depending on purities of massecuites and syrup, for example, back-boiling A molasses, increasing wash water in centrifugals, bypassing mills or stopping for maintenance. Often times, these voluntary changes are not recorded explicitly in the manufacturing report. Therefore, matching the simulation to factory data is difficult when these changes in conditions happen.

One way to overcome these barriers is using simulation results for relative comparisons instead of absolute values. This means that once a particular operation (or day) has been simulated, it would be set as the base case. New simulations will be run for different conditions and results will be given in terms such as “there will be an increase of 0.2 points in final molasses”, “sugar color will decrease by 100 units”, or “there will be a production of 5,000 lb of sugars more”. Sometimes, percentages are used for these comparisons.

Even with the challenges associated to the simulation of raw sugar production, results obtained provide useful information to improve operation.

## **Acknowledgements**

American Sugar Cane League for providing funding.

The following sugar factories and their personnel: Cajun, Lula-Westfield, M.A. Patout and Raceland.

## OFF-SEASON EVAPORATOR CLEANING WITH EDTA

*Franz Ehrenhauser, Cy Gaudet, Robert Whitelaw, Jonathan Bonin*

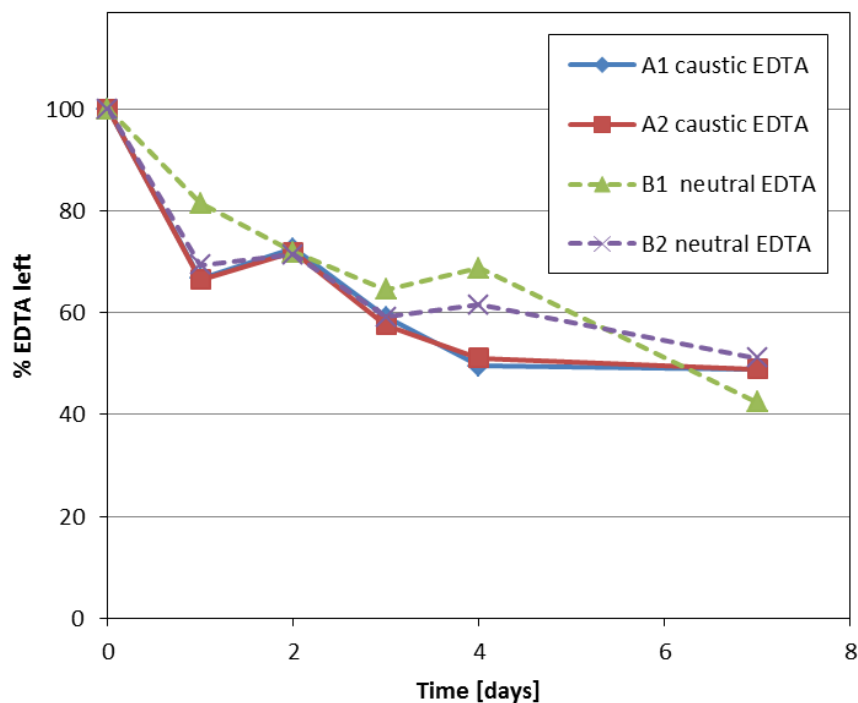
Evaporator cleaning is a time and cost intensive operation in Louisiana. Ideally evaporation of sugar cane juice to syrup would require only heat in form of steam and no significant chemical input. However, fouling of evaporator surfaces limits the heat transfer significantly, reducing throughput and steam efficiency. This performance-dampening fouling is addressed by regular cleaning of the evaporator. Typical cleaning procedures in the Louisiana industry involve the washing/boiling with water, following a caustic boil, an acid boil (with and without fluoride), neutralization and washing with water. The main goal of these procedures is to bring insoluble earth alkali salts (Ca, Mg), silica and any organic matter stuck on the evaporator surface back into solution. Depending on the location of the evaporator vessel the scale to be dissolved can vary greatly and might require specific cleaning protocols. Typically caustic soda, soda ash, hydrochloric and phosphoric acid as well as ammonium bifluoride are utilized. Ethylenediaminetetraacetate (EDTA) provides an alternative dissolution mechanism to the alkaline and acid mechanism. EDTA attacks scale through a complexation mechanism, i.e. it bonds strongly to metals ions such as iron, copper, calcium, magnesium. It has been reported as an effective, but rather expensive cleaning agent. EDTA complements caustic cleaning and provides an alternative to utilizing acids. Compared to acid cleaning it provides a much gentler approach as its corrosion rate is significantly less. The action of EDTA is unique as it proceeds very rapid even at room temperature, and it is used as analytical reagent for calcium and magnesium determination.

In June 2015 ASI was requested to provide assistance to the Westfield sugar factory, as a unique application for EDTA cleaning was suggested. An Alfa-Laval plate evaporator (ES700 WFM utilized as pre-evaporator) had been inspected and approximately 50% of the compartments had been found to be blocked, which limited efficiency significantly. Under normal circumstances opening the evaporator and a complete refurbishment would have been ideal; however, three month before the season the time and potential money (estimated >\$80,000.- for complete plate and gasket change) requirement were prohibitive of regular (mechanical) cleaning. Instead a cold cleaning procedure utilizing EDTA was attempted.

### LAB EXPERIMENTS

Scale was collected and analyzed at the LSU XRD facility. The main crystalline component was identified as silica, even though the scale did not represent a typical silica scale. As a first step lab dissolution studies were conducted. EDTA can be utilized as alkaline tetrasodium salt, neutralized or as an acidic solution. Once it is depleted either the cleaning is complete or the solution should be replaced with fresh EDTA. In order to measure the progress of EDTA utilization the EDTA has to be titrated with a calcium salt solution to determine its activity. During regular hot cleaning with EDTA, the solution is normally fairly clean and the end point of the titration easily visible, for off-season cleaning the solution is very turbid to begin with. Two methods were found to allow the titration to proceed without major issues: either centrifuging the solution to clarity or measuring the progress of the titration in a turbidimeter. The second method is preferred as it allows for easy endpoint detection.

6.00 g of the scale were subjected in duplicates to neutral and alkaline EDTA solution (5% of Dissolvine E-39 in deionized water). The solution was stirred and the EDTA strength followed by titration. Figure 1 shows the decline of EDTA concentration during the laboratory testing. There was no significant difference between the alkaline and neutral EDTA treatment. At the end of the experiment the remaining scale was filtered, dried and weighed. A mass loss of 64 % and 68 % (not significantly different) was observed for the alkaline treatment and the neutral treatment respectively. Based on this substantial mass loss the Westfield factory decided to go ahead with a full scale EDTA cleaning of the plate evaporator.

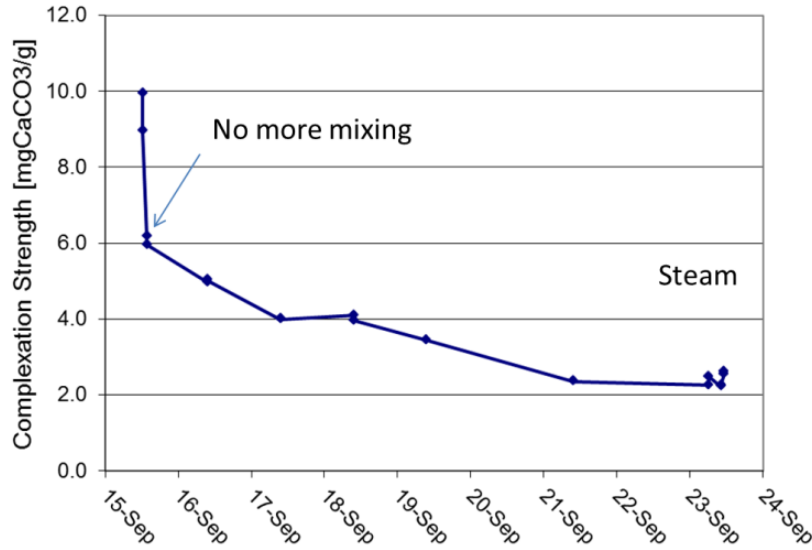


**Figure 1.** EDTA concentration progress during the laboratory dissolution test.

## FULL SCALE FACTORY TEST

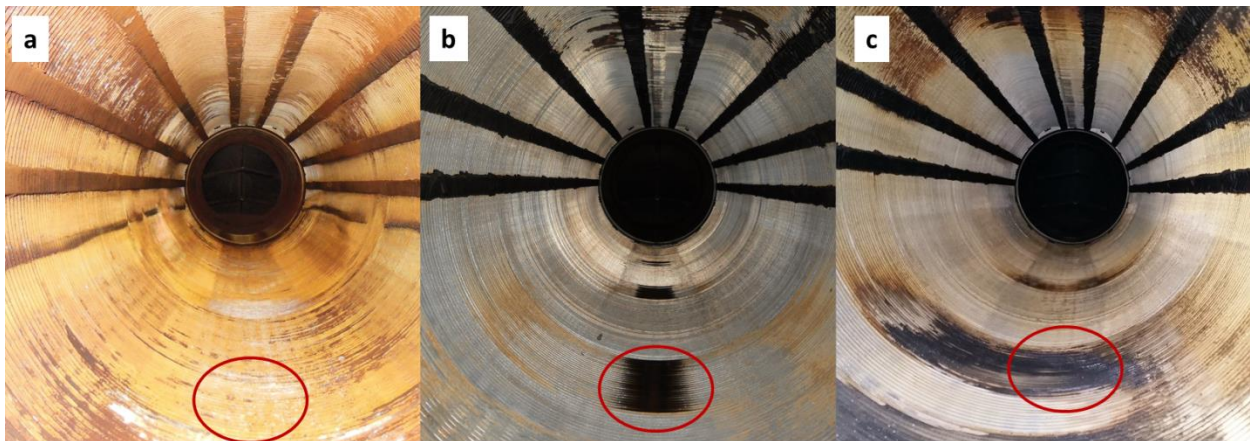
Dissolvine E-39 and approximately 2000 gal water were combined in a tank to a 5% solution and pumped in a closed loop through the evaporator for a total of nine days. Figure 2 shows the decline of EDTA strength during the operation. The steep initial decline in the first few hours was due to the incomplete mixing in the tank and the pump fulfilling the mixing operation. The complexation strength fell within 3 days to 66% of its original value and continued to decline until the sixth day to roughly a third. At the end of the cleaning period (September 24<sup>th</sup>) steam was available and the evaporator was operated briefly on steam. During this application the EDTA strength did not decline any further, indicating that it was too weak to further attack the scale and therefore completely exhausted.



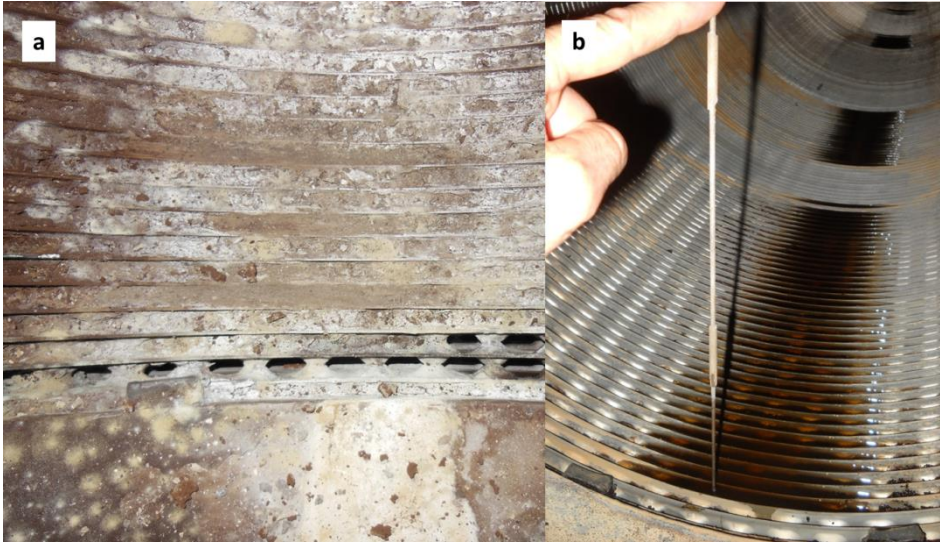


**Figure 2.** EDTA complexation strength during factory test

Figure 3a shows the pictures of the opened evaporator just before the season. One blocked part is highlighted with a red circle. Figure 4a shows the blockage at the front of the evaporator. The EDTA removed the scale of all minor scaled stainless steel surfaces bringing the surface back to a shiny finish (Figure 3b and 4b). For the blocked passages it fell short dissolving the entire suspected 5 feet of scale and could only dissolve approximately 8-12 inches as indicated by the insertion of the welding rod in picture in Figure 4b. The dark looking sections were still blocked and had water standing in puddles (shown in Figure 3b). Due to time constraints the evaporator was not further cleaned with EDTA and utilized partially blocked during the season. After the season the evaporator was opened and inspected. As can be seen in Figure 3c, the partially blocked passages had filled up with scale (the blockage below provides a pocket, where material can collect). Remarkable was the condition of the previously fully cleaned parts, as they had retained their shine and were essentially without scale. While the EDTA was not tested with sufficient time to see if it could eat through the entire scale buildup, it performed well as an off-season, cold-cleaning agent, enhancing the regular cleaning schedule during the season.



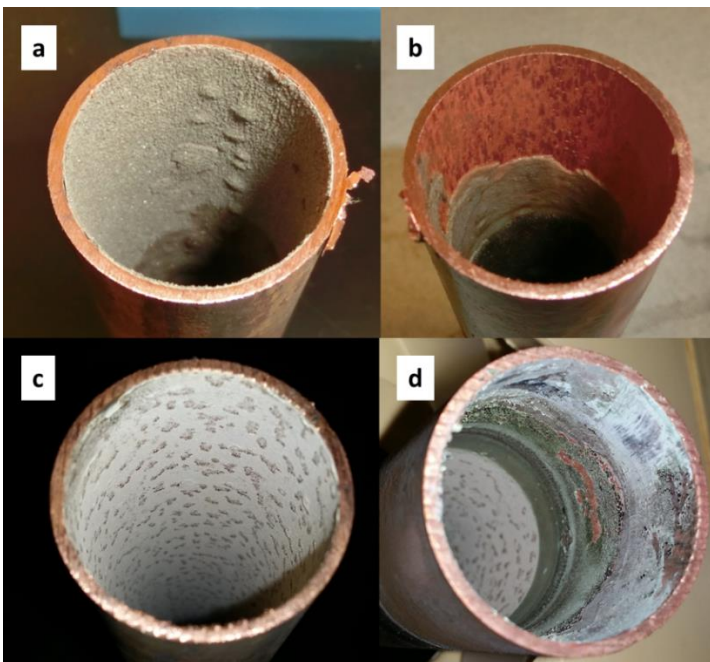
**Figure 3.** a) Evaporator inside after the 2014/15 season, b) after EDTA cleaning and c) after the 2015/16 season. The red circle marks one of the blocked passages.



**Figure 4.** Blockage at the front of the plate evaporator a) after the 2014/15 season and b) after EDTA cleaning.

#### OFF-SEASON COPPER TUBE CLEANING

Cut copper evaporator tubes from the Lula factory were also subjected to a 5% EDTA solution. Their scale exhibited small, clear crystals embedded in a grey mass (Shown in Figure 5a) and a white thin layer (Figure 5b). The tubes showed great susceptibility to cold EDTA cleaning and were within 4 days in a stirred solution of 5% Dissolvine E39 either bare of scale, or had very soft scale, which could be easily removed. For the tube shown in Figure 5a and b the scale had taken on a sandy gritty consistency, while the scale in Figure 5c and d became slimy. After drying up, it flaked off easily and the bare copper spot was achieved by just touching it.



**Figure 5.** Copper tubes cleaned with EDTA a) and c) before b) and d) after EDTA

## CONCLUSION

EDTA is not a cheap cleaning agent (\$ 0.70/lbs) and cannot be reused; however, it shows promise for cold off-season cleaning. It can be applied either in bulk (with a recirculation pump) or sprayed on. While scale might not be fully dissolved, it is likely softened, which facilitates its mechanical removal, reducing man hour requirements for cleaning of the evaporators and enhancing evaporator operation.

## ACKNOWLEDGEMENTS

The authors would like to acknowledge the American Sugar Cane League for providing funding for travel and salary. Jim LePage (Akzonobel), Bob Templet and Jon Pena (both Harcros Chemicals) contributed technical expertise and free samples of Dissolvine E-39. Peter Gaston helped with the analysis in the laboratory. The authors would also like to acknowledge the personal at the Lula and Westfield sugar factories for initiating and supporting this investigation.

# THE 2015 MOLASSES SURVEY

*C. Verret & C. Wall*

## 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 2015 survey season stretched from 10-04-15 until 01-03-16. A total of 154 molasses and syrup samples and 10 juice samples were analyzed in duplicate for the 2015 season. Including standards, this totaled 538 samples for 2015. Analyses included:

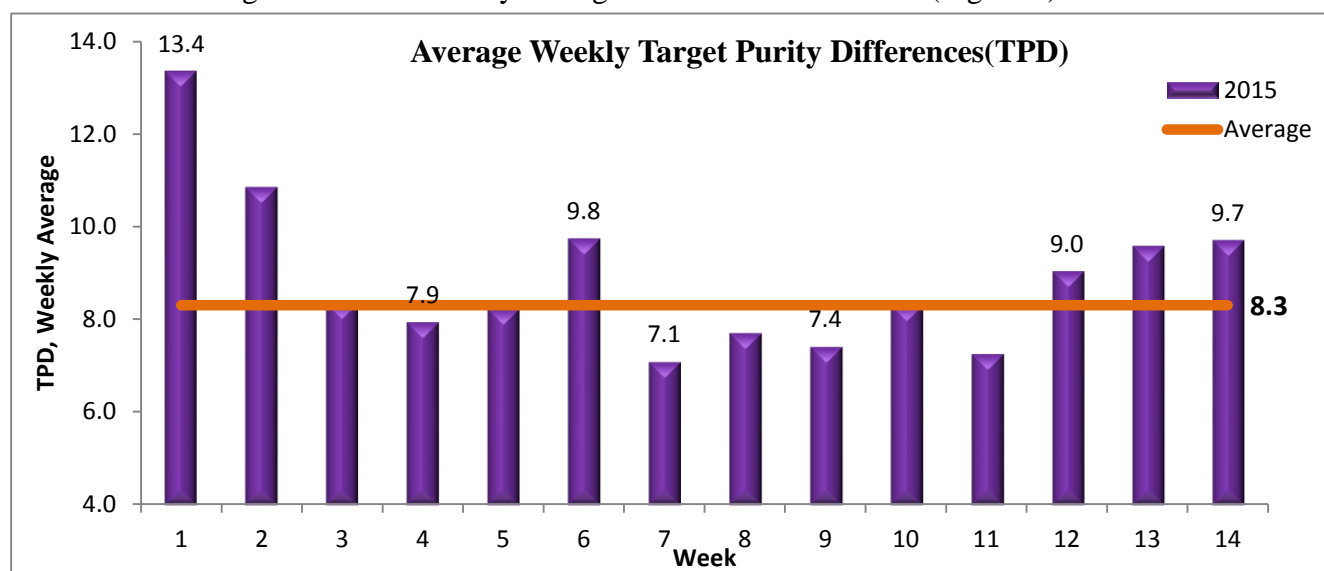
1. Refractometer Brix (ICUMSA GS4-13)
2. Sucrose, glucose and fructose by HPLC (ICUMSA GS7/4/8-23)
3. Sucrose via polarimetry\*
4. Conductivity ash (ICUMSA GS1/3/4/7/8-13)

\*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.

