

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

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## **LARGE SCALE RIPENER EVALUATION**

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### **INTRODUCTION**

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

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

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

### **STUDY**

A large scale field experiment was treated with glyphosate at 0.187 lb/A (210 g ae/ha) and Palisade at 0.312 lb/A (350 g ai/ha) and compared to the untreated control and harvest at 28 and 56 days after treatment, respectively, in 2012 (Table 1). Treatments were applied aerially to second stubble HoCP 96-540. Ripener treatments were applied at 3 gallons of spray mixture per acre. At each location, ripener treatments were applied once, and are considered a single replicate. The three locations were Blackberry Farms, Vacherie, LA, Hebert Brothers Farm, Thibodaux, LA, and Ronald Hebert Farms, Jeanerette, LA. Palisade treatment was applied at approximately 56 days before harvest while glyphosate treatment was applied at approximately

28 days before harvest. Both glyphosate and Palisade treatments, as well as, the untreated control were harvested on the same day for a given location. Cane was harvested by combine and scale weights were obtained from the factories where the cane was processed. Core sample analyses for obtaining the yield of theoretical recoverable sugar per ton of cane (TRS) were obtained from both front and rear compartments of all trucks that were part of the experiment.

## **RESULTS**

Mean values for Blackberry Farms, Ronald Herbert Farms, and Hebert Brothers Farm are presented in Tables 2, 3, and 4, respectively. Data were analyzed as a randomized complete block experiment, with each location representing one replication.

Both glyphosate and Palisade increased sugar per acre (Table 5); however, the increase for glyphosate came from increasing TRS by 10.2% while not affecting tonnage, and the increase for Palisade came from a reduced increase in TRS (4.9%) and a large increase in cane tonnage. It is interesting to note that Palisade is not as effective as glyphosate in increasing TRS even given the extra two weeks from treatment to harvest. However, from this experiment, Palisade actually accounted for a significant increase in tons of cane per acre when compared to the untreated control.

Table 1. Large scale field experiment comparing efficacy of glyphosate and Palisade to untreated control at Blackberry Farms (Vacherie), Ronald Hebert Farms (Jeanerette) and Hebert Brothers Farm (Thibodaux).

<b>Farm</b>	<b>Treatment</b>	<b>App. Date</b>	<b>Harvest Date</b>	<b>Harvest Int.</b>
Blackberry	Glyphosate	9/11/2012	10/8/2012	27
Blackberry	Palisade	8/13/2012	10/8/2012	56
Blackberry	Control		10/8/2012	
Ronald Hebert	Glyphosate	9/13/2012	10/11/2012	28
Ronald Hebert	Palisade	8/11/2012	10/11/2012	61
Ronald Hebert	Control		10/11/2012	
Hebert Brothers	Glyphosate	9/14/2012	10/15/2012	31
Hebert Brothers	Palisade	8/17/2012	10/15/2012	59
Hebert Brothers	Control		10/15/2012	

Table 2. Results from Blackberry Farms at Vacherie.

<b>Treatment</b>	<b>Acres Harvested</b>	<b>Mean TRS (lbs)</b>	<b>Mean Tons/A (tons)</b>	<b>Sugar/A (lbs)</b>
Glyphosate	1.68	200	46.6	9311
Palisade	1.47	186	52.1	9703
Control	1.60	180	49.5	8934

Table 3. Results from Ronald Hebert Farms at Jeanerette.

<b>Treatment</b>	<b>Acres Harvested</b>	<b>Mean TRS (lbs)</b>	<b>Mean Tons/A (tons)</b>	<b>Sugar/A (lbs)</b>
Glyphosate	2.77	210	35.2	7404
Palisade	2.52	203	39.1	7922
Control	2.91	190	32.8	6229

Table 4. Results from Hebert Brothers Farm at Thibodaux.

<b>Treatment</b>	<b>Acres Harvested</b>	<b>Mean TRS (lbs)</b>	<b>Mean Tons/A (tons)</b>	<b>Sugar/A (lbs)</b>
Glyphosate	2.23	193	45.4	8777
Palisade	1.96	184	52.4	9628
Control	2.20	177	44.7	7917

Table 5. Combined analyses for three locations

<b>Treatment</b>	<b>TRS (lbs)</b>	<b>Increase (%)</b>	<b>Tons/A (tons)</b>	<b>Sugar/A (lbs)</b>
Glyphosate	201 a	10.4	42.4 b	8497 a
Palisade	190 b	4.4	47.9 a	9084 a
Control	182 c	--	42.4 b	7693 b
F-Value	0.0014		0.0251	0.0107

# REMOVAL OF SUSPENDED SOLIDS FROM FILTRATE

*S. Grimaldo and V. Kochergin*

## INTRODUCTION

In the sugar cane process the filtrate juice is continuously recirculated back to the clarification process. This stream accounts for almost 15-20% of the mixed juice. The recirculation of this stream brings several disadvantages to the process including recirculation of sugar as well as non-sugars, inversion of sucrose, microbial activity, and color generation. If this stream is treated independently through clarification and the quality of the juice is sufficient to be mixed with the clarified juice that is forwarded to the evaporators, the performance of the process can be improved by eliminating this recirculation and increasing the clarification capacity by almost 20% (Bento & Cuddihy, 2006; Prasad & Kafukp, 2005). However, this operation has been attempted several times with low success due to the high residence times of the clarifiers and complex processes. Therefore, the aim of this research is to overcome these limitations through the development of a simple clarification process that involves the design of a Very Short Residence Time Clarifier (VSRT) utilizing Turbulence Reduction Devices (TRD'S).

## PROJECT GOALS

- Design a scalable pilot plant for filtrate clarification using turbulence reduction devices.
- Construct and tune-up the filtrate clarification pilot plant in a sugar mill.
- Operate and test the filtrate clarification pilot plant during the grinding season.
- Evaluate the performance of the pilot plant using standard analytical procedures.

## MATERIALS AND METHODS

### Process Design

A filtrate clarification pilot plant was designed to handle a maximum flow rate of 100 gallons per minute. The designed process is shown in Figure 1 and was constructed at Alma Plantation Mill. The process consists of heating the filtrate in a heat exchanger (E-101) up to 104°C (220°F). After this operation, lime saccharate (V-101) is added to the juice (hot liming) in order to start the flocculation process. Then the filtrate juice, which is slightly above its boiling point, is degassed in a flash tank (FH-101) to remove all the air entrained in this stream in order to enhance the clarification operation. When all the air has been removed, a flocculant in a dose between 4 ppm- 6 ppm is added to the juice to form the flocs. The flocculant is stored in a 30 gallon vessel (V-102) and is dosed using a metering pump (P-102). Finally, the juice is directed to the VSRT (C-101) where two phases will be obtained: The clarified filtrate as the overflow and the mud phase as the underflow. Finally, the two streams, clarified filtrate and the muds, is mixed again and returned back to the process.

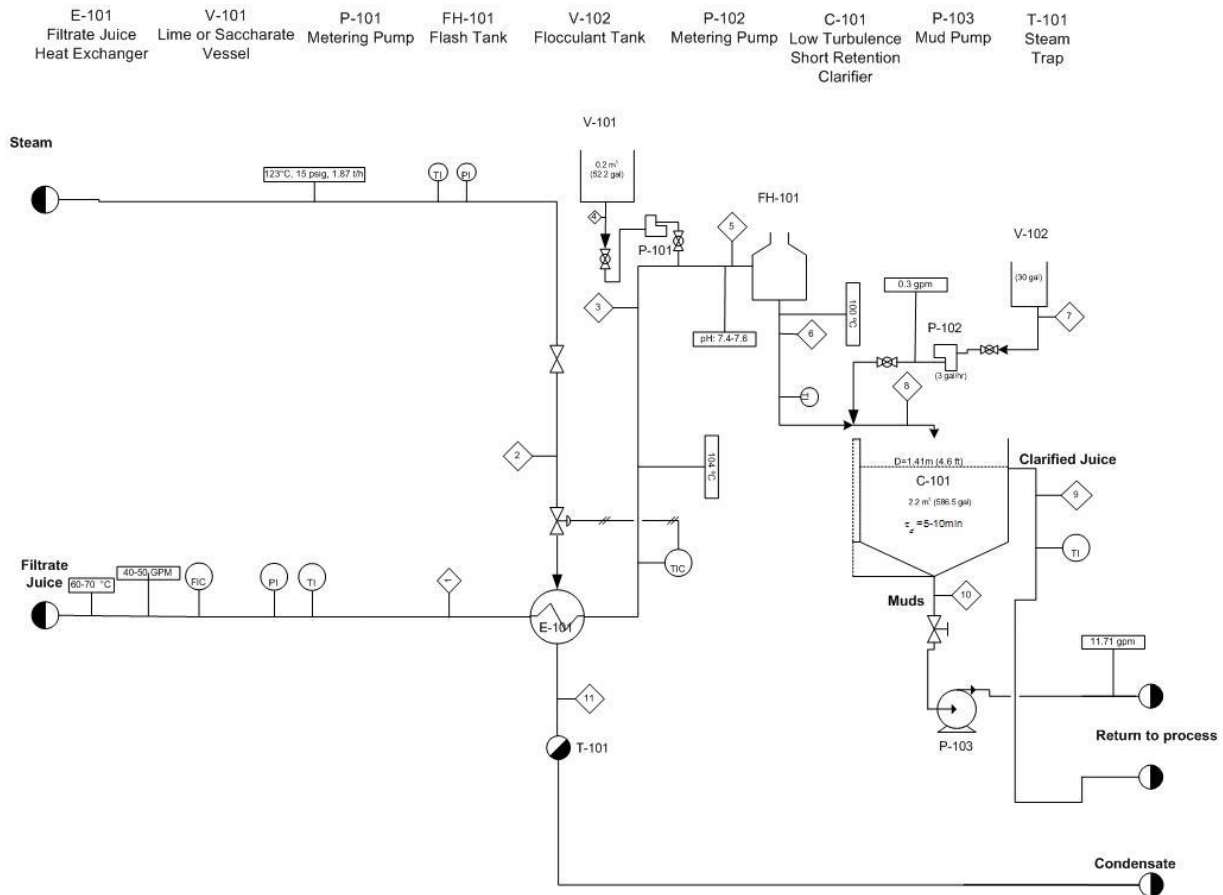


Figure 1. Filtrate Clarification Pilot Plant

### New Flash-Clarifier Design

Flashing is a very important step to achieve a good clarification. However, this operation is often overlooked and can bring consequences including re-entrainment of air and excessive presence of bagacillo in the clear juice not properly flashed. In order to minimize these effects a new invention has been devised that incorporates the flash and clarification operations into an integrated unit. The invention consists of a degassing trough around the clarifier and it will be positioned at the same level of a feed launder in the center of the clarifier. This condition will keep a level inside similar to a “Type B” flash tank (Rein, 2007). However, the main advantage of this degassing system is that the trough surrounding the clarifier will provide more area with less foot print guaranteeing a proper degasification of the liquid without re-entraining the air. The elimination of the incondensable gases will be done through vents surrounding the degassing trough. After the juice has been flashed it will be clarified using a Louisiana Low Turbulence Clarifier (LLT). A top and side view of the invention is shown in Figures 2 and 3. The whole unit has been filed for a Provisional Patent Application and the device was successfully tested at Andhra Sugars Ltd. in India.