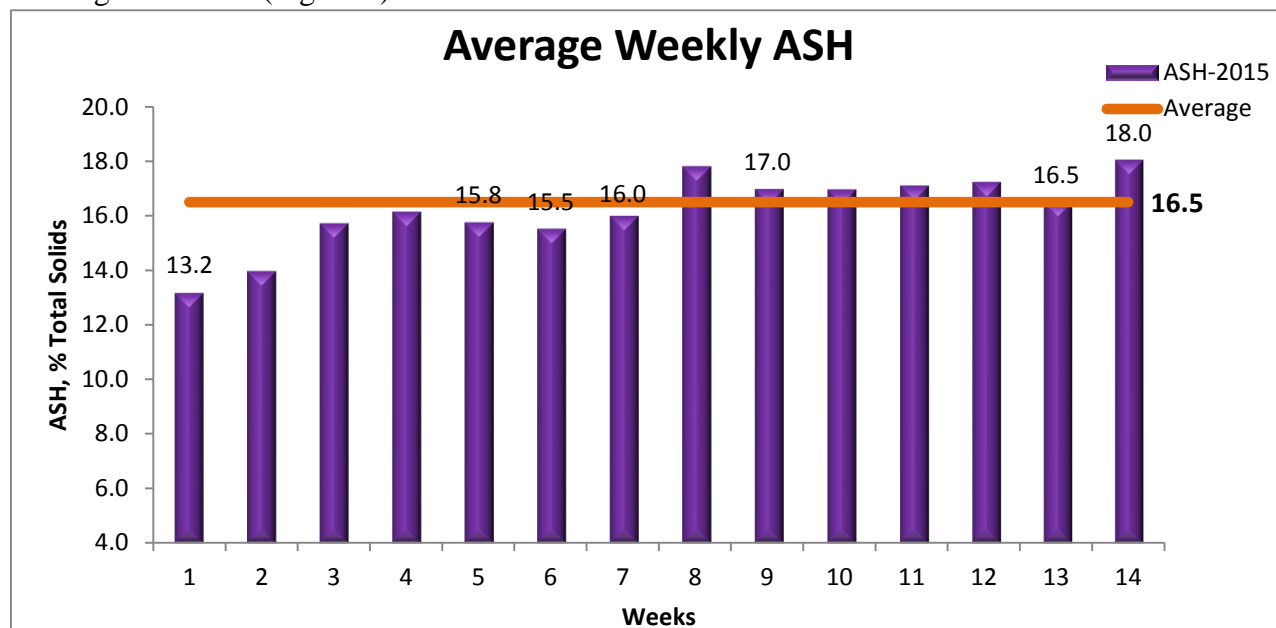
## RESULTS/DISCUSSION

The 2015 season operated for 14 weeks. The 2015 season maximum TPD weekly average was 13.4 and the minimum was 7.1. Throughout the season, the TPDs demonstrated the usual trend of decreasing TPD. The Industry average TPD for 2015 was 8.3. (Figure 1)



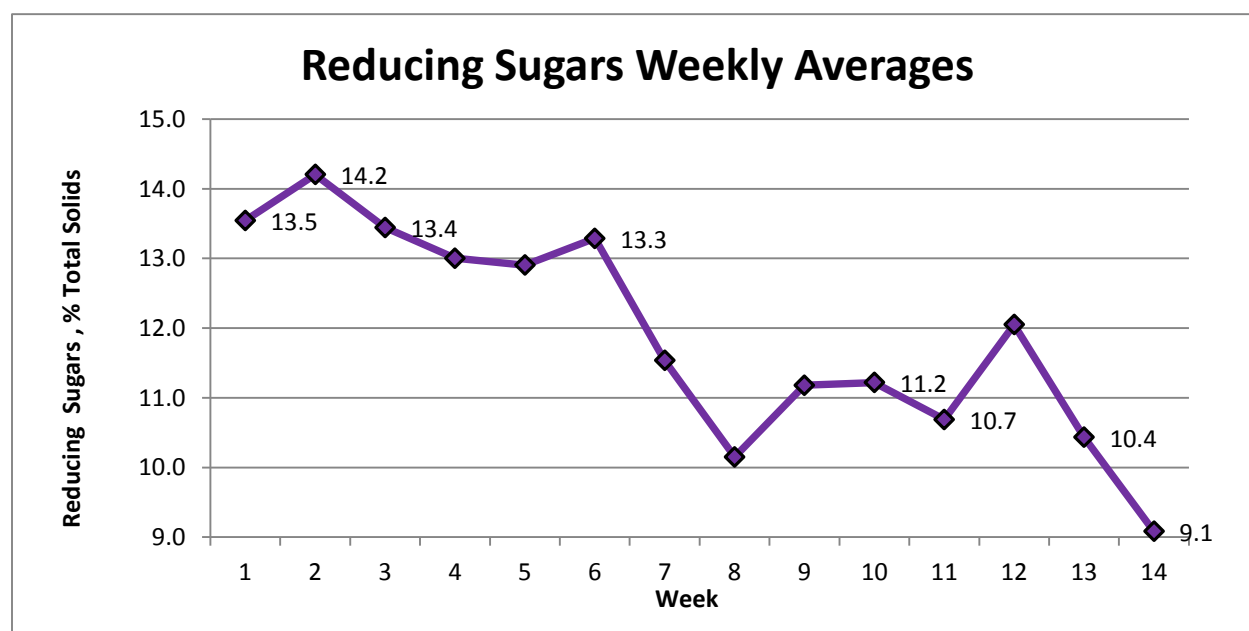
**Fig. 1** – 2015 Average Weekly Target Purity Difference

The conductivity ash component for the 2015 season started at the minimum value of 13.2. As the season continued the ash increased to the maximum value of 18.0. The conductivity ash average was 16.5. (Figure 2)



**Fig. 2 – 2015 Average Weekly Conductivity Ash**

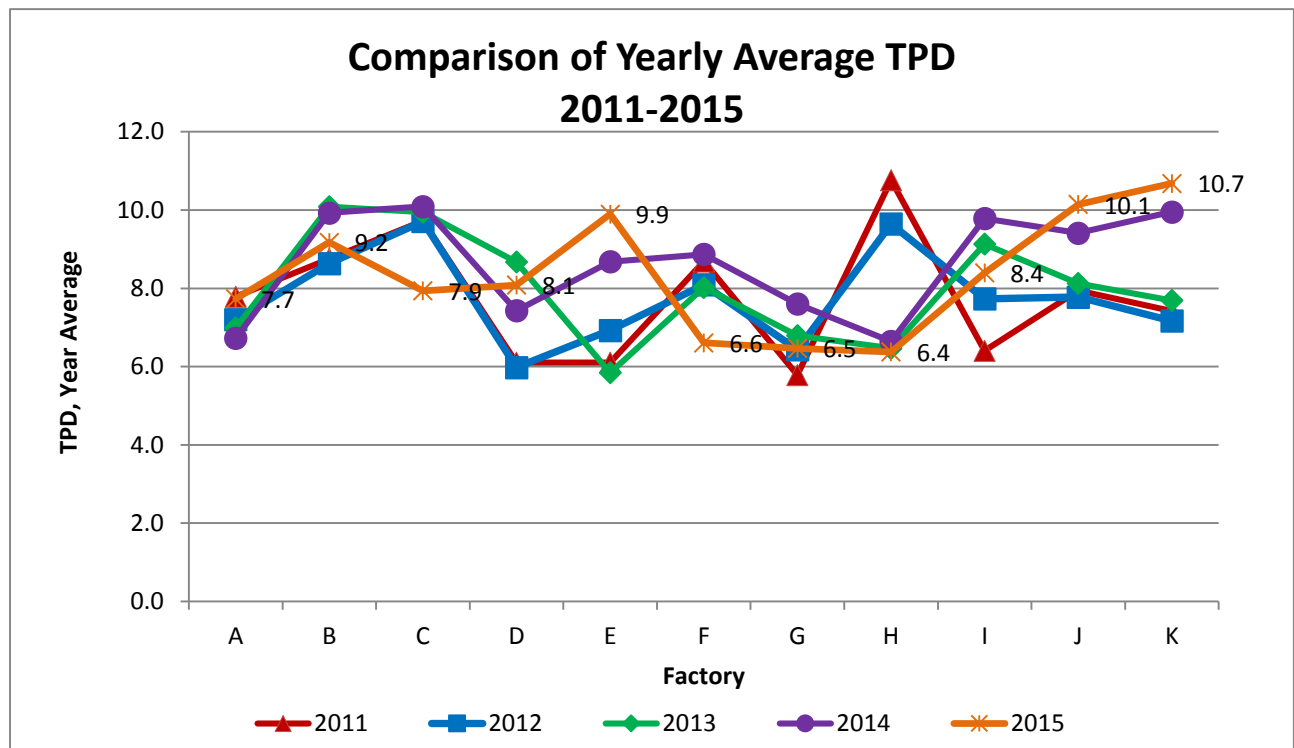
In general, there has been a significant downward trend, relative to time, in the amount of reducing sugar in final molasses. In the 2015 season, the reducing sugars was up and down throughout the season. The maximum was 14.2 and the minimum occurred at the end of 9.1. (Figure 3)



**Fig. 3 –2015 Reducing Sugars Weekly Averages**



Comparing the results from the 2015 season to the results from the 2011, 2012, 2013 and 2014 seasons showed the yearly average TPD increased. This is demonstrated in **Figure 4**. The 2015 season maximum TPD was 10.7. The minimum TPD for 2015 was 6.4. (Table 1)



**Fig. 4** – Comparison of Yearly Averages TPD 2011-2015

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
2015	6.4	10.7	8.3

**Table 1** – Summary of Yearly TPD 2011-2015

The sugar cane juice from the sugar mill has been analyzed for the past 6 years. In **Table 2**, shows the summary from the analysis. Over the last five seasons, the average brix is 14.4%, a true purity average of 88.9% and reducing sugars 5.1%.

<b>Juice Survey Summary for 2010-2014</b>			
<b>Year</b>	<b>Ref. Brix (%Juice)</b>	<b>True Purity (% Juice)</b>	<b>Reducing Sugars (%Juice)</b>
2010	14.5	89.3	3.8
2011	15.1	87.2	5.0
2012	14.6	88.1	3.6
2013	14.1	91.1	10.8
2014	14.8	88.7	4.1
2015	13.5	88.9	3.2
<b>Average</b>	<b>14.4</b>	<b>88.9</b>	<b>5.1</b>

**Table 2** – Summary of Juice Survey 2010-2015

## CONCLUSIONS

The seasonal average TPD was 8.3 for the 2015 season, which was a slight decrease from the 2014 season. The ash increased for the 2015 season to 16.5%. The reducing sugars decreased for the 2015 season to 11.9% 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 2015 season, the juice had an average brix of 14.4%, a true purity of 88.9% and reducing sugars of 5.1%.

## REFERENCES

1. Baddley Chemical. (2001). **Octapol and Octapol Plus**. <http://www.baddley.com/octapol.htm>
2. ICUMSA. (1994). **Method GS4-13: The Determination of Refractometric Dry Substance (RDS %) of Molasses – Accepted**. *ICUMSA Methods Book. Verlag Dr. Albert Bartens KG-Berlin. Supplement 2003. ISBN 3-87040-550-0.*
3. ICUMSA. (2002). **Method GS7/4/8-23: The Determination of Sucrose, Glucose and Fructose by HPLC -in Cane Molasses - Official -and Sucrose in Beet Molasses – Official**. *ICUMSA Methods Book. Verlag Dr. Albert Bartens KG-Berlin. Supplement 2003. ISBN 3-87040-550-0.*



4. ICUMSA. (1994). **Method GS1/3/4/7/8-13: The Determination of Conductivity Ash in Raw Sugar, Brown Sugar, Juice, Syrup and Molasses - Official.** *ICUMSA Methods Book. Verlag Dr. Albert Bartens KG-Berlin. Supplement 2003. ISBN 3-87040-550-0.*
5. Rein, P. (2007). **Cane Sugar Engineering.** *Verlag Dr. Albert Bartens KG-Berlin. ISBN 978-3-87040-110-8. pp. 459.*
6. Saska, M., Goudeau, S. and Andrews, L. (1999). **Molasses Exhaustion and Target Purity Formulas.** *Sug. J. 62. pp. 7, 20-24.*
7. Saska, M., Goudeau, S. and Beyene, F. (2010). **Exhaustibility of Louisiana Final Molasses and the Target Purity Formula: The 2009-2010 Season Results.** *Ann. Meet. Am. Soc. Sug. Cane Technol. La. Div. Hilton Lafayette, Feb. 1-3.*

## **Effect of Purity Drop and Crystal Size on Molasses Exhaustion in the C-Strike**

Daira Aragon\*, Iryna Tishechkina, Franz Ehrenhauser, Cy Gaudet and Tanner Faucheux  
Audubon Sugar Institute, LSU AgCenter

### **Summary**

Molasses exhaustion is one of the most important steps for sugar recovery. The comprehensive assessment of the performance of the boiling house requires the careful monitoring of purity drops, crystal content, retention time in crystallizers and crystal size, especially in the C-Strike including pans, crystallizers, reheaters and centrifugals. The information given by these measurements can be used by engineers to improve exhaustion. This paper presents a survey purity of C-massecurites, purity drops and crystal size across the C strike and final molasses purity at Louisiana sugar factories, and quantifies the effect of purity drop/rise and crystal size on final molasses purities, and crystal size on purity drop/rise.

### **Introduction**

The objective of the boiling house in a cane sugar mill is to recover the sucrose from the syrup. This is typically done in a series of steps including boiling the solution to crystallize sucrose and separating the crystals from the mother liquor in centrifugals. Molasses from the first boilings and sugar from the last boilings are usually recycled back. A commonly used boiling scheme comprises three stages. In the simpler three-boiling scheme, sugar from the third boiling, also called C-strike, is recycled into the first and second boilings (A and B strikes). Molasses from the last boiling are final, while molasses from A and B strikes are used in the B and C strikes, respectively. Raw sugar is the combination of the sucrose crystals separated from A and B strikes. Another commonly used scheme, the Double Magma, also comprises three boiling stages. Similarly to the three-boiling scheme, molasses from the last boiling are final, while molasses from A and B strikes are used within the process. However, in this scheme, both sugars from B and C strikes are recycled back and raw sugar is the product of A strike only.

The process of recovering the sugar in the boiling house is also known as molasses exhaustion. A great emphasis is put on the C strike, because it is here where the grain for A and B strikes is formed, and it is the last chance to recover sugar. Molasses exhaustion in the C strike involves the following steps:

1. Evaporation of massecurites to their maximum consistency in the vacuum pans.
2. Cooling down the massecurites in the crystallizers to crystallize any recoverable sucrose from the solution.
3. Reheating the massecurites to their saturation temperature to reduce viscosity.
4. Separation of sucrose crystals from the exhausted molasses in the centrifugals.

The purity drop of the mother liquor is an indication of the efficiency of the recovery process. It is suggested that at least 60-75% of the overall drop should occur in the pan, and the remaining in the crystallizers.

A good control of the C strike is necessary to obtain well exhausted molasses. Purities and crystal size are among the measurements that can help assessing performance and identifying potential problematic areas and, therefore, improving efficiency.

### Purities

- Massecuites and mother liquor at the pan outlet
- Mother liquor before and after reheater
- Final molasses
- Raw sugar

These purities are used to calculate crystal content of massecuites, purity drops, purity rise due to reheating, purity rise in centrifugals, efficiency in centrifugal purging and target purity difference.

### Crystal Size

- Massecuites before and after crystallizer
- C-sugar

This information can be used to evaluate if crystals fall between the expected sizes and if there is any false grain formation in the pan or crystallizers.

The analysis of the purity and crystal size data allows engineers to identify actions that can be taken to improve exhaustion, including:

- Adjusting amount of seed slurry used in graining to adjust crystal content and crystal size
- Decrease or increase boiling time to get target crystal size
- Change reheater temperatures to decrease melting of crystals
- Change temperature in crystallizers
- Adjust centrifugal wash water and/or change screens

### **Procedure**

The following samples were taken from the C strike at Louisiana sugar mills during 2015: massecuites and Nutsch molasses from the pan, after the crystallizer and after the reheater, and molasses and sugar from each centrifugal. Purities were measured for massecuites, Nutsch molasses and final molasses. Crystal size was measured for massecuites and C sugar. Figure 1 shows sampling points used. Purity drop in pan, purity drop in crystallizers, purity rise in reheater and purity rise in centrifugals were calculated. Purity of final molasses and mean crystal size of C sugar were calculated as the average for all centrifugals in operation.

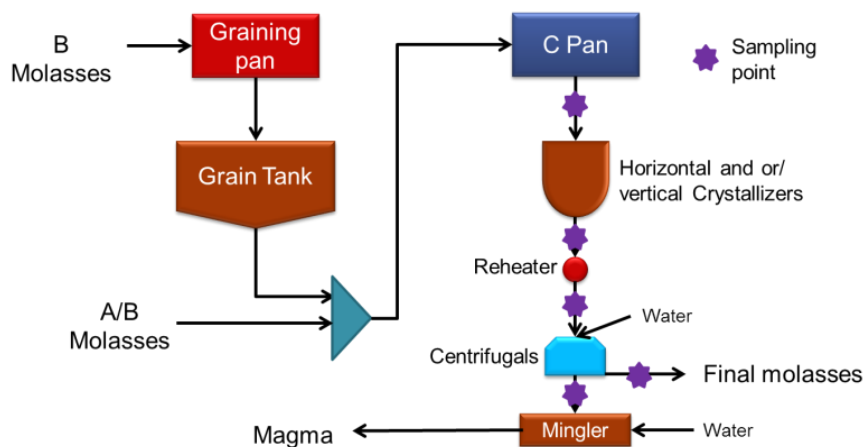


Figure 1. Sampling points in the C strike

## Results

### Boiling House Survey

Table 1 presents the summary of the purities and crystal size for the samples taken during 2015. Nutsch and final molasses purities varied up to 10.5% among factories, while massecuite purities differed up to 3.5%. For all the factories, mean crystal sizes are within 22%. Finally, the variation among purity drop from pan to reheater and the purity rise in the centrifugals goes up to 50%. The higher variation in purity drop correlates with the higher variations in molasses purities and crystal size.

Table 1. Summary of purities and crystal size

Measurement	Sample	Average	Minimum	Maximum
App. Purity (%)	C mass pan Nutsch	37.78	33.22	41.82
	C mass before reheater Nutsch	31.50	26.76	38.91
	C mass after reheater Nutsch	32.02	27.53	38.91
	Final Molasses	35.76	31.68	44.11
	C mass pan	56.42	53.96	59.88
	C mass before reheater	56.18	52.97	59.12
	C mass after reheater	56.04	52.63	58.98
Mean crystal size (microns)	C mass pan	159	122	225
	C mass before reheater	186	121	249
	C mass after reheater	185	111	229
	C sugar	203	127	271
Purity change	Pan (drop)	18.64	14.37	23.57
	Crystallizers (drop)	6.27	0.53	10.49
	Centrifugals (rise)	0.68	-0.41	2.49

C mass = C massecuites

revised 4/19/16 JK

### Effect of Purity Drop in Final Molasses Purity

Figure 2 shows the percent of the total purity drop (pan and crystallizer) that occurs in the pan and the crystallizers. Figure 2a indicates that factories in Louisiana follow the recommendation given in the literature, with at least 60% of the total drop obtained in the pan.

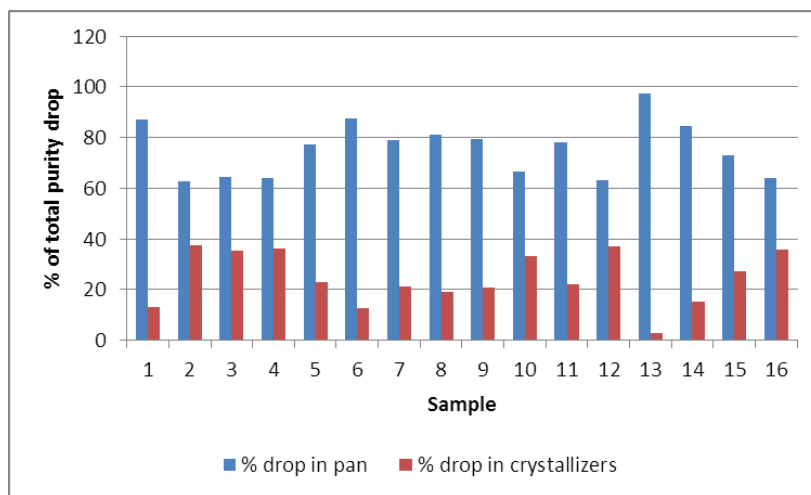


Figure 2. Percent of total purity drop occurring in pan and crystallizers

Figure 3 shows the effect of the purity drop in the final molasses purity. For each purity point dropped in the pan, final molasses purity decreased by 0.47 points (figure 3a); while for each purity point dropped in the crystallizer, final molasses purity decreases by 0.49 points (figure 3b). This finding suggests that the points dropped are important and not where it occurs (pan or crystallizers).