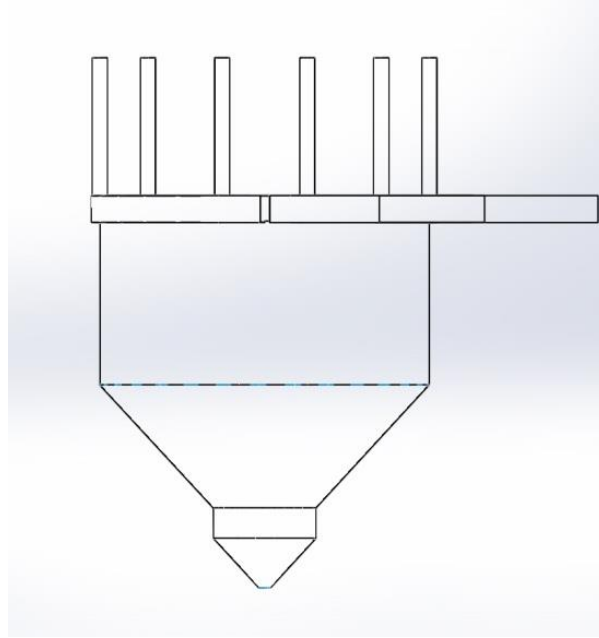


Figure 2. Side View of the Degassing-Clarification Station

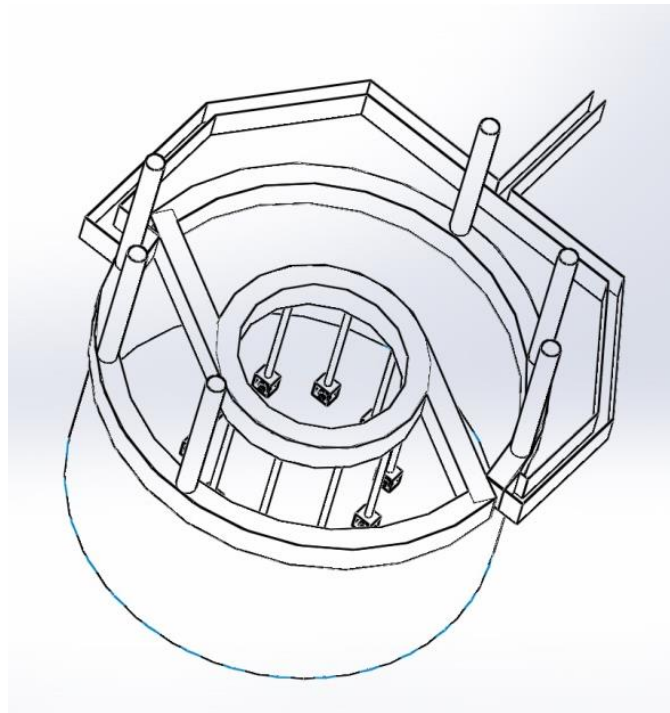


Figure 3. Top View of the Degassing-Clarification Station

### Sampling and Data Analysis

The filtrate clarification pilot plant was tested during the 2012 harvesting season at Alma Plantation mill. Even though the pilot's designed capacity was 100 gpm, flow supply restrictions limited the flow to 60 gpm; which is equivalent to 8 minutes residence time of clear juice. In order to assess the performance of the filtrate clarifier, three different conditions were tested:

- Filtrate flow of 60 gpm with a 6 ppm dose of flocculant
- Filtrate flow of 60 gpm with a 4 ppm dose of flocculant
- Filtrate flow of 40 gpm with a 4 ppm dose of flocculant

## RESULTS AND DISCUSSION

### Suspended Solids

The suspended solids were analyzed in Audubon Sugar Institute from samples of raw and clarified filtrate collected during the trials. In Figure 4 it can be observed that the concentration of suspended solids for the filtrate varied between 6349 to 8510 ppm and after clarification the suspended solids concentration decreased significantly to values that ranged from 469 to 1038 ppm. It is worth to mention that the concentration of suspended solids of the clarified filtrate still slightly high but within the range compared to values (133 ppm to 1064 ppm) of suspended solid in clear juice reported in the South African sugar industry (Jullienne, Matic, & Teokarovic, 1970). Finally, the percent removal of suspended solid is shown in Figure 5 where it ranged between 84 to 95%.

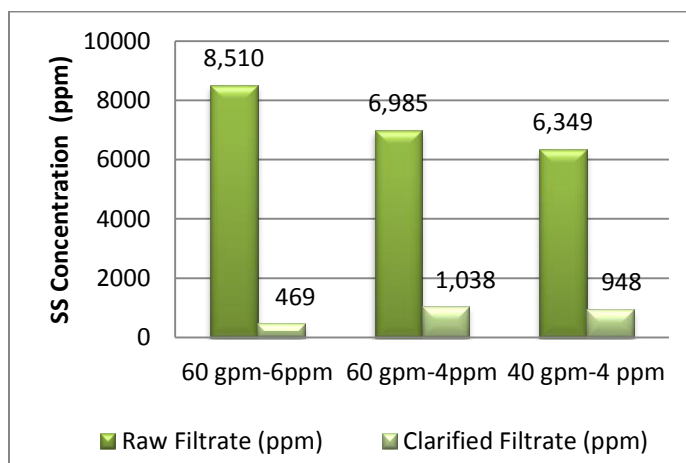


Figure 4. Concentration of Suspended Solids in Filtrate and Clarified Filtrate.

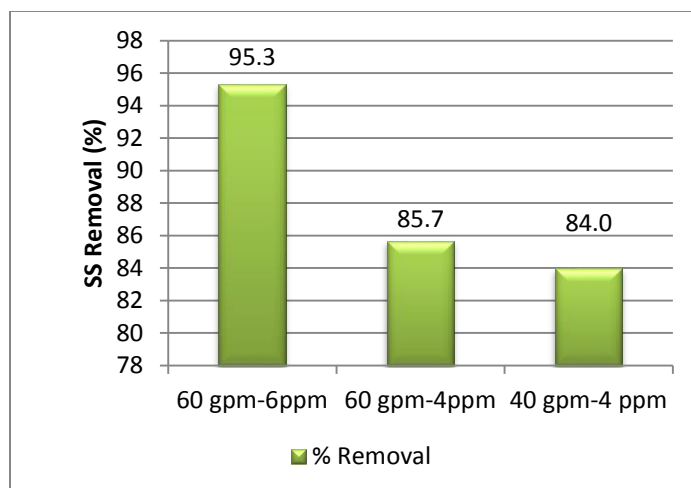


Figure 5. Percent Removal of Suspended Solid in the Clarifier.

### Turbidity

In order to assess the performance of the clarifier, turbidity was measured and recorded for different samples: clarified filtrate, the clear juice, a composite obtained from mixing the clear juice and the clarified filtrate in a proportion of 1:5 (Proportion of clarified filtrate to mixed juice) and the turbidity from jar tests that were done at the same time as the pilot plant was being operated. These turbidities are shown in Figure 6 for each experiment. It can be inferred from the figure and also from the application of statistical analysis that there are still significant differences (P-value <0.05) between the clear juice from the main clarifiers and the clarified filtrate. However, the differences between the turbidity of the mix and the clear juice are not significant (P-value >0.05) suggesting that these two stream can be mixed.

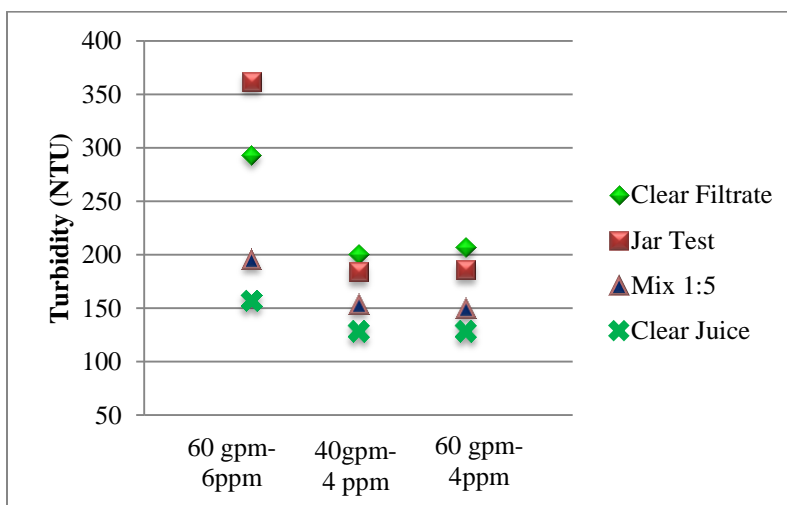


Figure 6. Turbidity Profile of Different Samples.

Finally, the turbidity means and 95% confidence intervals of the clear filtrate and the 1:5 composite are summarized in Table 1.

Table 1. Turbidity Confidence Intervals for Clarified Filtrate and 1:5 Mix.

Experiment	Sample	Mean (NTU)	95 % Confidence Intervals (NTU)	
<b>60 gpm-6 ppm</b>	Mix 1:5	196.0	107.1	284.9
	Clarified Filtrate	293.2	259.6	326.7
<b>60 gpm- 4 ppm</b>	Mix 1:5	152.8	126.9	178.7
	Clarified Filtrate	200.4	181.1	219.7
<b>40 gpm- 4 ppm</b>	Mix 1:5	149.5	101.7	197.3
	Clarified Filtrate	207.1	187.5	226.7

### Flash-Clarifier Design

The new flash-clarifier design was tested in the Andhra Sugars Corporation. In order to assess the performance of this new invention, two clarifiers were compared: the first one was an LLT and a common flash tank. The latter was leveled with the clarifier overflow. The second clarifier was the proposed design where a flash trough comes built-in the clarifier; therefore the flashing and clarification takes place in the same equipment. The results shown in Table 2 demonstrate that the new design is fully functional and its implementation provides additional degassing area with fewer footprints. Finally, the unit operating in India is shown in Figure 7.

Table 2. Comparison between an LLT with Flash and without Flash Trough

Sample	% Turbidity Reduction	
	Without Flash Trough	With Flash Trough
<b>1</b>	93.77	94.36
<b>2</b>	93.31	93.54
<b>3</b>	93.04	93.59
<b>4</b>	92.83	93.37
<b>5</b>	92.22	92.55
<b>6</b>	91.16	92.08
<b>7</b>	90.04	90.54
<b>AVERAGE</b>	<b>92.47</b>	<b>92.99</b>



Figure 7. Flash-Clarifier Design Operating in India.

## CONCLUSIONS

- A Very Short Residence Time Clarifier (8 min) has been designed and implemented for filtrate clarification.
- LLT Technology has proved to be robust for different clarification applications.
- The quality of the obtained clear filtrate was good. The SS removal was greater than 84%; also when mixed with clear juice in a 1:5 proportion it can be observed that the turbidity fits in the clarification target (~150 NTU).