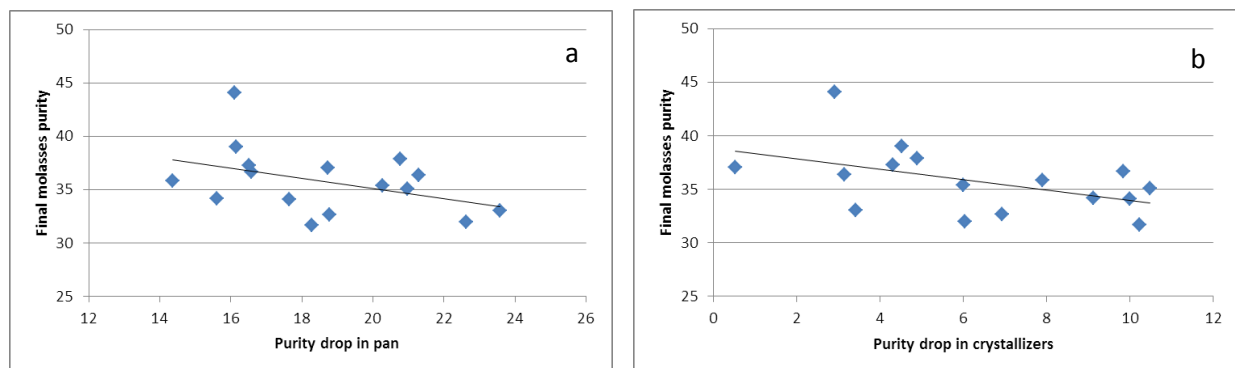


Figure 3. Effect of purity drop (a) in pan and (b) in crystallizer on final molasses purity

#### Effect of crystal size on final molasses purity

The objective of the centrifugals is to separate the crystal out of the massecuite. Smaller crystals will likely pass through the screens into the final molasses. Figure 4 shows that in most samples, the mean crystal size of the C sugar was larger than massecuites. In a few cases, the C sugar had smaller crystal size than the massecuite, indicating high dissolution or breakage of crystals.

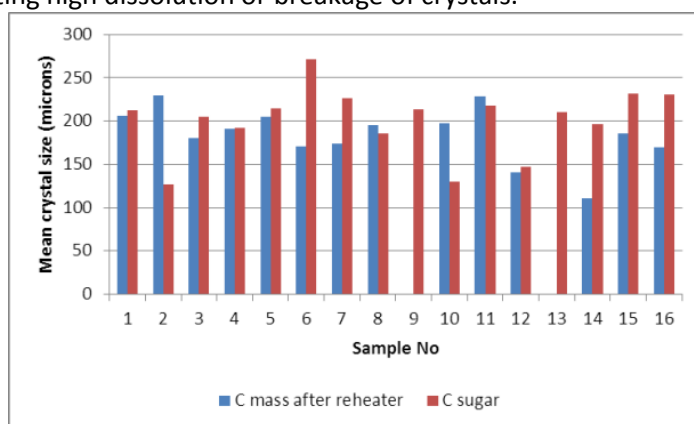


Figure 4. Mean crystal size of C massecuites after reheater and C sugar

Figure 5 shows the effect of the mean crystal size of massecuite on the purity rise in the centrifugals, and the effect of crystal content on the final molasses purity. The trend in figure 5a indicates that higher crystal sizes in massecuites correspond to lower purity rise; for every 10 microns increase in crystal size, the purity rise decreased by 0.1 points. Purity rise of less than 3 points was not obtained with smaller crystal sizes (<150 microns). On the other hand, increasing the crystal content in of the massecuites by 1% decreased the final molasses purity by 0.58 points, as shown in figure 5b.

It is worth noting that during normal operation, there are many factors influencing the purging efficiency at the centrifugals. The values presented here are only an indication of the effects on final molasses purity and purity rise in the centrifugals. Conditions found in each individual mill such as high viscosity massecuites, crystal elongation, among others, may affect the final molasses purity in different ways.

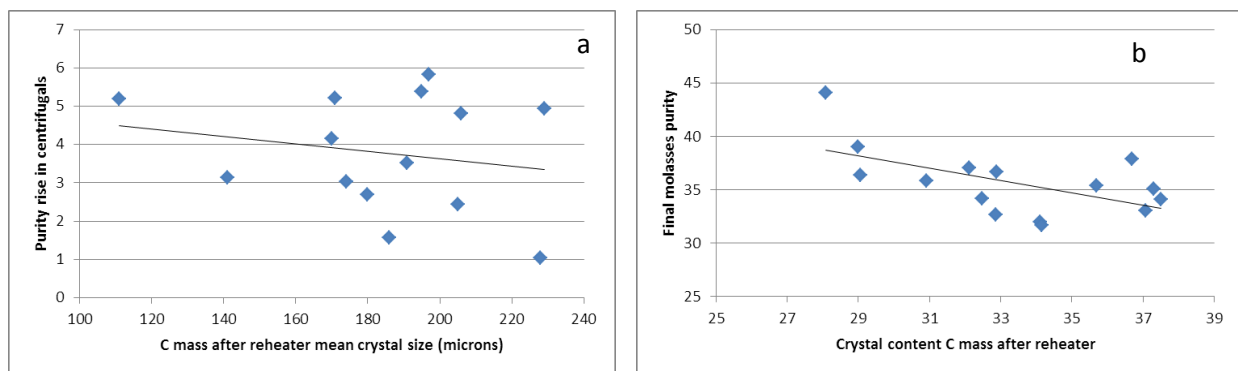


Figure 5. (a)Purity rise in centrifugals vs. mean crystal size of C massecuite after reheater and (b)final molasses purity vs. crystal content of C massecuite afte reheater

## Conclusions

Data taken in Louisiana sugar factories during 2015 indicate that reducing purity drop in pan or crystallizers, may reduce the final molasses purity by 0.47-0.49 points. Increasing the crystal size of C massecuites by 10 microns, may reduce the purity rise across centrifugals by 0.1 points, while increasing the crystal content of C massecuites by 1% may reduce the final molasses purity by 0.58 points.

It is clear that purity drop in pan and crystallizers, and crystal size of massecuites have great effects on the purity rise in the centrifugals and the final molasses purities. Therefore, the successful control of the C strike requires careful monitoring of these measures to get highly exhausted molasses. The results presented here are an indication of these effects. Nonetheless, experiments under controlled conditions are necessary to evaluate isolated effects and individual trends in each factory.

## Acknowledgments

American Sugar Cane League for providing funding for this project  
All sugar factories in Louisiana and their personnel for the support during sampling

## **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.

Samples were collected at all raw sugar factories over the 2014 and 2015 crops 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 A, B and final molasses, B sugar, and C sugar as well as filtrate juice from the filters. For typical factories, the color of all major streams are shown on a solids basis in Figures 4, 6 and 8 and on a non-pol solids basis in Figures 5, 7 and 9, respectively. The average data for all factories is shown in Figure 10 on a solids basis and in Figure 11 on a non-pol solids basis.

Mixed juice color at all factories is shown in Figure 12 on a solids basis and in Figure 13 on a non-pol basis. Similarly, sugar color is shown in Figure 14 (solids basis) and in Figure 15 (non-pol solids basis). Figure 16 shows both mixed juice and sugar color on a non-pol solids basis.

Figures 17, 18 and 19 show the color of the A, B and C sugars together with the corresponding molasses on a non-pol solids basis.

The following sources of color increase were considered: boiling of strikes, C massecuite in crystallizers and pan steamout. Massecuite color in and out of crystallizers is shown in Figure 20 on a solids basis and in Figure 21 on a non-pol solids basis. The residence time in C crystallizers contributed to an approximate 1% increase in color measured. The color of A, B and C strikes and their corresponding steamouts is shown in Figure 22 (solids basis) and Figure 23 (non-pol solids basis). There was an increase in color of about 11% per strike boiled while the color increase due to pan steamout was negligible (about 6% on 0.25-2% of strike solids).

Results indicate:

1. The color of the non-pol solids (impurities) in the mixed juice entering the factory varied from 114,000 to 245,000 and averaged 177,000.
2. The color of the non-pol solids in the sugar varied from 20,000 to 616,000 and averaged 301,000. This indicates that color increased on average about 70% through the processing operations.
3. The color of the non-pol solids in the B sugar and B molasses were similar, as was the color of the non-pol solids in the C sugar and final molasses. In the case of the As, the A sugar was somewhat higher color than the corresponding A molasses, but this may be due to the difficulty in accurately determining the small quantity of non-pol solids in the A sugar.
4. Filtrate juice typically had the lowest color non-pol solids.
5. Typical color generated was as follows:

a. Clarification	10-15%
b. Evaporators	5-25%
c. Vacuum pans	$\approx$ 33%
d. Crystallizers	$\approx$ 1%
e. Pan steamout	<u>Negligible</u>
Total Factory	70%

We would like to acknowledge and thank the American Sugar Cane League for funding this project and all of the sugar factories, and especially their chief chemists, for the time and help generously given to us.



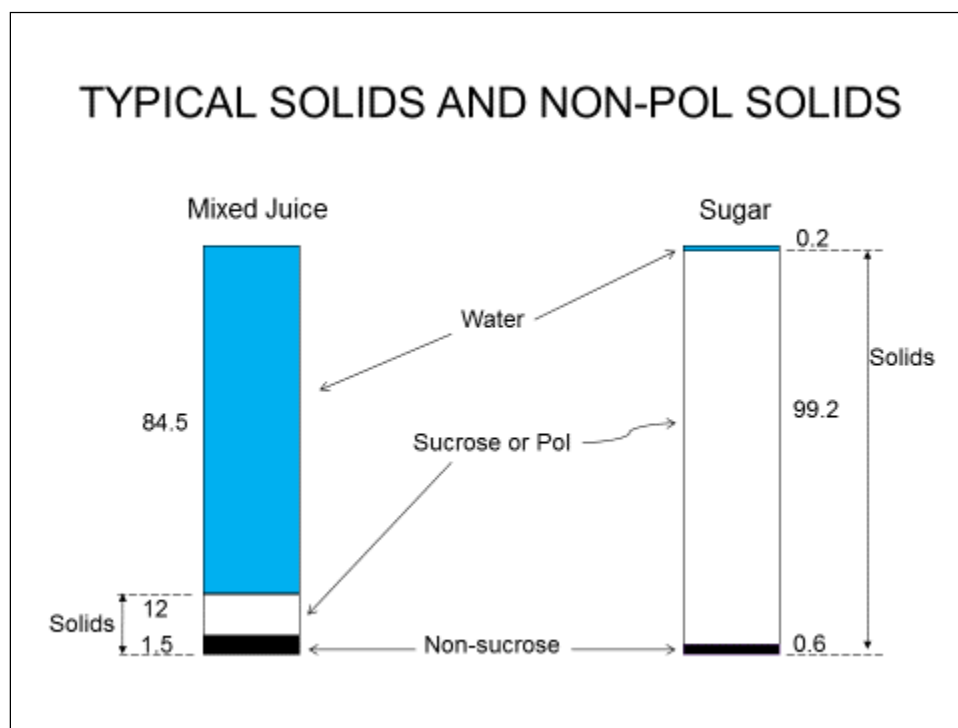


Figure 1. Typical solids and non-pol solids in mixed juice and sugar.

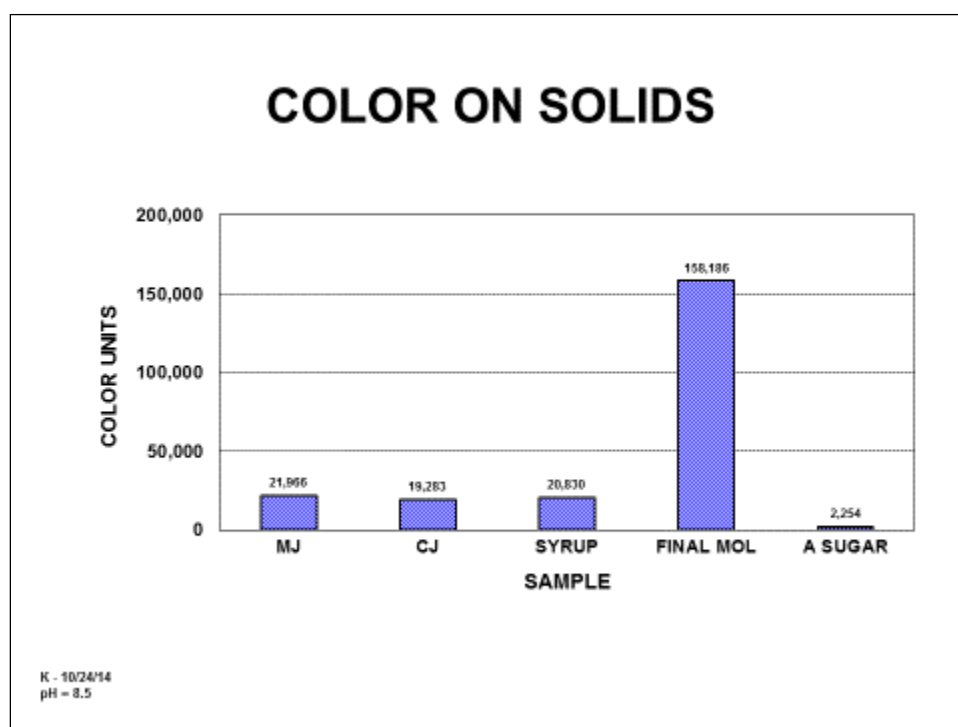


Figure 2. Color on solids for samples from Factory K.

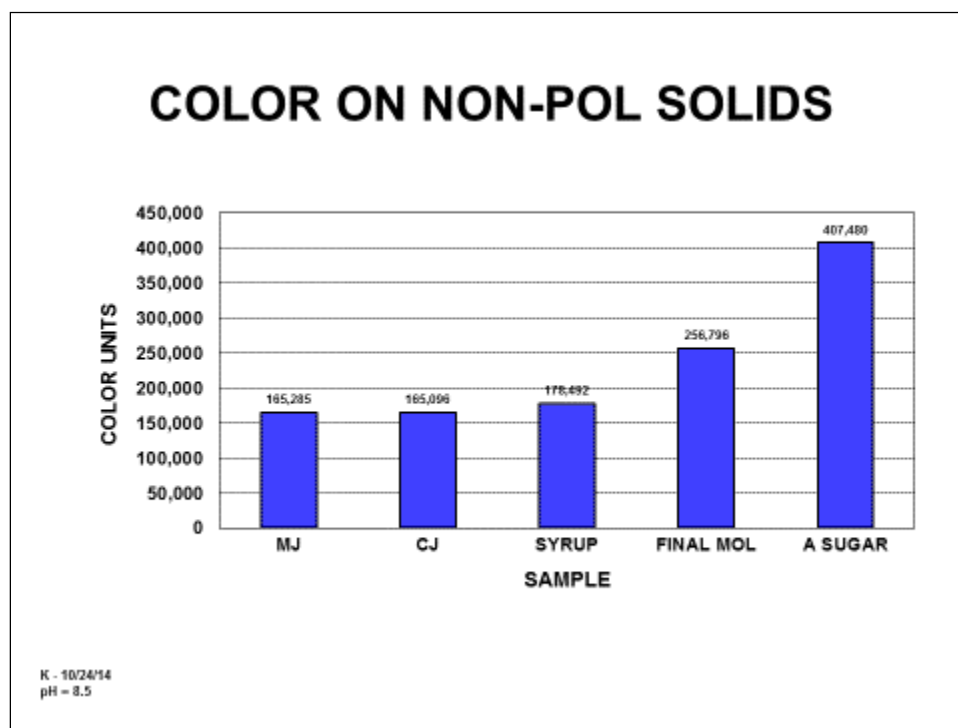


Figure 3. Color on non-pol solids for samples from Factory K.

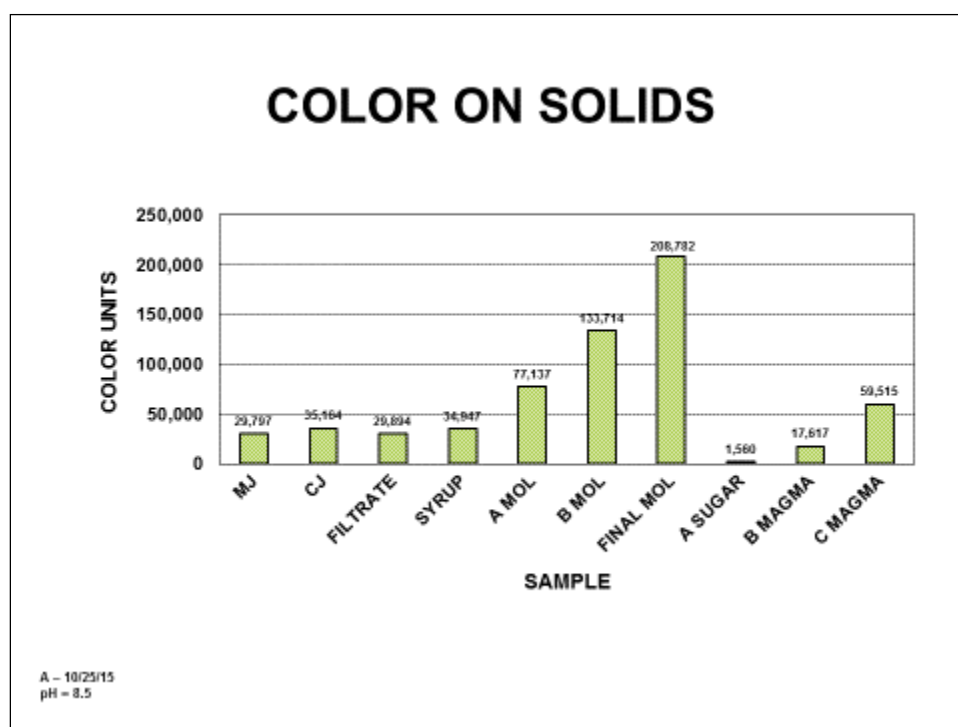


Figure 4. Color on solids for samples from Factory A.

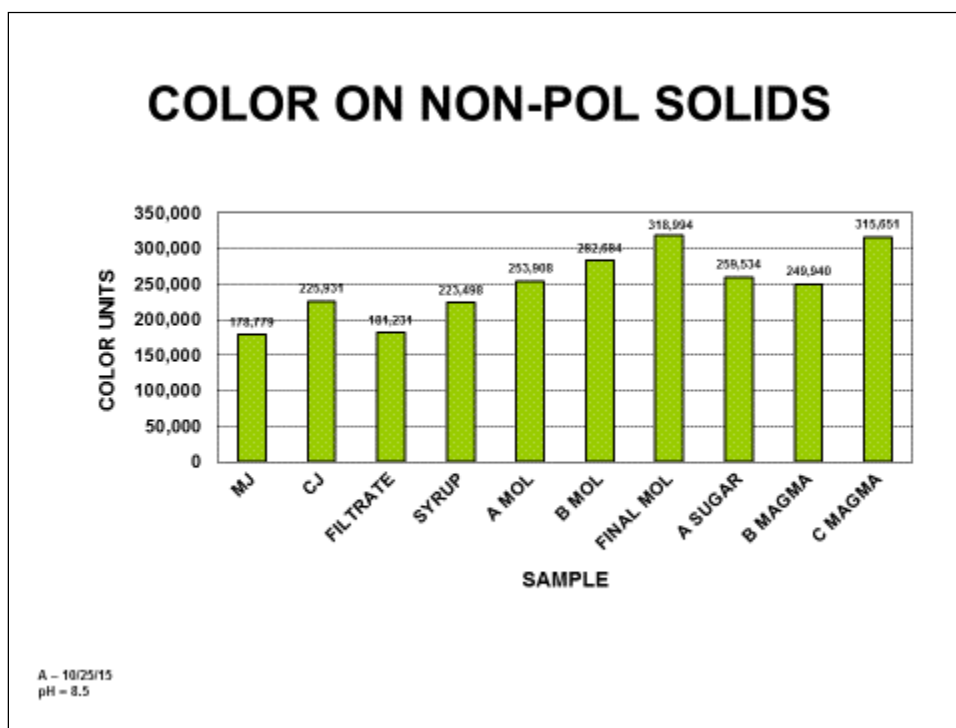


Figure 5. Color on non-pol solids for samples from Factory A.

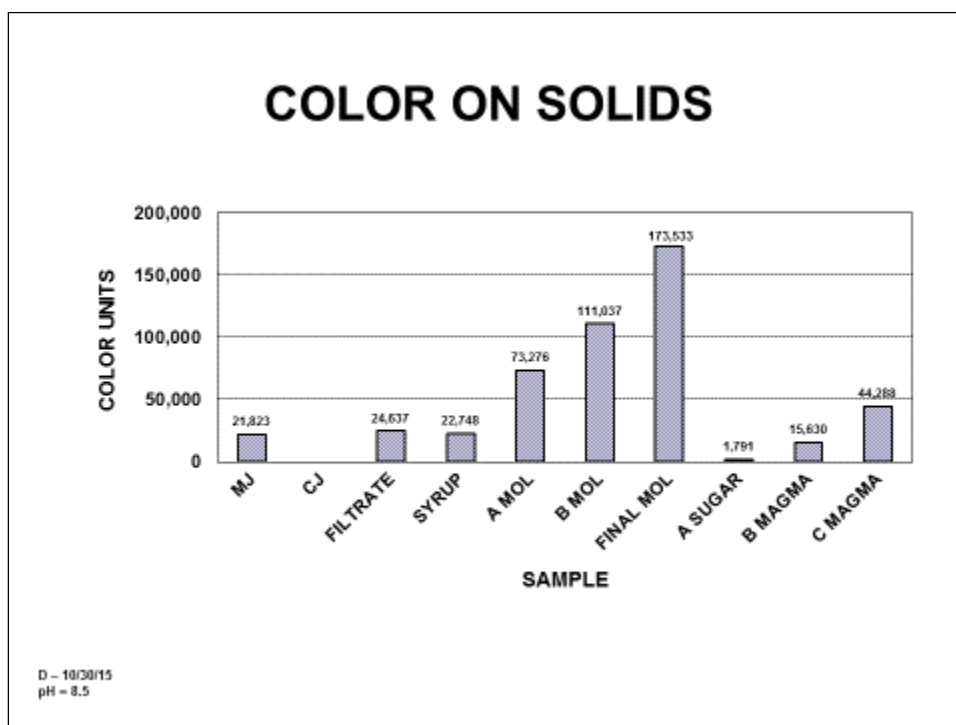


Figure 6. Color on solids for samples from Factory D.

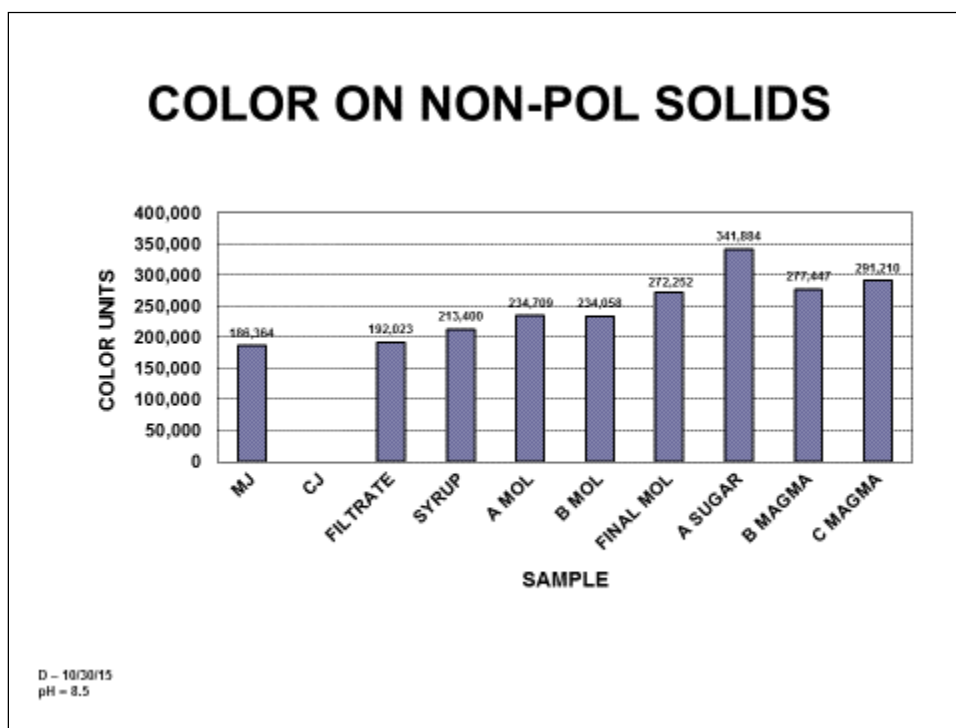


Figure 7. Color on non-pol solids for samples from Factory D.

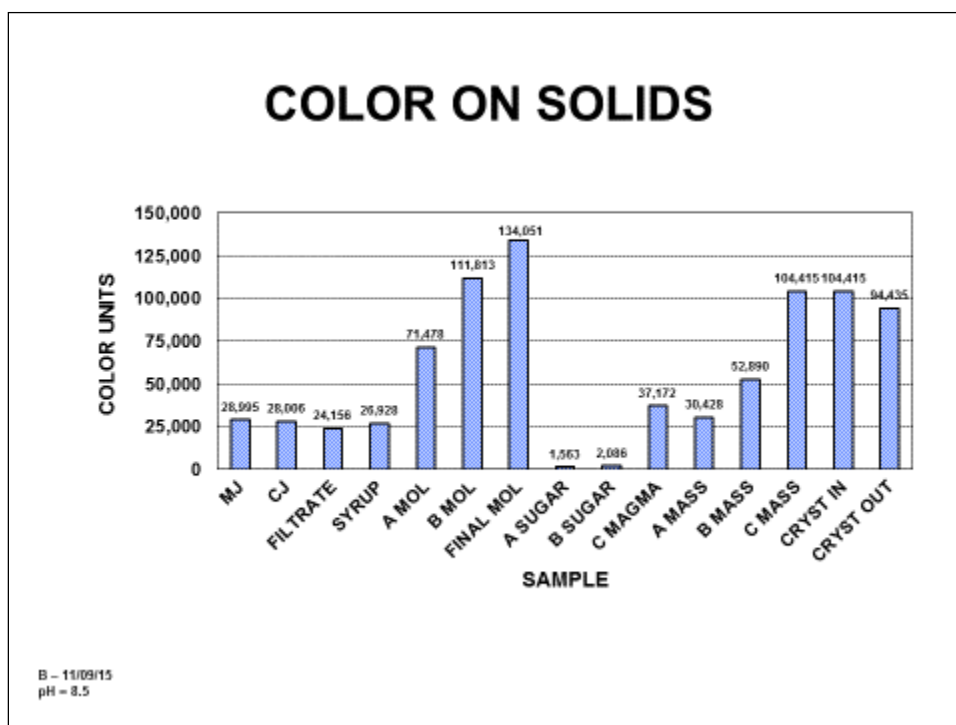


Figure 8. Color on solids for samples from Factory B.

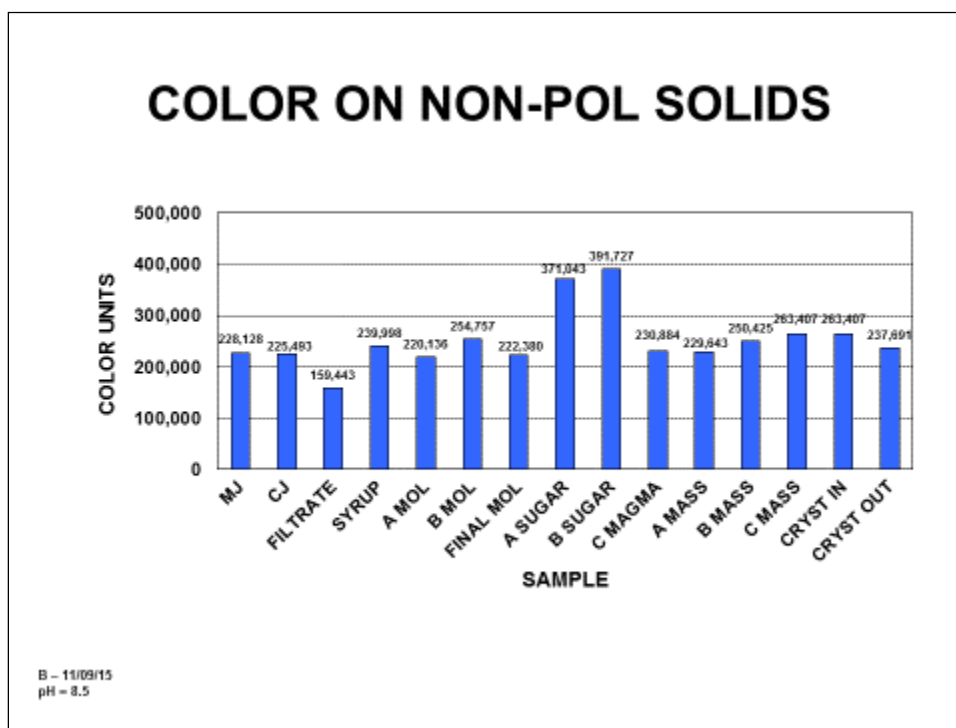


Figure 9. Color on non-pol solids for samples from Factory B.

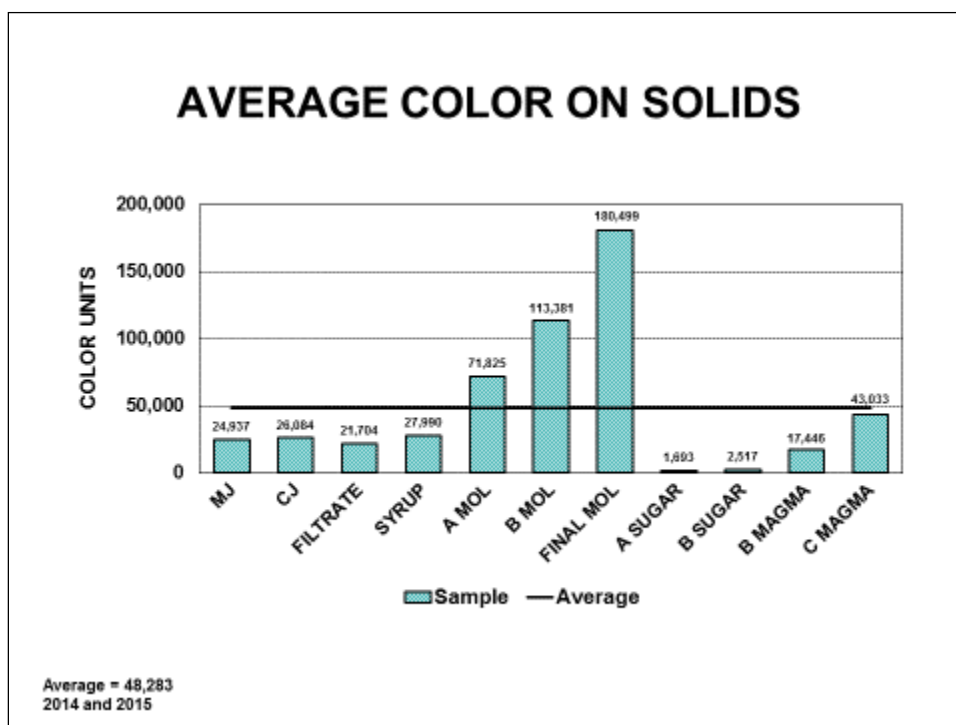


Figure 10. Average color on solids for samples from all factories.

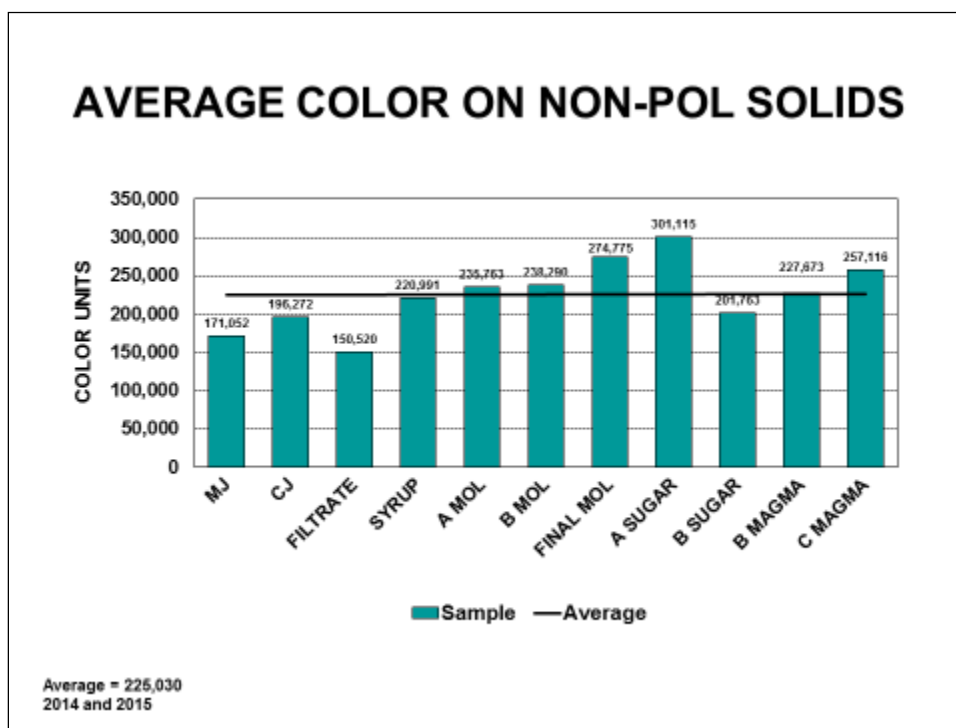


Figure 11. Average color on non-pol solids for samples from all factories.

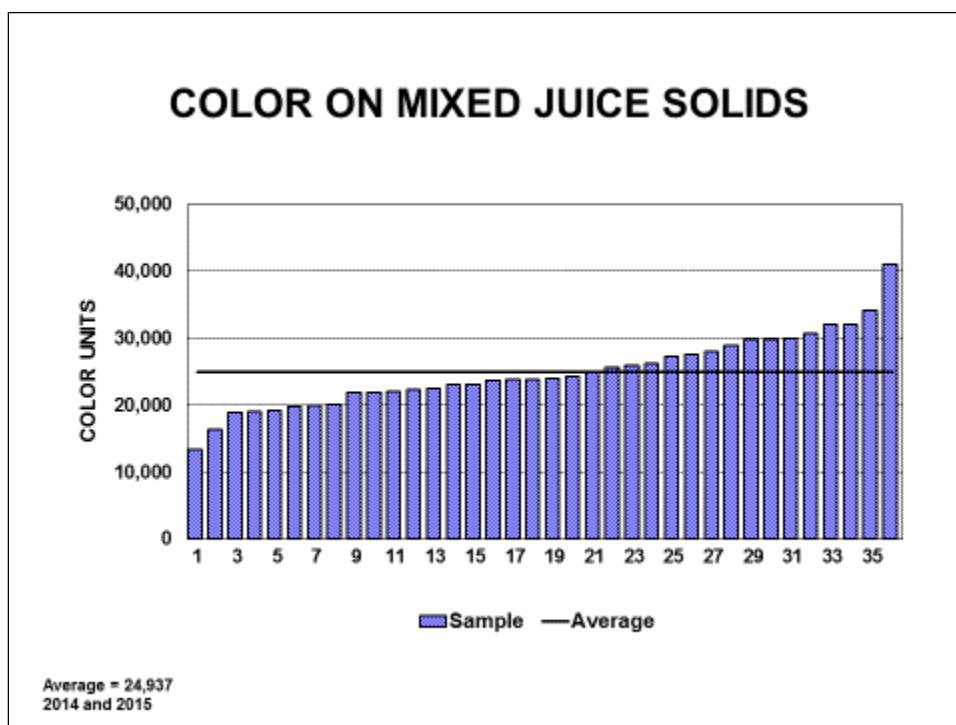


Figure 12. Color on mixed juice solids for samples from all factories.

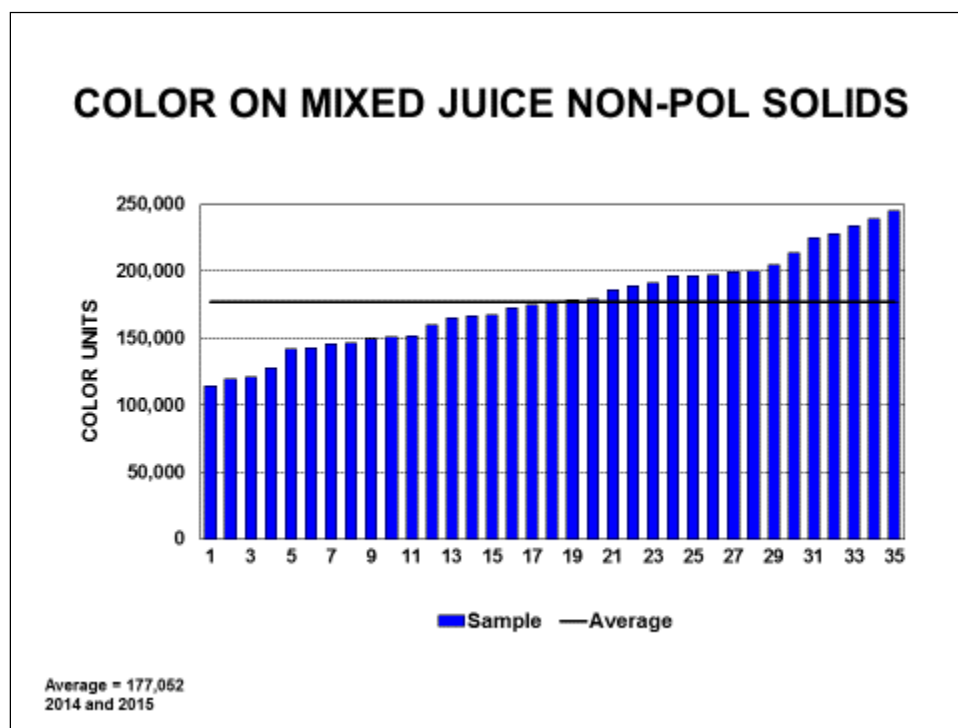


Figure 13. Color on mixed juice non-pol solids for samples from all factories.

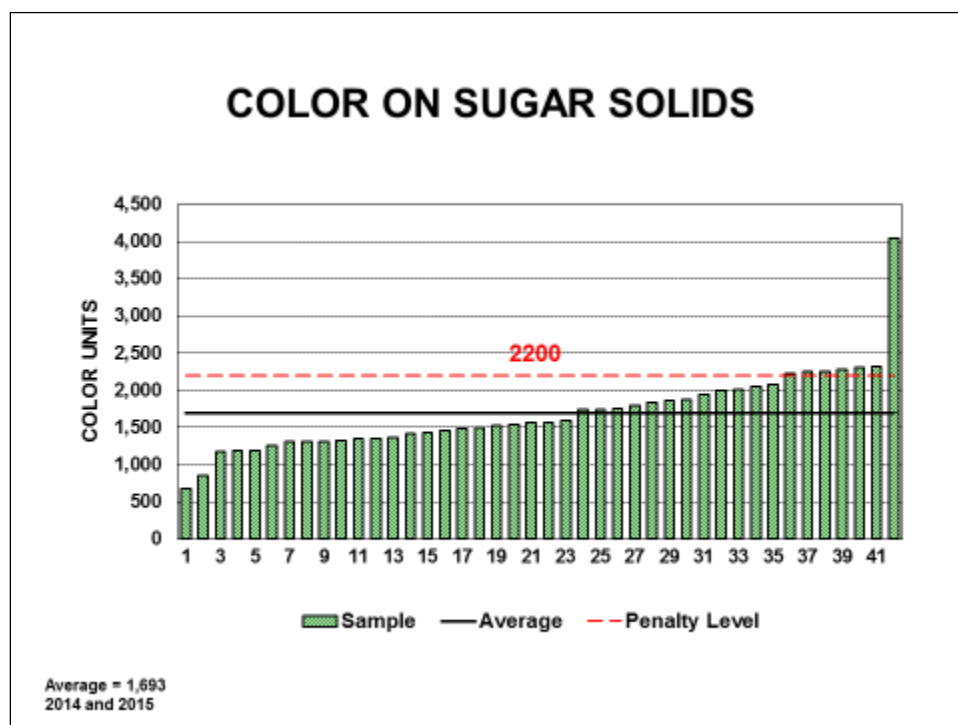


Figure 14. Color on solids in sugar samples from all factories.