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- David Stewart, Mario Acevedo, Josemanuel Henriquez, Nelson Dieguez, Alvaro Oquendo, Rogelio Piñeiro, Belisario Montes, Rosenelly Corrales, Rubén Vasquez the personnel from Alma Plantation Ltd for all their support.
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# PRELIMINARY INVESTIGATION OF FILTER STATION OPERATIONS

*H. Birkett and J. Stein*

## INTRODUCTION

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

## METHOD

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

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

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

## RESULTS AND DISCUSSION

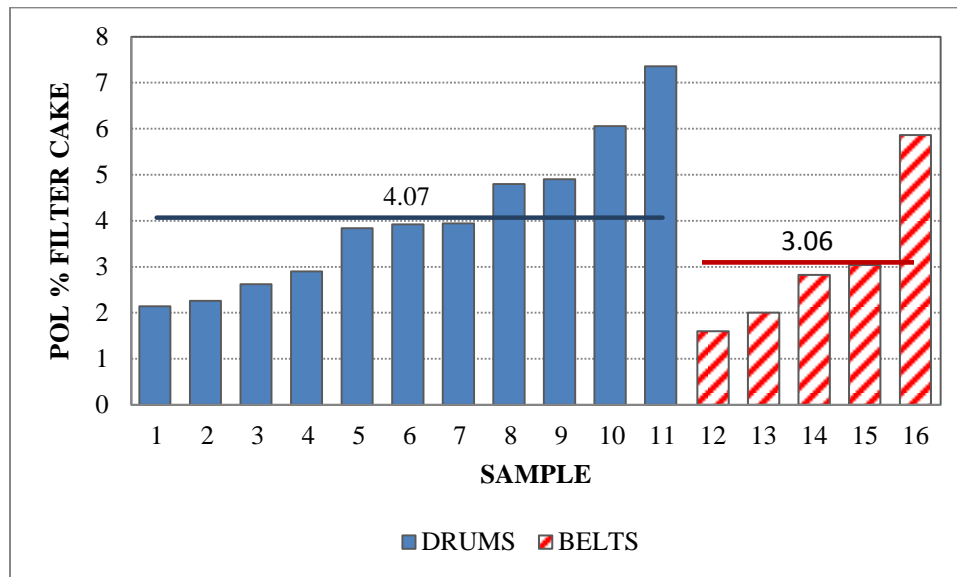


Figure 1. Pol % filter cake for drum and belt filters.

Figure 1 shows the pol % filter cake average of 3.75. Pol % filter cake from drum filters ranged from 2.14 to 7.36 and averaged 4.07% pol. Belt filters had an average pol % filter cake of 3.06 with a range of 1.60 to 5.86%.

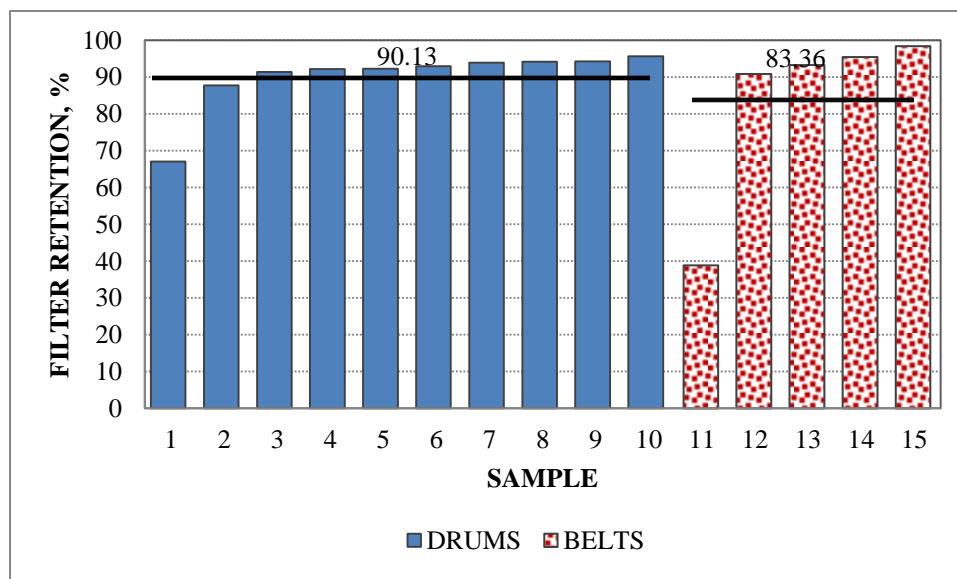


Figure 2. Filter retention for drum and belt filters.

The retention of all filters tested averaged 87.88% as shown in Figure 2. Retention varied from 38.89 to 98.38%. Retention rates above 90% are desirable.