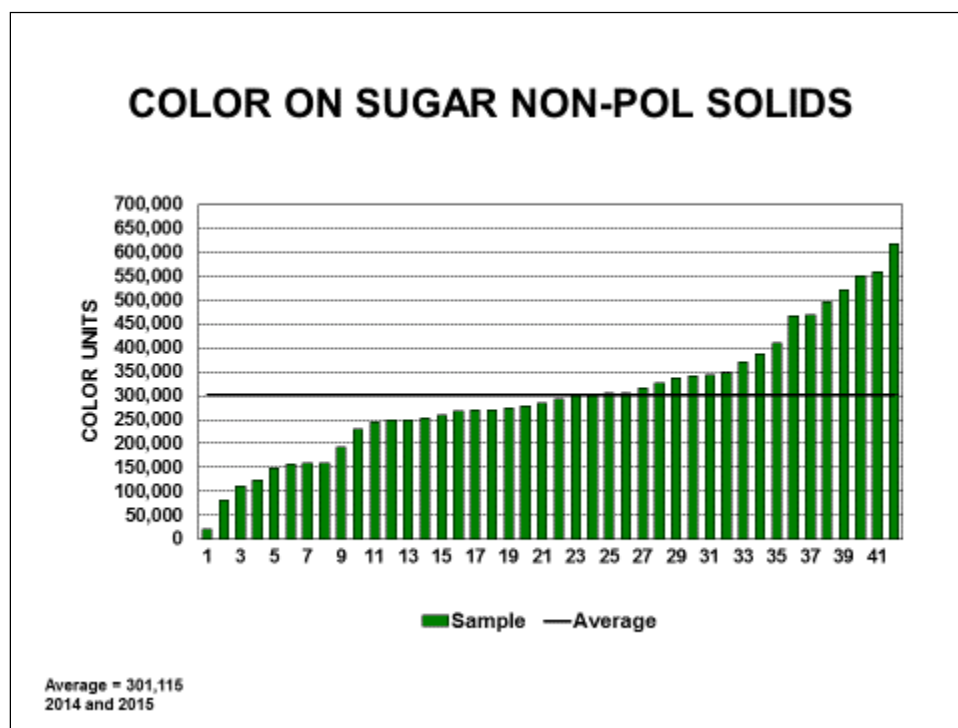


Figure 15. Color on non-pol solids in sugar samples from all factories.

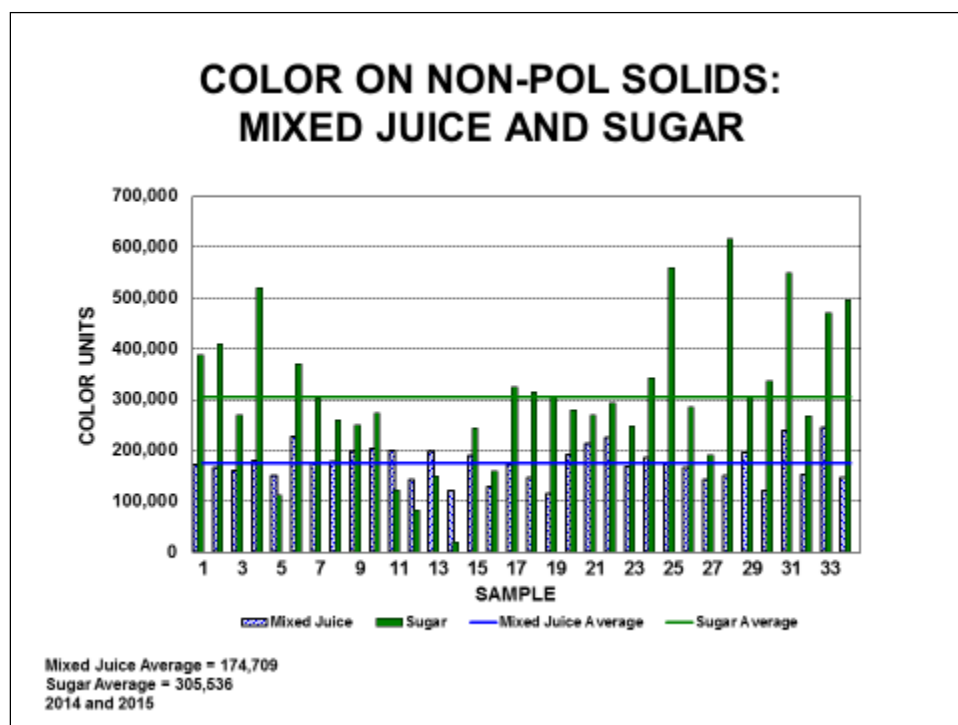


Figure 16. Color on non-pol solids of mixed juice and sugar.



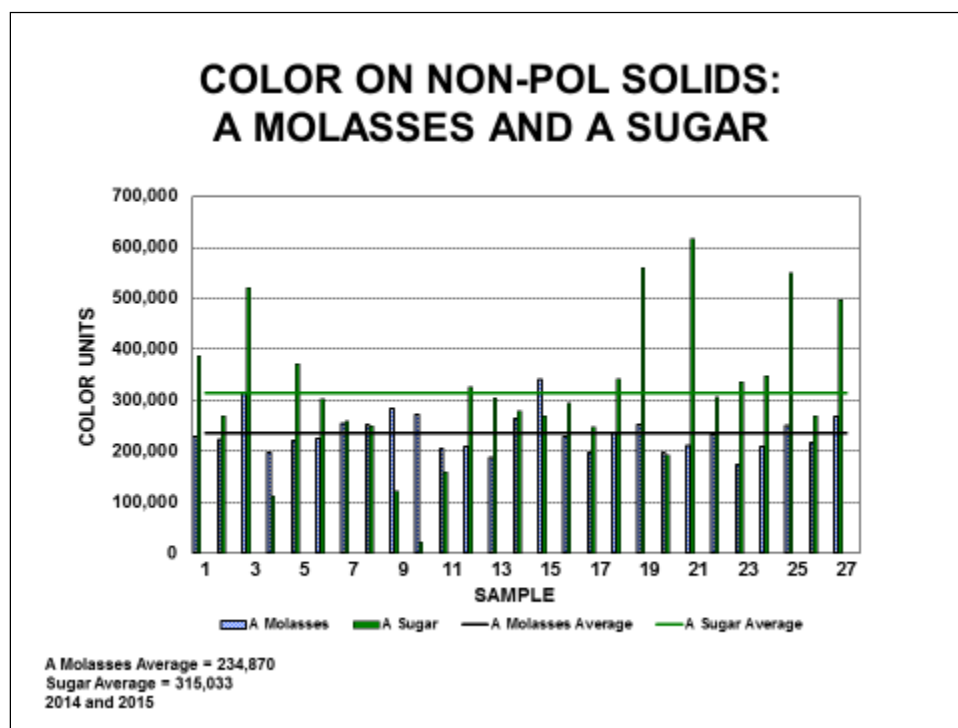


Figure 17. Color on solids of A molasses and A sugar.

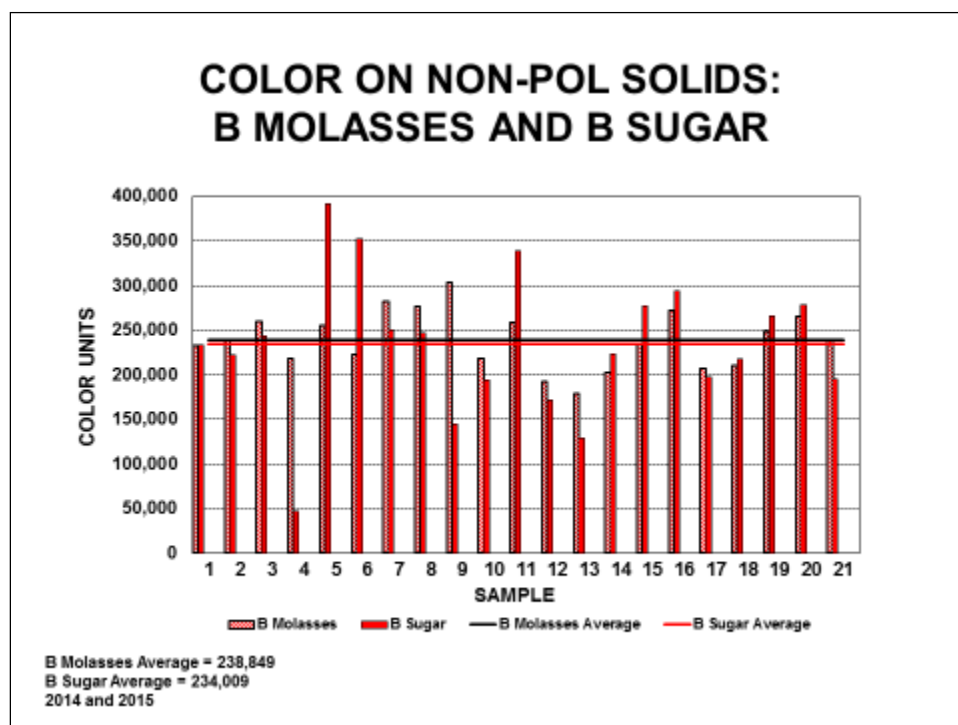


Figure 18. Color on non-pol solids of B molasses and B sugar.

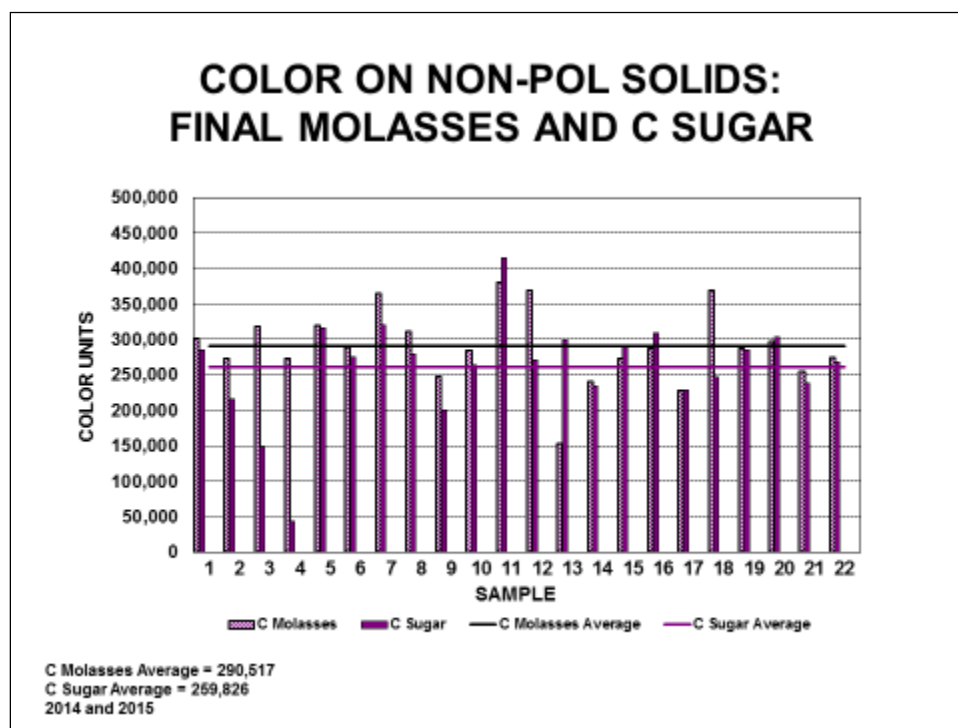


Figure 19. Color on non-pol solids of final molasses and C sugar/C magma.

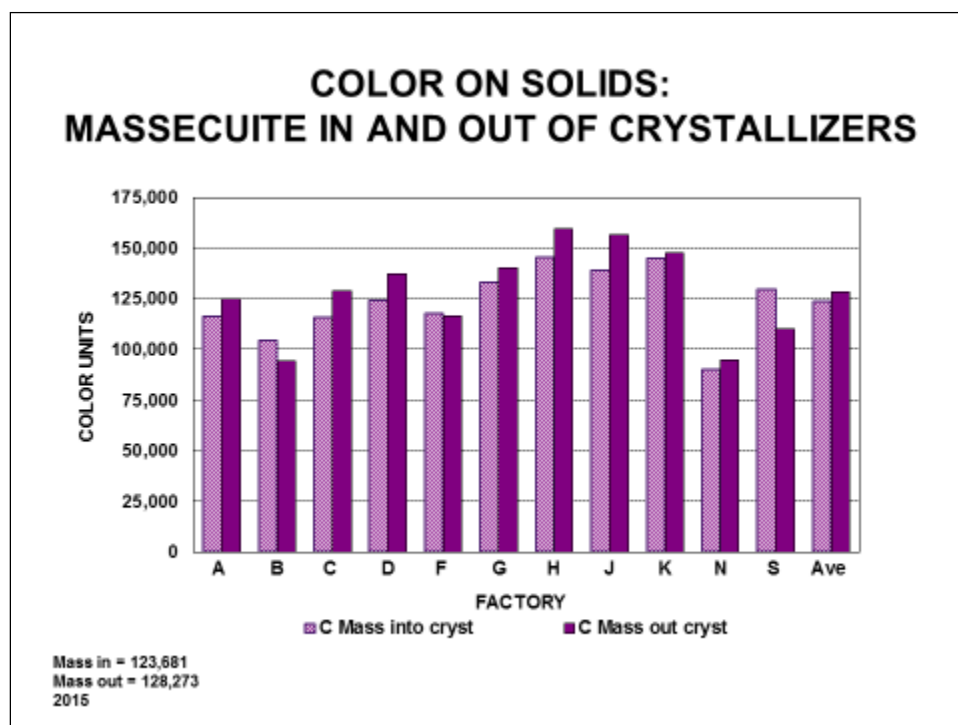


Figure 20. Color on solids of massecuite in and out of crystallizers.

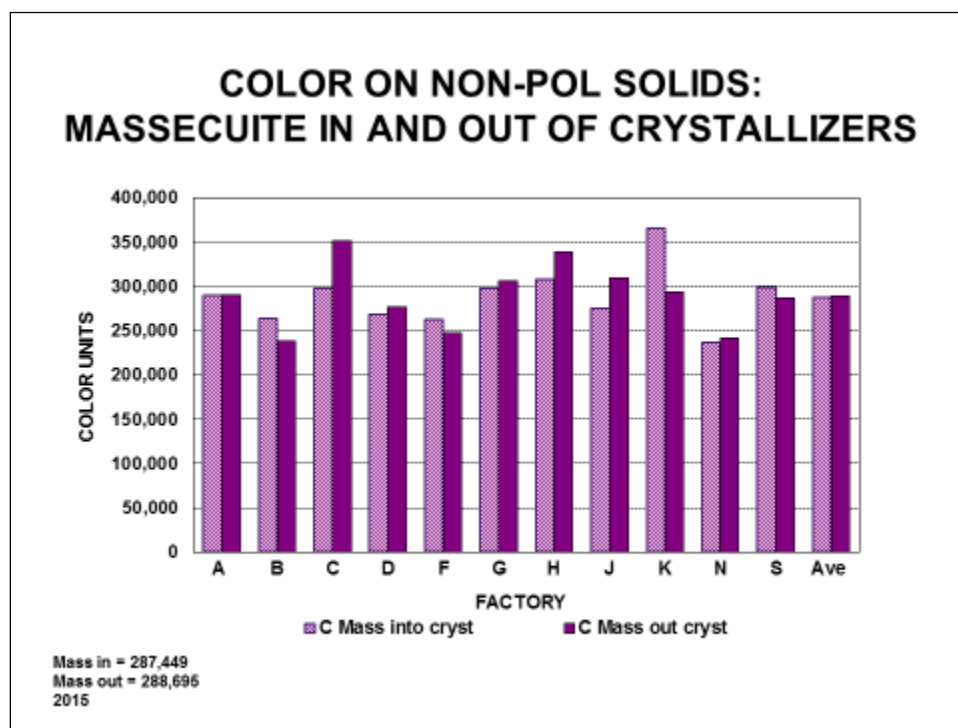


Figure 21. Color on non-pol solids of massecuite in and out of crystallizers.

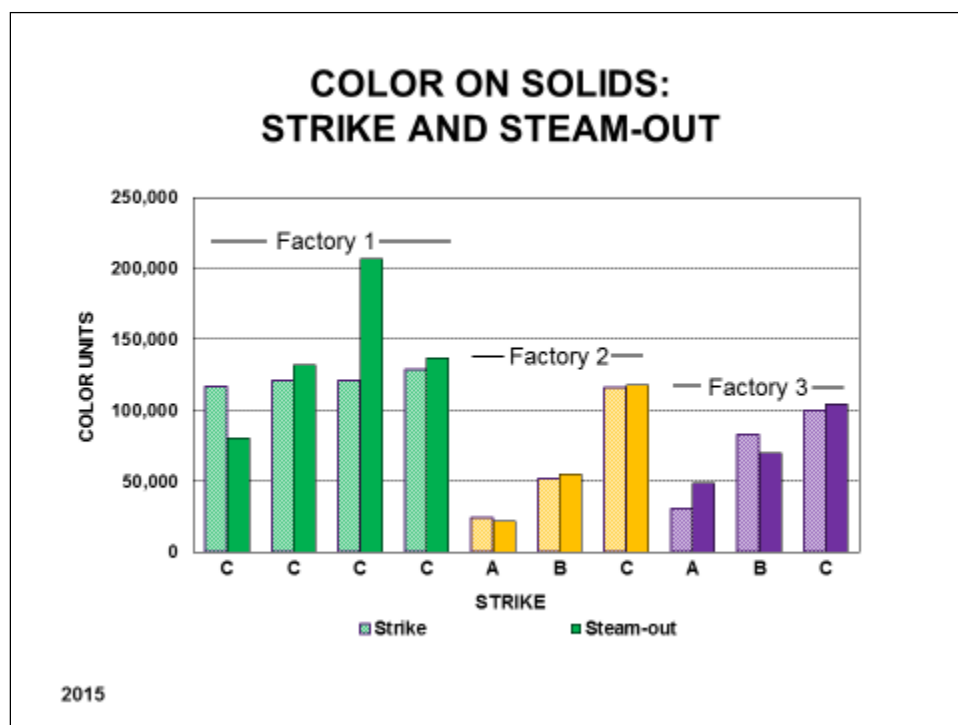


Figure 22. Color on solids on A, B and C strikes and corresponding steamouts at three factories.

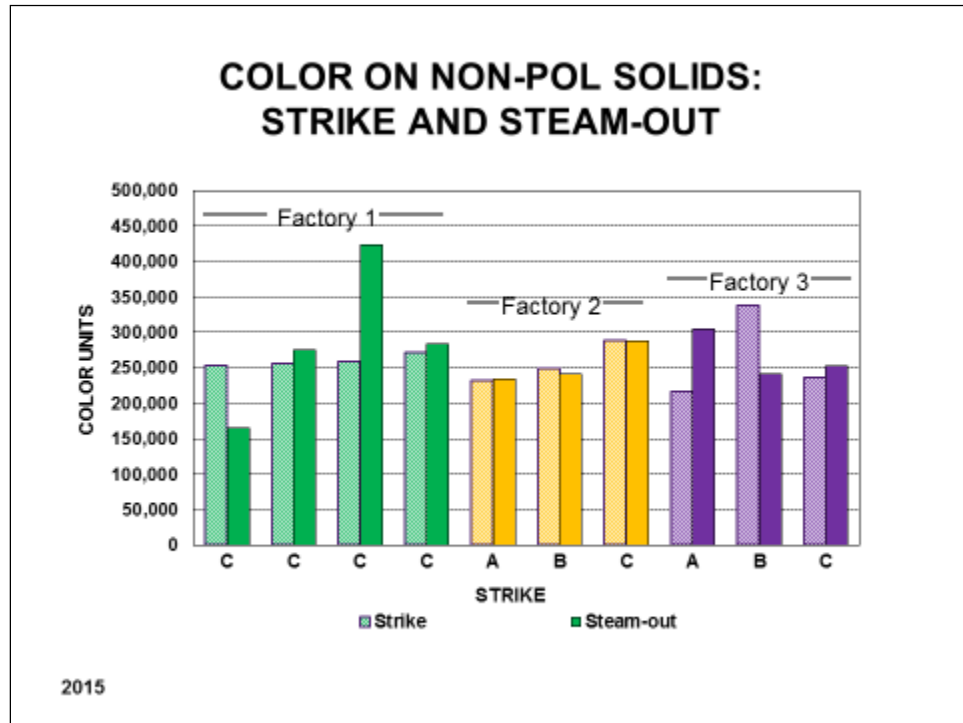


Figure 23. Color on non-pol solids of A, B and C strikes and corresponding Steamouts at three factories.

## SYRUP STORAGE

*Stephanie Linares, Franz Ehrenhauser*

### INTRODUCTION

Sugarcane syrup in Louisiana could serve as feedstock for year-round operating biorefineries, and storing syrup could extend boiling house capacity in raw sugar mills. Six month syrup storage is already practiced in the beet sugar industry to increase the capacity of the factory [1]. Raw sugar mills may be coupled with biorefineries to create raw sugar, fuels, and chemicals from sugarcane, with syrup associating these two industries together.

Storage of sugarcane syrup for raw sugar production comes with certain challenges that do not affect the biofuel and chemical industry. The sugar industry must store syrup to ultimately maintain the highest purity, or sucrose for raw sugar production. The biofuel and chemical industry on the other hand, must ultimately store fermentable sugars (sucrose, glucose and fructose) and thus does not worry about inversion of sucrose into glucose and fructose. A shared issue for both biofuel and sugar industry is that syrup storage must overcome sugar loss due to biological degradation in tanks.

### MAIN PARAMETERS TO COMBAT MICROBIAL DEGRADATION

Biological degradation is caused by microbes present within the syrup that consume sugars for their metabolism. Microbial degradation is the major cause of fermentable sugar loss and inversion in storage of syrup. Cane syrups leaving the evaporators have a microbial flora consisting of bacterial spores and heat-resistant yeast cells [2]. These spores can germinate under suitable conditions leading to major sugar losses. The main parameters to consider for storing syrup are water activity, pH, and temperature. Water activity ( $a_w$ ) is the ratio between the vapor pressure of the syrup when it is completely undisturbed and in balance with the surrounding air media ( $p$ ), and the vapor pressure of pure water under identical conditions ( $p_0$ ) [3].

$$a_w = \frac{p}{p_0}$$

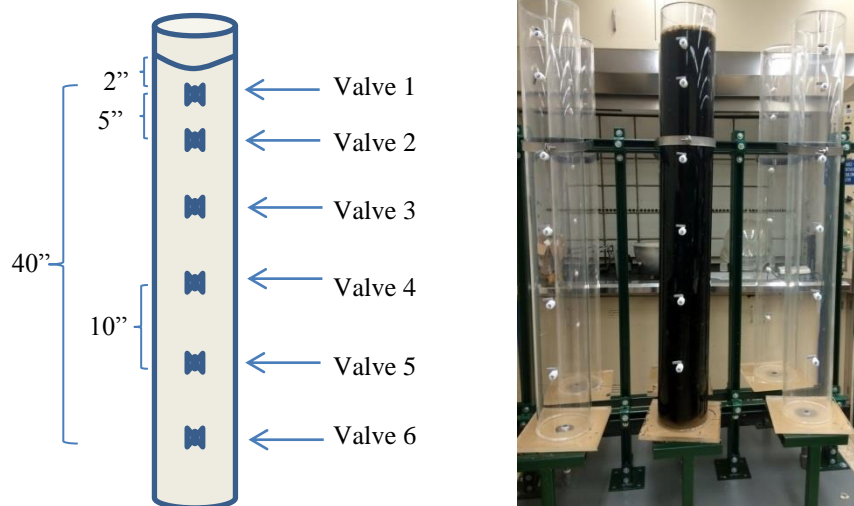
Bacteriostatic or biocidal effects are seen for most microorganisms in the following conditions: water activity  $< 0.6$  [4], 67-69°Bx, pHs greater than 8 [1, 5] or lower than 2 [2], and temperatures below 30°C [5]. A high refractometric dry substance (RDS) content would signify a low amount of water activity in the syrup. High microbial growth can occur at the syrup surface layer caused by tank headspace condensation which increases water activity; moreover, it enables spore germination. By maintaining a water activity below 0.6 [4] (high dry solids content of the syrup), microbial growth is limited or prevented. Literature states a minimum of 67.5°Bx for beet thick juice must be maintained for long term storage [5-7]. Besides water activity, temperature, and pH, purity and conductivity ash are also important parameters.

Analysis of fermentable sugars and true purity was conducted via high performance liquid chromatography (HPLC) in order to determine how much sucrose, glucose, and fructose is present in any given sample. Similarly, samples should also be measured periodically for conductivity ash.

Some storage methods require treatments to the syrup which involve increasing conductivity ash, i.e. applying caustic to preserve beet thick juice at pH higher than 9. Raising conductivity ash raises costs during downstream processing to sugar, thus should be avoided.

## RESULTS

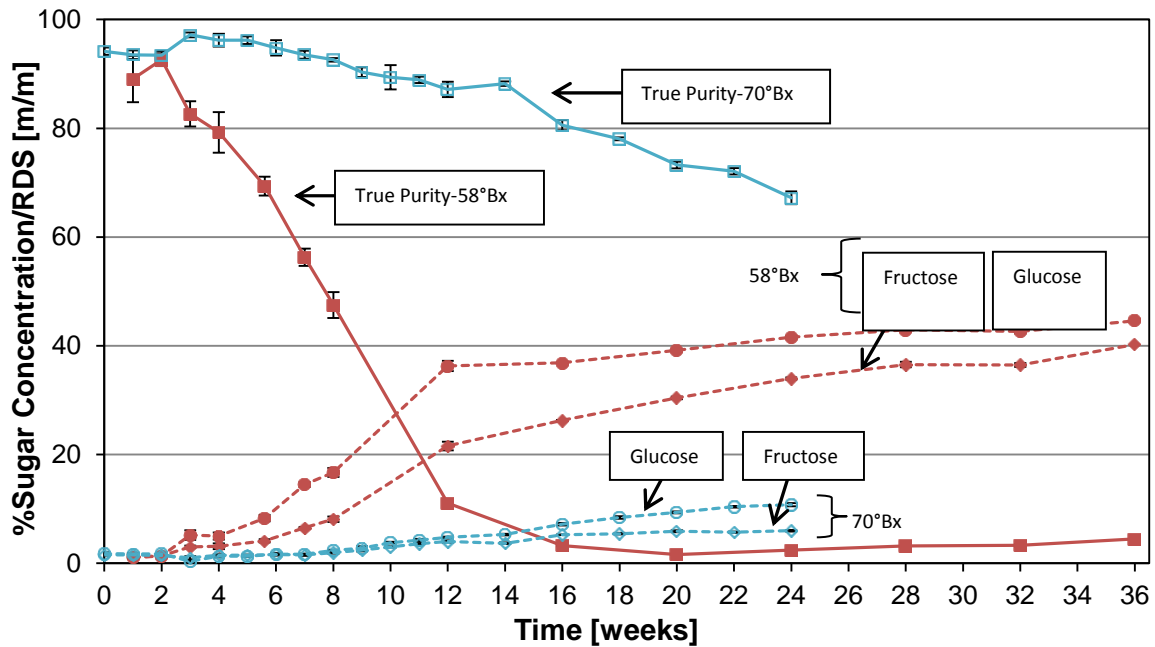
Sugarcane syrup at 58°Bx and at 70°Bx was used to evaluate sucrose, glucose, and fructose loss during a 36 and 24 week storage test, respectively. Syrup was stored in a tank-simulating, acrylic glass column with six valves at various depths of the column as shown in Figure 1. Samples were taken from each column to determine if degradation occurred at different rates at various depths of the column.



**Figure 1.** Storage columns

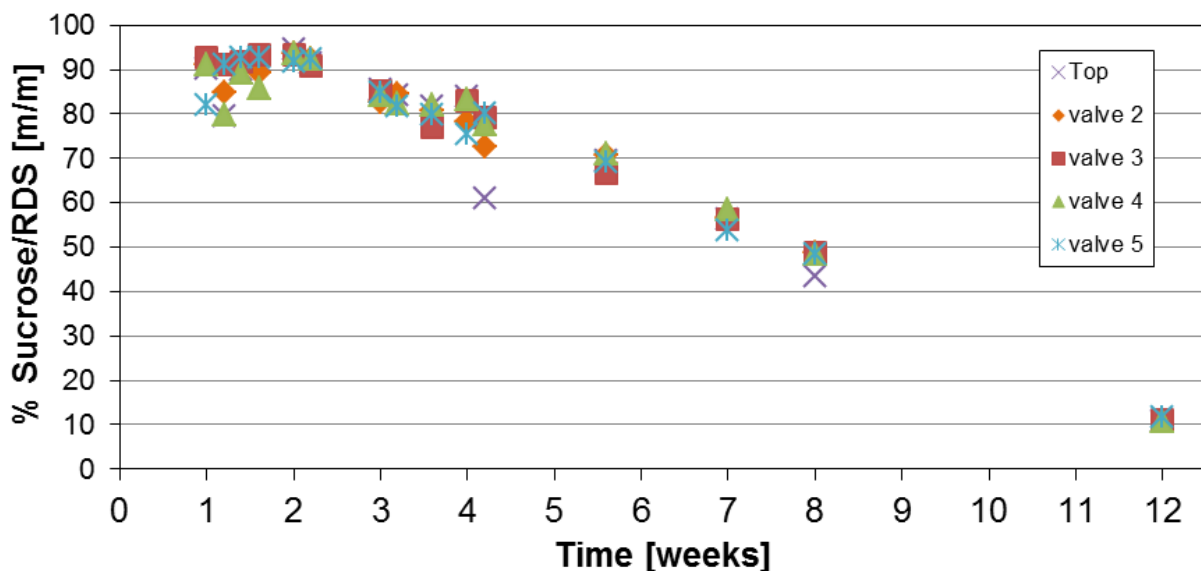
For the 58°Bx storage experiment, the syrup volume did not exceed the “valve 1” location. For this column only, values for “valve 2” are from “valve 3” in the schematic, values for “valve 4” are from “valve 5” in the schematic, and so on. Thus, when evaluating results for this column, data only appears for up to “valve 5.” All “valve 1” or “top” values were taken 2 inches from the surface of the syrup.

Both syrups were left untreated to evaluate degradation at a low and high RDS content. The average true purity, glucose/RDS, and fructose/RDS concentrations from the samples are seen in Figure 2. It is apparent that 70°Bx syrup stores longer than 58°Bx syrup, which was expected. There is no statistically significant change in purity for the 58°Bx syrup in the first two weeks of storage. At the third week of storage, mold began to grow on the surface of the 58°Bx syrup which could explain the dramatic drop in purity at week 3. At week 3 there was a total of 7.8% loss of purity. The 70°Bx syrup showed a much slower and gradual loss in purity over time. To reach the same loss in purity as the 58°Bx syrup, it took approximately 15 weeks. Storing at 70°Bx caused some crystallization both at the bottom of the column and at the syrup surface, which is of concern in an industrial setting. The RDS content for both syrups did not change significantly, and neither did the conductivity ash. Conductivity ash was not expected to change as nothing was added to the syrup. pH decreased from 6.5 to 6 in the 58°Bx syrup and from 6.5 to 5.7 in the 70°Bx syrup.



**Figure 2.** Sucrose, glucose, and fructose degradation over time for 58°Bx and 70°Bx syrup

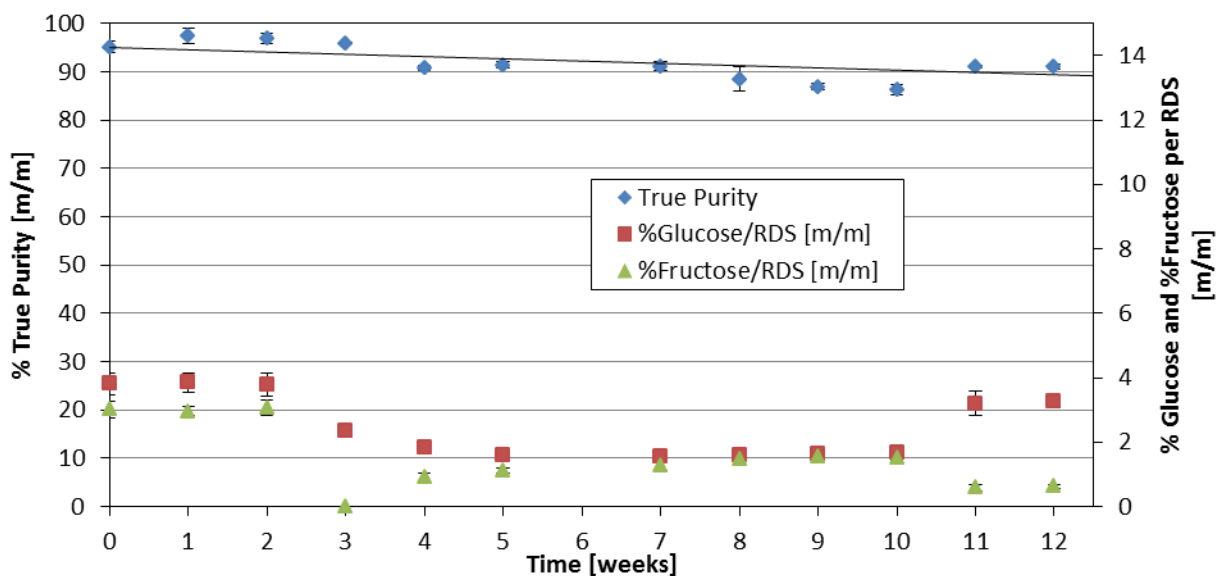
To analyze whether degradation occurred at different rates depending on depth, samples were taken from different depths of the column. From these samples, we can analyze if degradation is greater at the surface or if it occurs throughout the entire bulk of the syrup column. As evidenced by Figure 3, there is no significant difference in true purity between valves for the 58°Bx syrup. Left untreated, syrup will begin to degrade equally throughout the entire column over time, even with mold growing on surface, which was observed by the beginning of the third week. If syrup is to be stored for longer than two weeks, the bulk of the tank must be treated to deter microbial infection. Considering syrup at both RDS contents have more than 10% purity loss at six months, further investigation on possible treatments for syrups is warranted.



**Figure 3.** True purity decrease in 58° Bx syrup over time

Previous results established that bulk degradation occurs in untreated syrup; however, further experimentation was needed to see if a treated syrup column would also degrade in bulk. Two experiments were conducted to evaluate syrup degradation, one with a surface treatment and one with a bulk treatment. The first treatment involved using propylene glycolate, a mixture of 1,2-propanediol (propylene glycol) and caustic soda[8], to act as a surface barrier between the syrup surface and the surrounding air. This type of treatment was demonstrated in beet thick juice in Brawley, CA during the months of April-September [8]. Their thick juice was stored at 70°Bx, pH >9, and 25°C. The storage tanks were ellipsoidal, and held a capacity of 1.8 million gallons. A spray nozzle was used to apply a seal to the juice surface with a 2 mm thickness. After 6 months of storage, pH remained nearly the same, and the quality of the thick juice remained stable except for two tanks which showed a rise in color. The industrial success of this treatment warranted further investigation on sugarcane syrup.

A 68°Bx syrup column was stored at 20°C for 12 weeks. A surface treatment of propylene glycolate was applied to the syrup surface at the beginning of the experimental trial. Figure 4 describes the average % change in true purity, glucose/RDS, and fructose/RDS over time in a column treated with propylene glycolate. The surface treatment alone does not protect the entire column from purity loss. Though the propylene glycol remained afloat on the syrup surface during the storage period, the caustic dissolved in the syrup raising the pH of the bulk syrup from 6.5 to 8.5 in 9 weeks. The amount of caustic dissolved into the bulk of the column was not sufficient to prevent bulk degradation. There was no difference between the different valve depths, which is consistent with previous results of having bulk degradation in syrup. Another experiment was conducted where a bulk treatment was applied.

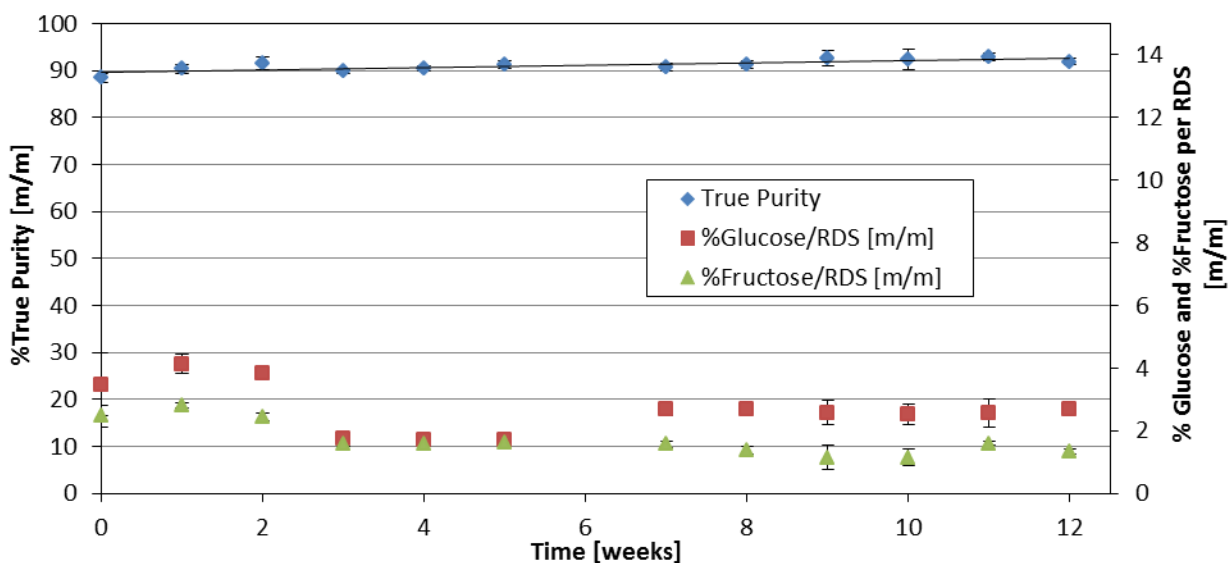


**Figure 4.** Propylene glycolate surface treatment on sugarcane syrup

The purpose of the bulk treatment experiment was to investigate if a bulk treatment alone could be sufficient to contain true purity through the storage period. Formula 31-A biocide (Garratt Callahan) was utilized to treat an entire syrup column for 12 weeks at a 400 ppm concentration. During this period, no statistically significant change in true purity was detected. The data from the experiment are seen in Figure 5. pH changed from 6.5 to 6. Signs of microbial activity were



observed by rising CO<sub>2</sub> bubbles in the column that caused foaming at the surface after 3 weeks of storage. However, the microbial activity was not significant enough to measure a change in purity. No major difference in degradation was measured between the different valve depths. Purity was maintained in the bulk of the column with the biocide treatment for at least 12 weeks at 20°C without a surface barrier.



**Figure 5.** Biocide bulk treatment in sugarcane syrup

## CONCLUSION

Syrup storage can expand the capacity of sugar mills while coupling the industry with biorefineries. High brix syrup (> 68°Bx) will store longer than low brix syrup; however, if left untreated both succumb to microbial degradation over time. Syrup must be treated in bulk to ensure purity for storage, surface treatment alone is not sufficient. Using biocide as a bulk treatment is considered a promising option to maintain true purity and total fermentable sugars in syrup for long term syrup storage in Louisiana.

## ACKNOWLEDGEMENTS

The authors would like to thank the faculty and staff at the Audubon Sugar Institute for their support and help during research. We would also like to thank Mr. Robert Hatch (Organic Defoamer Group, WY) with his assistance on providing technical information from his work and the Louisiana sugar mills for providing sugarcane syrup for this investigation. This project was supported by the Agriculture and Food Research Initiative Competitive Grant no. 2011-69005-30515 from the USDA National Institute of Food and Agriculture.

## REFERENCES

1. Asadi, M., *Beet Sugar Handbook*. 2006, Hoboken, New Jersey: John Wiley & Sons, Inc.
2. Day, D.F., *Spoilage in the Sugar Industry*. The Lactic Acid Bacteria in Health and Disease. Vol. 1. 1992: Springer US.
3. *Water Activity (aw) in Foods*. Inspection Technical Guides 2015; Available from: <http://www.fda.gov/ICECI/Inspections/InspectionGuides/InspectionTechnicalGuides/ucm072916.htm>.
4. Childs, S. and B. Chabot. *Measuring and Adjusting Invert Sugar in Maple Sugar*. 2007 [cited 2015; water activity]. Available from: <http://maple.dnr.cornell.edu/pubs/confections/Confection%20Notebook%20section2.pdf>.
5. Willems, K.A., et al., *Microbiological observations during storage of thick juice on a pilot and industrial scale*. CITS proceedings: Madrid 2003; 22nd general assembly. 2003: Berlin Bartens.
6. Honig, P., *Principles of Sugar Technology*. Vol. 3. 1963, Amsterdam, London, New York: Elsevier Publishing Company.
7. Sargent, D., S. Briggs, and S. Spencer, *Thick juice degradation during storage*. Zuckerindustrie (Germany). Aug 1997. v. 122(8) p. 615-62.
8. Hatch, R., et al., *Propylene glycolate used as a top coat seal to protect thick juice from degradation in ellipsoidal storage tanks*. Zuckerindustrie, 2013. **138**: p. 215-218.