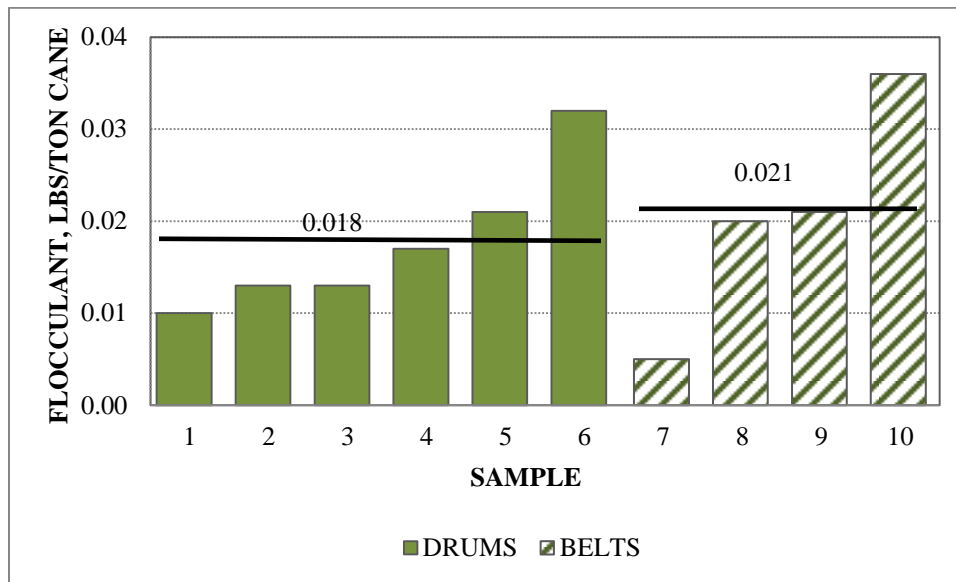


Figure 3. Crop average flocculant usage at the filter station.

Crop flocculant usage averaged 0.019 lbs./ton cane as shown in Figure 3. Belt filters used a little more flocculant (0.021) on average than drum filters (0.018).

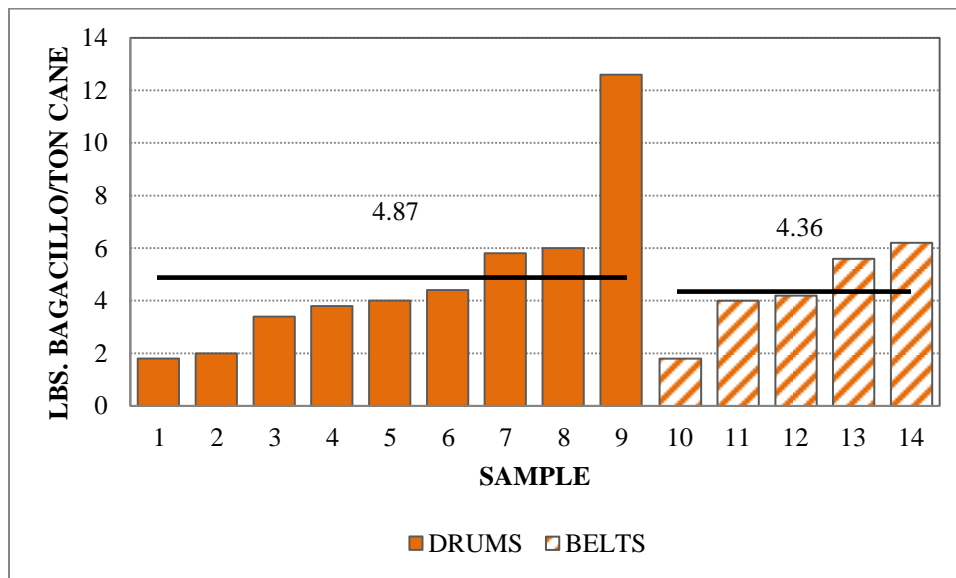


Figure 4. Bagacillo per ton of cane for drum and belt filters.

Figure 4 gives the bagacillo per ton cane for drum and belt filters. Drum filters averaged 4.87 lbs. bagacillo/ton cane ranging from 1.8 to 12.6. Belt filters averaged 4.36 lbs./ton cane of bagacillo with a range of 1.8 to 6.2 lbs./ton cane. The overall average was 4.69 lbs. bagacillo/ton cane.

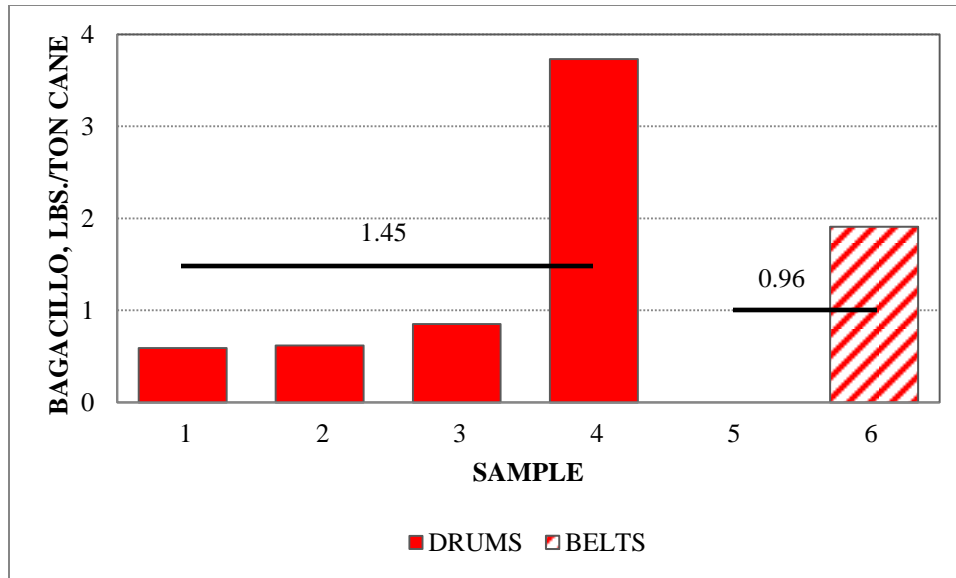


Figure 5. Pounds bagacillo per ton cane for drums and belts.