## BAGACILLO IN RAW SUGAR

*Cy Gaudet and Franz Ehrenhauser*

### INTRODUCTION

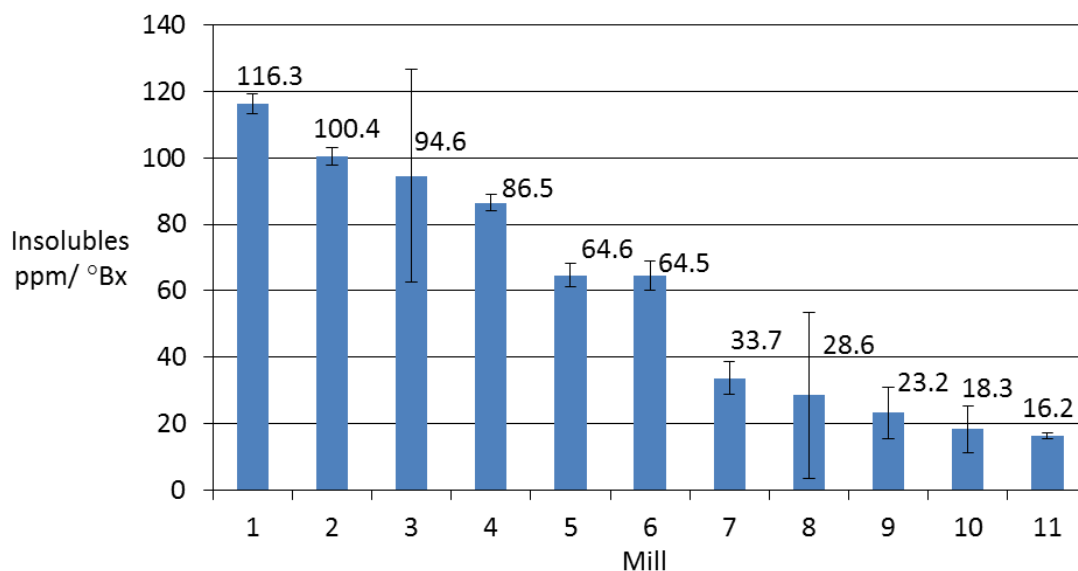
In the raw sugar factory insoluble material is inadvertently separated along with the raw sugar in the centrifugals. The insoluble particles can be fine bagasse fibers, metallic particles, or scale. This insoluble material consists of carryover from the clarification process, extraneous matter from evaporators, vacuum pans, piping, and holding tanks in the process. The insoluble matter can plug the screens in the centrifugals and reduce sugar filterability.

### METHOD

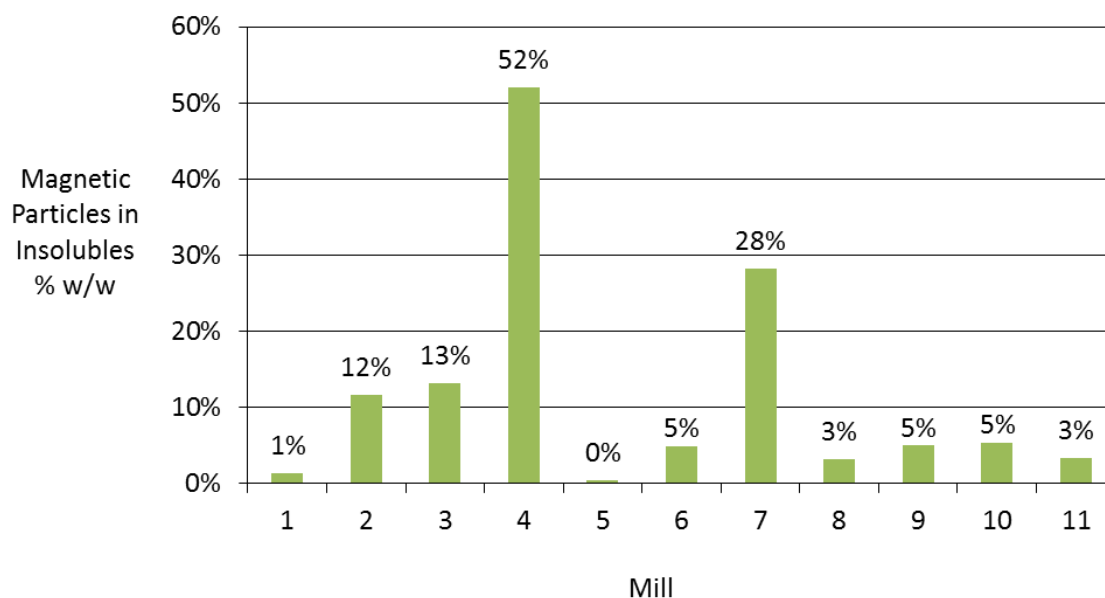
It was of interest in this study to determine the typical insoluble matter concentration in raw sugar. Samples were taken of A-sugar streams and raw sugar from warehouses of different sugar mills and analyzed for insoluble content. Each sugar sample was dissolved and filtered through screens to filter the insoluble matter. The insoluble matter and screens were rinsed to remove any residual soluble matter. The screens were then dried in a vacuum oven. The samples were dried until the weight was constant over a 24 hour period. After the drying process was completed, the difference between the final weight of the screen and insoluble matter and the initial weight of the screen, divided by the weight of the initial sugar sample, resulted in the insoluble matter concentration on a brix basis. This established a baseline for insoluble matter concentrations in raw sugar.

### RESULTS

Samples of A-sugar were taken at each sugar mill from A-centrifugals and conveyors. Taking the samples in these locations eliminated possible changes in insoluble concentrations that may occur during warehouse slinging, handling, and airborne contamination as compared to warehouse raw sugar samples. These samples were obtained within a one week period to minimize any influence of variations in cane quality and weather. Each sample was mixed prior to analysis, ensuring that any clumps were broken, before subsampling. Three replicates of 500 grams of sugar were analyzed from each sample. Figure 1 shows the average insoluble matter concentration of each subsample, using a 300-mesh [53- $\mu$ m aperture opening] stainless steel screen. The insoluble content of the samples ranged between 116.3 ppm/ °Bx to 16.2 ppm/ °Bx with an average of 58.8 ppm/ °Bx of all A-sugar samples analyzed. The average standard deviation of each sample was 8.52 ppm/ °Bx. There is high variability of the individual mill 3 and 8 subsamples in Figure 1 standard deviation of 32.1 ppm/ °Bx and 25.0 ppm/ °Bx, respectively.



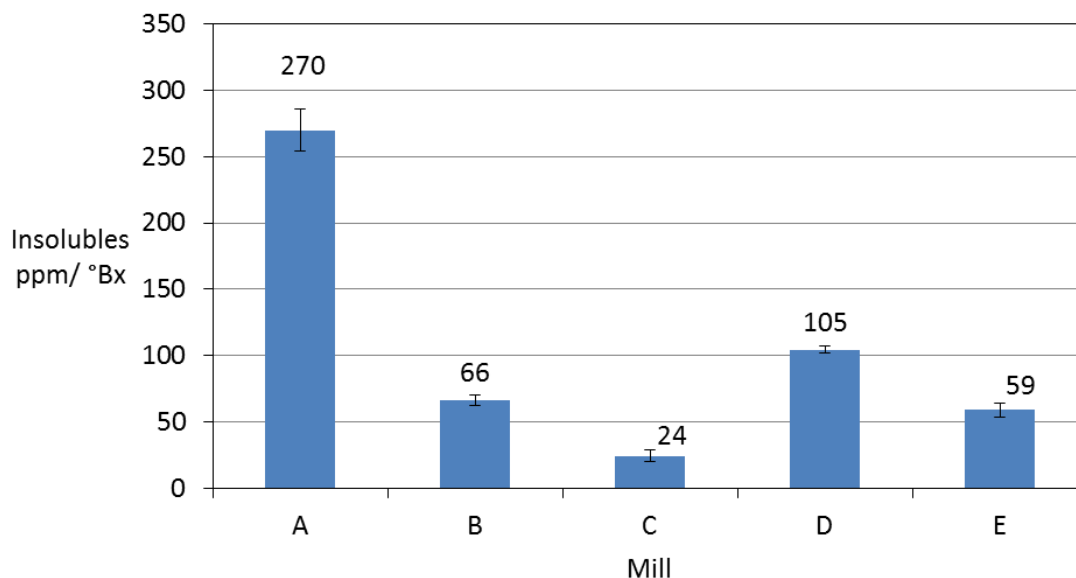
**Figure 1.** Insoluble matter concentration of A-sugar on a subsample average per mill basis using a 300 mesh [53- $\mu$ m aperture opening] stainless steel screen.



**Figure 2.** Percent of magnetic particles in insoluble samples on a mass basis.

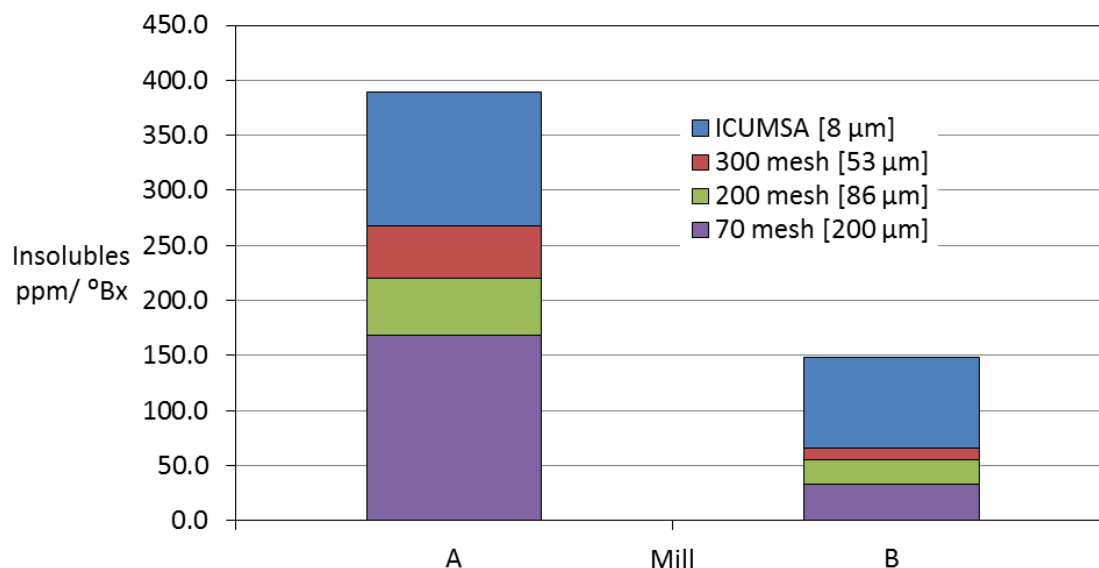
After the initial sample drying and weighing, a magnet was used to remove any magnetic metallic particles. The remaining material was re-weighed and the mass of magnetic particles removed was determined. Figure 2 depicts the subsample average % mass removed by the magnet. The magnetic particles on average accounted for 12% of the weight of insolubles. This demonstrates that insoluble matter is not purely fiber, and a significant amount of insoluble material can be of metallic origin.

During the off-season of 2015, raw sugar samples were taken from the warehouses of five mills. Each sample was taken from two locations in the warehouse and samples from both sampling locations were combined and well mixed. The samples were filtered using a 300-mesh [53- $\mu$ m aperture opening] stainless steel filter. Three subsamples were filtered from each warehouse sample. The averages of the three subsamples for each mill sample are reported in Figure 3. These samples ranged from 270 ppm/ °Bx to 24 ppm/ °Bx, with an average of 105 ppm/ °Bx. The standard deviation of each sample ranged from 15.8 ppm/ °Bx to 2.4 ppm/ °Bx, with an average standard deviation of each measured sample of 6.4 ppm/ °Bx.



**Figure 3.** The insoluble concentration of warehouse raw sugar samples for five mills using a 300-mesh [53- $\mu$ m aperture opening] stainless steel filter.

The warehouse samples of mills A and B were filtered through various screen sizes to determine the size of the insoluble material, and the removal effectiveness of different screen sizes. The stainless steel screens used included 300 mesh [53- $\mu$ m aperture opening], 200 mesh [86- $\mu$ m aperture opening], and 70 mesh [200- $\mu$ m aperture opening], as well as an 8- $\mu$ m membrane as described by ICUMSA Method GS 2/3/9-19 to determine the total insoluble material. The results are shown in Figure 4. Comparing to the ICUMSA 8- $\mu$ m membrane, the 300-mesh screen removed 69% and 45% of the insoluble matter at mills A and B respectively. The 200-mesh screen removed 57% and 37% of the insoluble material at mills A and B, and the 70-mesh screen removed 43% and 22% of the insoluble material at mills A and B.



**Figure 4.** The insoluble concentration of warehouse raw sugar samples of two mills using different size screens.

## CONCLUSION

It was found that the average A-sugar insoluble content was 58.8 ppm/ °Bx. Magnetic particles averaged 12% of the total insoluble matter weight. One sample had magnetic particles contributing to 52% of the total insoluble content weight, proving that metallic particles can contribute significantly to insoluble content. Raw sugar samples taken from the warehouse averaged 105 ppm/ °Bx. Results were also obtained to investigate the efficacy of using different screen sizes to remove insoluble content. Though technologies such as vibrating screens, self-cleaning filters, and syrup clarification can be used to reduce insoluble content, the current data suggest that it is possible for mills to reduce insoluble content to below 20 ppm/ °Bx by taking precautions using proper mill design and operating procedure. Further studies will have to be undertaken to determine what the significant sources of bagacillo are; whether preparation index, flash tank temperature, clarifier design and operation, contamination due to piping and vessels, or airborne contamination in open top holding tanks and vacuum breakers in the pans are relevant.

## ACKNOWLEDGEMENTS

The authors would like to thank the sugar mill personnel for discussions outlining a concern about their processes. We would like thank the various sugar mill personnel for their assistance in sample collection. The authors would also like to thank the American Sugarcane League for providing funding that makes these research projects possible.

## SUGAR CANE STARCH REFERENCE MATERIALS

*Franz Ehrenhauser, Cy Gaudet, Jonathan Bonin*

Sugar cane starch is a natural impurity of the sugar cane plant and particularly concentrated in the growing portions (top and leaves). During a warm season such as the 2015 grinding season, where cane without ripener continued growing into 2016, starch is omnipresent. Starch is an issue as it hampers the performance of the refinery and raw sugar factory. In the refinery filterability and viscosity are the main impact of starch; whereas in the raw sugar factory the impact on performance is mainly due to the increased viscosity. Cold-liming and/or amylase application are typically employed to reduce the starch concentration.

Starch itself is an inhomogeneous material consisting of granules made of amylose and amylopectin, usually associated with other colloidal material such as silica. Currently, sugar processing laboratories and researchers utilize substitute materials such as potato and corn starch for sugar cane starch determination and to evaluate mitigation methods such as amylases. This crude simulation of real sugar cane starch is always a point of discussion and results need to be validated against factory and refinery streams to confirm the laboratory results. In order to facilitate the research progress on starch, Audubon Sugar Institute has undertaken a research project funded by the American Sugar Cane League aiming at the production of more realistic reference materials, i.e. pure sugar cane starch and particulate matter from clarified juice.

### Clarified Juice Particulate Matter

Clarified juice from all Louisiana factories was obtained between October 22<sup>nd</sup> and November 4<sup>th</sup> and frozen prior to use. 5 gal of each juice were screened with a 150-micron mesh and microfiltered through a Graver 0.1- $\mu$ m titania-stainless-steel composite membrane to a retentate volume of approximately two liters. The average observed fluxes (monitored for nine mills) were  $18 \pm 2$  L/hr/m<sup>2</sup>/bar, on average 55% of the clean water value (1% of the virgin clean water flux). The obtained permeate was perfectly clear and always exhibited a turbidity below 1 NTU. The retentate was further diluted with deionized water and microfiltered, until its refractometric dry substance did not change much after dilution and was below 2 °Bx. The retentate was spray dried and the resulting powder further dried in a vacuum oven at 50 °C.

Table 1 shows the yield of the obtained particulate matter. The yield varied between 1.63 % to 6.29 % (based on juice) particulate matter (PM). The collected particulates account for 1.17 % to 5.46 % of the refractometric dry substance. The samples were packaged in 2 g aliquots for further characterization and research.

### Sugar Cane Starch

Crusher juice was obtained from the Cora-Texas sugar factory on October 6<sup>th</sup> 2015. The juice was stabilized with biocide and copper sulfate to minimize starch degradation. 15 kg of the juice were screened through a 150-micron mesh to remove bagacillo, micro-filtered (0.1 micron Graver membrane) to obtain a concentrated retentate (3.9 kg). The retentate's pH was adjusted with 10 mL concentrated hydrochloric acid to a pH of 2. The turbid juice was mixed 1:1 with

ethanol and the precipitate centrifuged at 7000 rpm for 20 min yielding a crude precipitate. This crude precipitate was extracted with chloroform, until the chloroform extract had taken on only a minor coloration. The extracted precipitate (approx. 250 g wet) was split in 2-g aliquots and extracted with approx. 7.5 g chloroform/toluene (75:25) mixture and 15 mL water in 50-mL centrifuge tubes. The chloroform/toluene mixture was adjusted so that the solid precipitate disk was sitting at the top of the conical bottom of the tube. Upon centrifugation (7 min 2700 rpm) a white layer separated on top of the solids and was scraped off, yielding crude starch mixture (approx. 100 g) The crude starch mixture is then again subjected to a chloroform/toluene extraction to yield white sugar cane starch granules. Complete characterization is currently still ongoing and will be reported separately.

**Table 1.** Particulate matter yields from clarified juice

Mill	% PM on Juice	% PM on Bx	PM collected [g]
1	0.32	2.65	30.0
2	0.46	3.63	48.9
3	0.51	3.89	45.0
4	0.46	3.17	42.8
5	0.63	4.67	62.5
6	0.29	1.71	31.2
7	0.40	2.86	41.5
8	0.38	2.78	41.0
9	0.30	2.64	30.3
10	0.42	5.46	12.6
11	0.16	1.17	24.6
AVERAGE	0.39±0.13	3.15±1.23	

#### **Acknowledgements:**

The authors would like to thank all Louisiana sugar mills and their personnel for their support, in particular Timmy Charlet from Cora-Texas for the help in obtaining the crusher juice, as well as the funding received from the American Sugar Cane League.



## Deashing of Sugar Streams

Tip P. Luangrujiwong, Peter Gaston, and Franz S. Ehrenhauser

### INTRODUCTION

Sweet sorghum, energy cane, and molasses are attractive biomass feedstocks because of the low cost and ability to produce high-value fermentable sugars. However, high level of impurities in these sugar sources limits their use in bioenergy and industrial applications. Contaminants, such as inorganic ions (ash), deactivate the catalyst in bioforming processes [1], inhibit the growth of yeast in fermentation process [2], and complicate product separation. To increase the value of sweet sorghum, energy cane, and molasses, it is important to remove ash from these sugar sources. De-ashing processes can also be used to remove impurities from sugar cane syrup to produce high quality syrups and increase sugar recovery in the sugar industry.

Sweet sorghum syrup, one of the most impure syrups, was selected to test with a series of deashing processes at Audubon Sugar Institute. Sweet sorghum syrup comprises sucrose, glucose, fructose, water, suspended solids, silica, and inorganic ions (i.e. potassium, calcium, chloride, and aconitic acid). In this work, the three steps used for deashing the sweet sorghum syrup are microfiltration, electrodialysis, and ion-exchange. Microfiltration is used to remove suspended solids and silica, electrodialysis is used to reduce ash content, and ion exchange is used to remove residual ash to meet the required levels. The goal of this work is to achieve a level of turbidity below 1 NTU, 0.05 % ash, and 65  $\mu\text{S}/\text{cm}$  in sweet sorghum syrup, which are the established levels for high value feedstock such as refined liquid corn syrups [3].

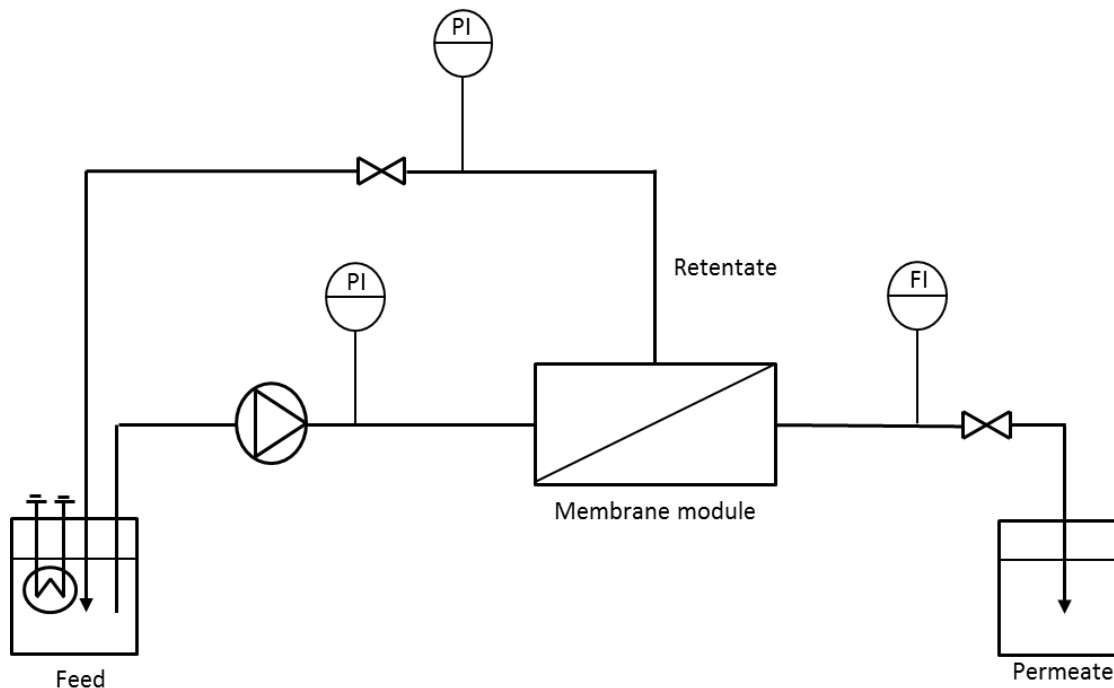
### MICROFILTRATION

Microfiltration was used to remove suspended solids and silica from sweet sorghum syrup. The turbidity of filtered syrup (permeate) should be noticeably lower than the syrup prior to the filtration.