Pounds of bagacillo/ton cane added to filter cake averaged 1.28 overall. Shown in Figure 5 is the amount added for drums, ranging from 0.59 to 3.73 and averaging 1.45 lbs./ton cane. The addition of bagacillo for belt filters averaged 0.96 lbs./ton cane. Less than 30% of bagacillo in filter cake was added as screened bagacillo from mill-run bagasse with the vast majority provided from the clarifier underflow.

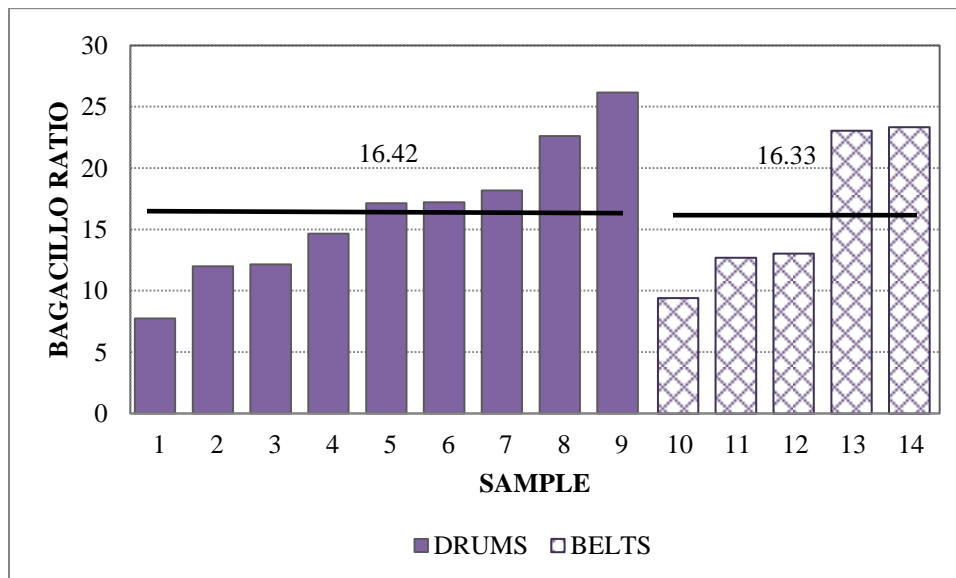


Figure 6. Bagacillo ratio for drum and belt filters.

The overall bagacillo ratio averaged 16.39 which was similar for both the drum filters (16.42) and belt filters (16.33) as displayed in Figure 6. Internationally, bagacillo ratios of 80 are recommended. To achieve this ratio about five times as much bagacillo as currently used would be required.

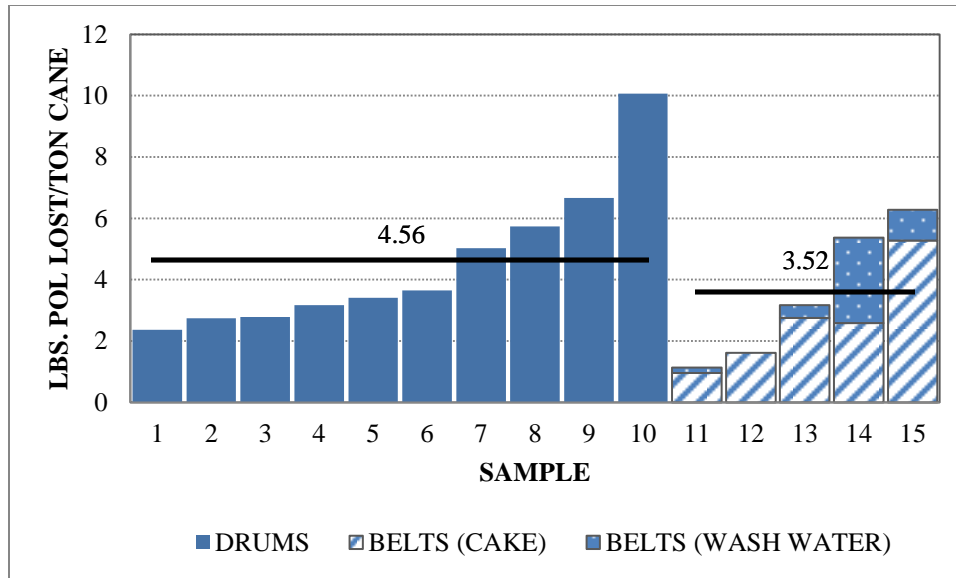


Figure 7. Pounds pol lost per ton of cane with belts based on 100 gpm wash water.

Figure 7 shows that on average 4.56 lbs. pol/ton of cane are lost in filter cake for rotary drum filters. Belt filters lost on average 3.52 lbs. pol/ton cane. This included losses in the belt wash water based on a rate of 100 gallons per minute.