#### Experiments:

The microfiltration unit was configured as shown in Figure 1. A Scepter<sup>®</sup> TiO<sub>2</sub>/stainless-steel composite 3/8" ID tubular crossflow microfiltration membrane was purchased from Graver Technologies (Delaware, USA). The membrane pore size was 0.1 micron, and membrane effective area was 0.11 m<sup>2</sup>. Feed solution was filled in a 5 gallon feed tank, and the temperature was controlled at 60 °C using the electrical heating coil. The feed was pumped through the membrane module at 5 GPM and 90 psig. Retentate was recirculated through the feed tank. The feed pressure was controlled by the valve on the retentate side.

The sweet sorghum syrup was divided and diluted to three different concentrations (40, 50, and 60 °Bx). Weight and sugar concentration of feed, permeate, and retentate were measured. Dry substance in syrup was measured in °Bx unit using refractometer. Permeate flow rate and pressure at feed and permeate were periodically measured and recorded during the filtration. When the permeate flow rate was lower than 100 mL/min (the minimum detection limit for the digital flow meter), it was manually measured using a volumetric cylinder and stopwatch. Permeate flux and permeability were calculated. Turbidity of the final permeate was measured and compared to the initial feed.



**Figure 1.** Schematics of crossflow microfiltration unit

### Results and discussion:

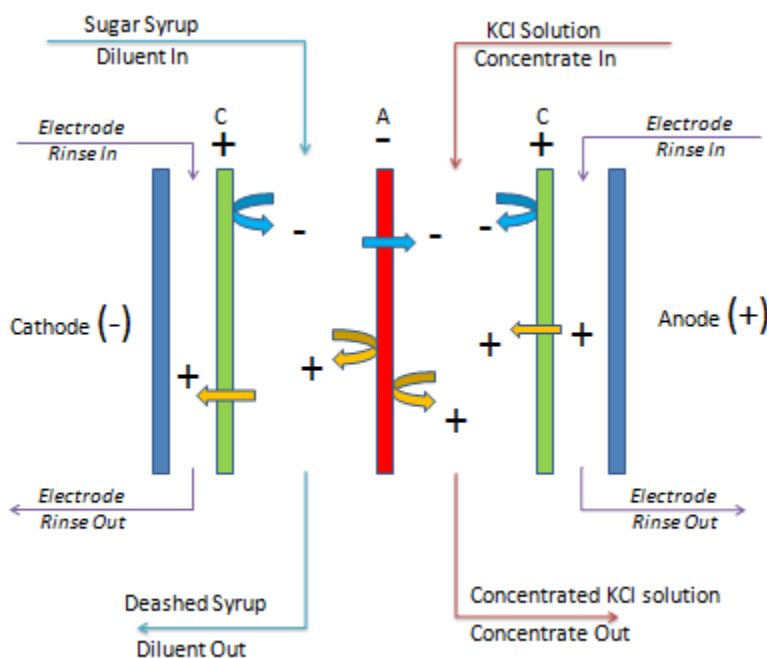
The initial permeate flux of sweet sorghum syrup at 40, 50, and 60 °Bx was 25.06, 18.57, and 4.00 L/h·m<sup>2</sup>, respectively. Permeate flux of sweet sorghum syrup was significantly lower than water flux (155.45 L/h·m<sup>2</sup>). However, it was comparable to the permeate flux of a clarified cane juice (15 °Bx) through 100 micron spiral membrane at 40-60 L/h·m<sup>2</sup> [4] and through 10 micron non-coated stainless steel membrane at 170 L/h·m<sup>2</sup> [5]. The permeate flux of the sweet sorghum syrup decreased with time due to the accumulation of particles that form a layer on the microfiltration membrane surface. The layer increases a resistance to permeation. When the convection of particles toward the layer by permeate flow is balanced by diffusion and inertial lift of particles away from the layer, the permeate flux reaches steady state.

At 60 °Bx, permeate flux dropped nearly immediately and stayed constant at 4.0 L/h·m<sup>2</sup>. The permeate flux at 60 °Bx was one order of magnitude smaller than the fluxes at 40 and 50 °Bx. This indicated that the membrane was fouled very quickly at high concentration. This could be due to the high viscosity of 60 °Bx solution (at 40 °C, viscosity of sucrose solution at 40, 50, and 60 °Bx is 3.3, 7.0, and 20.9 cP, respectively [6]) resulting in a thicker boundary layer and lower mass transfer rate. According to these results, it is recommended to use feed solution for microfiltration tests at 50 °Bx or less.

The final permeate was a clear brown solution. The average turbidity of feed and permeate was 294 and 8.76 NTU, respectively. The turbidity was significantly decreased after the filtration.

## ELECTRODIALYSIS

Electrodialysis was used to reduce dissolved ash in the microfiltered sweet sorghum syrup. As shown in Figure 2, an electrodialysis cell contains a positive electrode (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, cations (+) move to the negative electrode and 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 solution is a high conductivity solution which provides for high efficient current transfer and minimizes undesired electrode reactions. For the practical application sugar syrup would be pumped through “dilute in” into the diluent chamber of the electrodialysis cell, and ions move from the syrup through membranes to the concentrate chamber by the applied electric field. As a result, the deashed syrup is recovered from the diluent chamber and the dissolved ash is recovered from the concentrate chamber.



**Figure 2.** Schematics of electrodialysis cell

### Experiments:

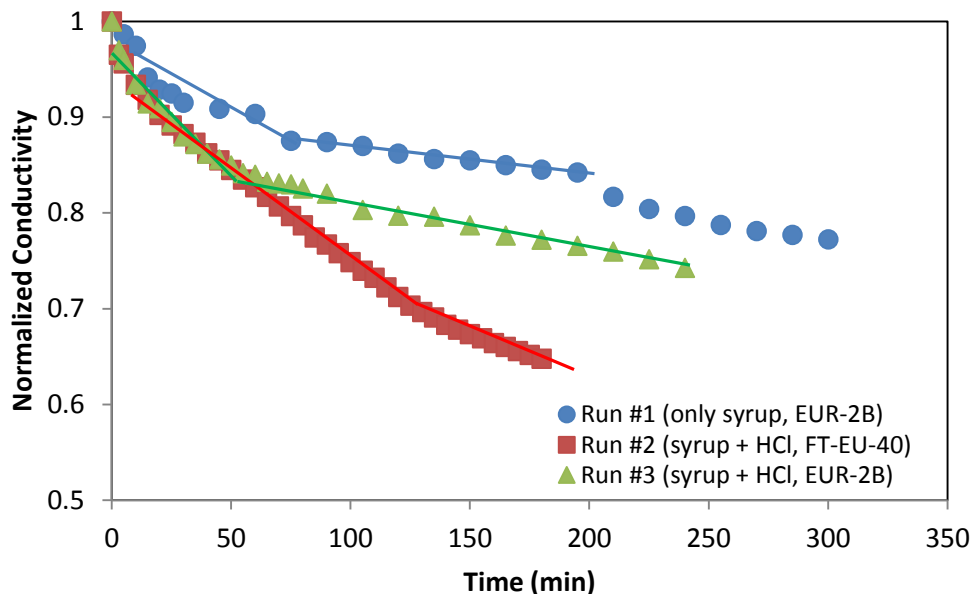
Two electrodialysis cells, a lab-scale (36 cm<sup>2</sup> membrane area per cell) and a pilot scale (0.2 m<sup>2</sup> membrane area per cell), were used. A lab-scale electrodialysis cell FT-EDR-40 was purchased from Fumatech GmbH (Bietigheim, Germany). The three-chamber cell consists of two titanium-iridium plasma coated stainless steel electrodes, PP endplates and 22 PVC spacers providing a housing for up to ten membrane pairs. Electric current was supplied by an Extech DCP60 600 W, 120 V adjustable DC power supply. The ion exchange membrane pairs (anion/cation) AMX/CMX were purchased from Ameridia (New Jersey, USA). Flows to the

individual chambers were provided by peristaltic pumps (Cole-Parmer MODEL 77250-62 with L/S 35 silicone tubing). 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 Pt100 controlled hot plate. All experiments were carried out at 1 A of constant current. A pilot-scale electrodialysis cell EUR-2B-10 was purchased from Ameridia. The setup of the pilot-scale cell was similar to the lab-scale cell. All solutions (the concentrate, diluent, and electrode rinse) were operated at 0.8 GPM, and the temperature of the solutions was controlled by using electrical heat coils. The experiments on the pilot-scale cell were carried out at 40 V of constant voltage. Potassium chloride (99%, ACS grade) and potassium sulfate (99%, ACS grade) were purchased from Alfa Aesar and used without further purification.

The sweet sorghum syrup from the microfiltration was combined and mixed well prior to electrodialysis. This mixed syrup (30 kg, 47.25 °Bx) was used as diluent solution, deionized water was used as concentrate solution, and 0.2 M K<sub>2</sub>SO<sub>4</sub> was used as electrode rinse solution. All experiments were conducted in batch mode where the solutions were recycled through their reservoirs. Weight, RDS, conductivity, and pH of the diluent, concentrate, and electrode rinse solutions were measured and recorded before and after each experiment. Conductivity, pH, voltage, and current were recorded throughout the experiment. Conductivity and pH were monitored with an Oakton PC700 pH/conductivity meter in the respective reservoirs.

### **Results and discussion:**

The plots of normalized conductivity of the diluent with time are displayed in Figure 3. The results show that the normalized conductivity decreases with time. The reduction in conductivity indicates the ash removal of the diluent. The first experiment of electrodialysis (Run #1, blue-circle plot) was performed using the pilot-scale EUR-2B cell. The reduction of the normalized conductivity decreased dramatically after 75 minute run time, which can be seen from the change of the slopes. It was suspected that the membrane was fouled due to the precipitation of calcium aconitate on the membrane surface, resulting in the decrease of ash removal. This could be explained that at the beginning when the concentration of potassium was high, aconitic acid was likely in potassium aconitate form which dissolves readily in aqueous solution [7]. During electrodialysis, potassium ions were preferentially removed from the diluent and the relative calcium concentration in the diluent increased, leading to calcium aconitate precipitation. Calcium aconitate precipitates at a pH above 4.83 [8]. To prevent the precipitation of calcium aconitate on the membrane surface, the pH of the sweet sorghum syrup was adjusted to below 4.83 using hydrochloric acid. The acidic syrup was tested with the lab-scale FT-EDR-40 cell as Run #2, and the normalized conductivity is shown as the red-square plot in Figure 3. Red line (Run #2) was drawn to compare its slope with the blue line (Run #1). The rate of ash removal from Run #2 was significant higher than Run #1. This indicated that adjusting pH improved the ash removal and implied that the membrane fouling due to calcium aconitate precipitation was resolved. It should be noted here that changing pH will cause sugar inversion. In Run #3, the diluent from Run #2 was mixed with the rest of syrup from Run #1, adjusted to pH 4.75, and then tested with the pilot-scale EUR-2B cell. The normalized conductivity from Run #3 is presented as green-triangle plot in Figure 3.



**Figure 3.** Plots of normalized conductivity from all electro dialysis experiments vs. time

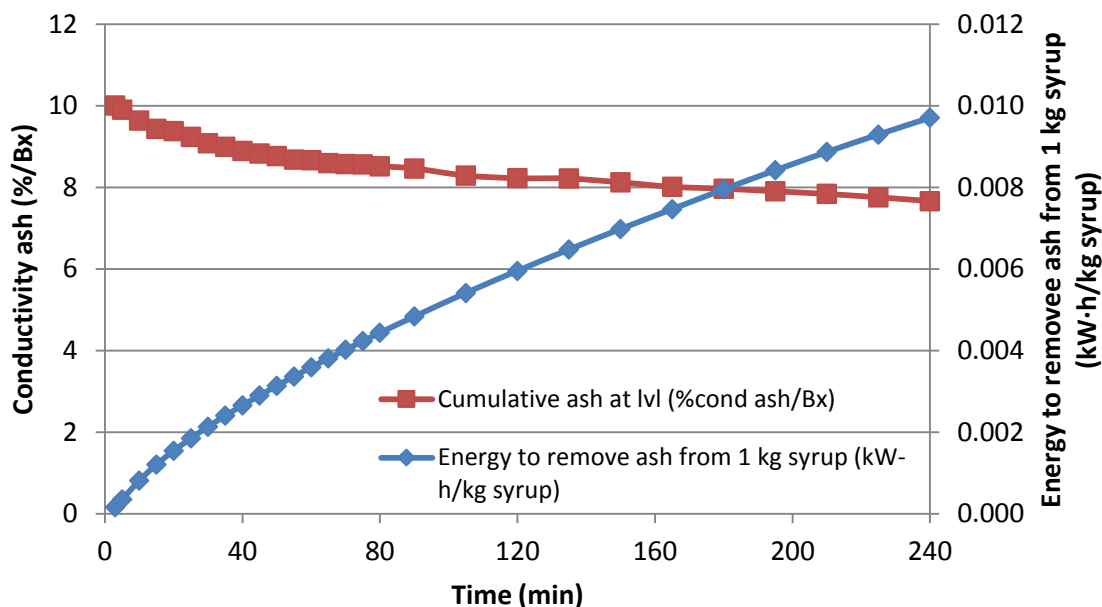
**Table 1.** The summarized results of electro dialysis experiments for deashing sweet sorghum syrup.

	Run #1 (syrup, EUR-2B)		Run #2 (added HCl, FT-40)		Run #3 (added HCl, EUR-2B)	
	Initial	Final	Initial	Final	Initial	Final
Syrup weight (kg)	30.032		3.671		27.253	
RDS (°Bx)	47.25		46.86		46.36	
pH	6.15		4.4		4.75	
Conductivity (mS/cm)	12.64	9.32	12.06	7.81	11.23	8.54
Conductivity ash* (%/°Bx)	15.22	10.84	10.84	7.02	10.32	7.67
Ash removed** (%)	26.03		35.24		25.73	
Conductivity ash removed (%/°Bx)	49.61					

\* based on the initial ICUMSA conductivity ash assuming total ash is 100% KCl [9]

\*\* based on conductivity decrease

The results from the electro dialysis experiments for deashing sweet sorghum syrup are summarized as shown in Table 1. The overall ash removed from the sweet sorghum syrup by electro dialysis was 49.61 %/Bx. From Run #3, the conductivity ash and energy used to remove 1 kg syrup were calculated and plotted with time as shown in Figure 4. An advantage of electro dialysis is its volume flexibility. We can select to conduct it at a level of ash removal that is economically feasible.



**Figure 4.** Plots of conductivity ash and energy used to remove 1 kg syrup vs. time using data from electrodialysis Run #3

## ION EXCHANGE

The conductivity of sweet sorghum syrup after electrodialysis was 8.54 mS/cm (at 46.36 °Bx) and 7.67 % ash. The conductivity after ion exchange was targeted at a level below 65  $\mu$ S/cm (at 30 °Bx) and 0.05 % ash. Residual cations and anions were removed from the syrup using a cation exchange and anion exchange system.

### Experiments:

Two ion-exchange columns were setup in parallel. A cation-exchange column was packed with a strong acid cation exchange resin Dowex™ Monosphere™ 88; an anion-exchange column was packed with a weak base anion exchange resin Dowex™ Monosphere™ 66. Both resins were purchased from Dow Chemical (USA). The ion exchange columns must be homogeneously packed to obtain the maximum efficiency. Each run was ended when the resin was fully exhausted (breakthrough). Deionized water was used to rinse syrup from the ion exchange system and collected as dilute sweet water. The resin in the column was regenerated after exhausted using hydrochloric acid solution for cation regeneration and sodium hydroxide solution for anion regeneration. Deionized water was used to rinse the columns until there was no residual acid or base in the column. All solutions were degassed prior to applying to the columns to eliminate bubbles entering the column that could cause irregularity in the packed bed.

The sweet sorghum syrup from electrodialysis (26.88 kg total) was run through the 2-pass ion exchange system in batches at 60 °C. The second pass was necessary to meet a lower level of conductivity. Conductivity, weight, RDS, and pH of the syrup were measured and recorded.

## Results and discussion:

As shown in Table 2, conductivity of sweet sorghum after the first pass through the ion exchange system reduced from 8.54 mS/cm to 0.18 mS/cm. After the second pass, the conductivity was achieved at a level of 7.9  $\mu$ S/cm (at 31 °Bx), which was significantly less than the target value of 65  $\mu$ S/cm (at 30 °Bx).

**Table 2.** Summarized results of sweet sorghum through the 2-pass ion exchange system

	Weight (kg)	RDS (°Bx)	Conductivity (mS/cm)	pH
Syrup from electrodialysis	26.45	46.92	8.54	4.91
After 1 <sup>st</sup> -pass ion exchange	29.19	31.82	0.178	3.77
After 2 <sup>nd</sup> -pass ion exchange	29.27	31.12	0.0079	4.74

## SUMMARY AND CONCLUSIONS

We successfully deashed sweet sorghum syrup and produced a pure syrup that exceeds the criteria for high value feedstock. Our de-ashing processes removed ash from 12.6 mS/cm to  $7.9 \times 10^{-3}$  mS/cm (at 31 °Bx), which is equivalent to a 99.94 % ash removal. The final sweet sorghum syrup was 0.3-0.7 NTU and 7.9  $\mu$ S/cm (at 31 °Bx), whereas the goal of this work was aimed at a level of turbidity below 1 NTU, 0.05 % ash, and 65  $\mu$ S/cm in sweet sorghum syrup.

## ACKNOWLEDGEMENTS

This project was supported by Agriculture and Food Research Initiative Competitive Grant no. 2011-69005-30515 from the USDA National Institute of Food and Agriculture. The authors would like to thank Chardcie Verret and Stephanie Linares (Audubon Sugar Institute) for their help with the analytical assessment.

## REFERENCES

1. Hydrolysate Conditioning in the Catalysis of Lignocellulosic Sugars Strategy. (2013, January 1). Retrieved from [http://www.nabcprojects.org/pdfs/hydrolysate\\_conditioning\\_cls\\_010113.pdf](http://www.nabcprojects.org/pdfs/hydrolysate_conditioning_cls_010113.pdf).
2. Park, C. B. and Lee, S. B. (1999). Inhibitory Effect of Mineral Ion Accumulation on High Density Growth of the Hyperthermosphilic Archaeon *Sulfolobus solfataricus*, J. Biosci. Bioeng, 87(3), 315-319.
3. Clearsweet® 99% Refined Liquid Dextrose Corn Syrup. (2012, August 14). Retrieved from <https://www.cargillfoods.com/wcm/groups/public/@cseg/@food/@all/documents/document/na3014963.pdf>.
4. Saska, M., McArdle, J., and Eringis, A. (1999). Filtration of clarified cane juice using spiral polymeric membrane configuration. Proc. Int. Soc. Sugar Cane Technol., XXIII Congress, India.

5. Steindl, R.J. and Doyle, C.D. (1999). Applications and benefits of membrane filtration for the Australian sugar industry. *Proc. Aust. Soc. Sugar Cane Technol.*, 21, 406-411.
6. Swindells, J. F., et. al. (1958). Viscosities of Sucrose Solutions at Various Temperatures: Tables of Recalculated Values. 1-7.
7. Watts, H. (1879). *A Dictionary of Chemistry and the Allied Branches of Other Sciences*, Volume 1, 54.
8. Greenwald, I. (1938). The dissociation of some calcium salts. *J. Boil. Chem.*, 124, 437-452.
9. ICUMSA. (1994). The determination of conductivity ash in raw sugar, brown sugar, juice, syrup and molasses – Official. Method GS1/3/4/7/8-13, 1-2.