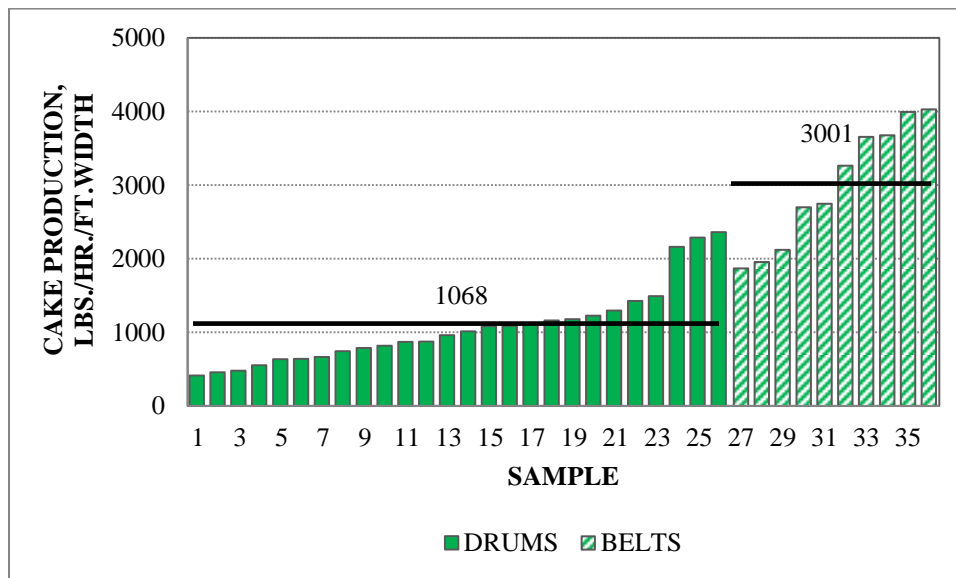


Figure 8. Filter cake production for both drum and belt filters.

The overall average filter cake production was 1605 lbs./hr./ft. width as shown in Figure 8. Drum filters averaged 1068 lbs. filter cake and ranged from 413 to 2362 lbs. filter cake/hr./ft. width. Belt filters averaged 3001 lbs./hr./ft. width, almost three times the amount for drums, and ranged from 1870 to 4027 lbs./hr./ft. width.

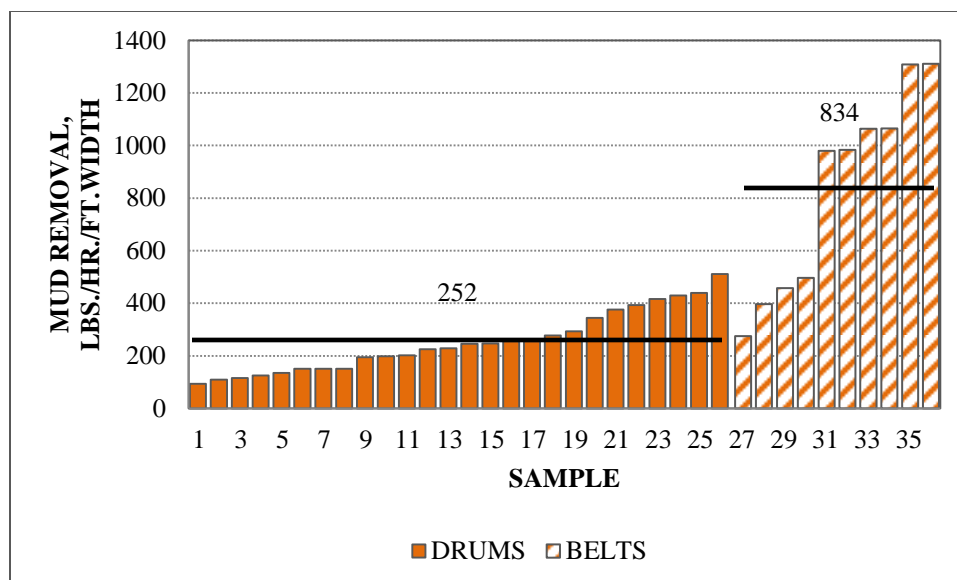


Figure 9. Amount of mud removal for drum and belt filters.

Mud removal averaged 413.81 lbs./hr./ft. width for all filters tested. Figure 9 shows the average mud removal for drum filters was 252 lbs. mud/hr./ft. width and ranged from 94 to 511 lbs. Mud removal for belt filters is also shown, averaging 834 lbs. mud/hr./ft. width and varying from 275 to 1311 lbs./hr./ft. width.

## SUMMARY

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

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

## ACKNOWLEDGEMENTS

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

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# **STARCH ANALYSIS OF FIRST EXPRESSED JUICE AND RAW SUGAR**

*Giovanna M. Aita*

## **INTRODUCTION**

Project 12-121 was funded by the American Sugar Cane League in 2012 and it is one of several future projects with aim at finding some answers to a common concern among sugar processors on the discrepancies of starch levels found in raw sugar. It is unclear whether this difference starts in the juice as it enters the factory or it is due to how efficient processing methodologies are employed at each mill. The objectives of this project were to analyze cane juice (first expressed juice) and raw sugar for the presence of starch during early and late grinding season.

## **ACCOMPLISHMENTS**

All eleven mills participated in this study. Samplings of first expressed juice and raw sugar (before entering the warehouse) were taken simultaneously at all mills on Wednesday, October 17<sup>th</sup> (early grinding season), Wednesday, October 24<sup>th</sup> (early grinding season), Wednesday, December 5<sup>th</sup> (late grinding season), and Wednesday, December 12<sup>th</sup> (late grinding season). Dr. Aita met with chief chemists from all eleven mills to coordinate the sampling of materials and discuss sampling methodologies. Sampling instructions were provided to each mill and discussed in detail with each chief chemist and personnel. Labeled sampling containers were personally delivered by Dr. Aita to all mills. A survey was developed to gather additional information about the weather, source of sugarcane (cane yard, fresh cane), and use of amylases during sampling. Samples and surveys were collected from mills at the end of each sampling date. Samples were analyzed for starch content at the Audubon Sugar Institute.

## **RESULTS**

Overall starch concentrations during early grinding of first expressed juice (120-327 ppm/Brix, Table 1) and raw sugar (178-388 ppm/Brix, Table 2) were higher than those observed for first expressed juice and raw sugar (106-239 ppm/Brix, Table 3; 133-257 ppm/Brix, Table 4) during late grinding, respectively. The weather conditions at the time of sampling during early grinding were mostly sunny and warm (70-85°F). Precipitation and cool (55-70°F) temperatures were reported at the first sampling during late grinding, and sunny and cold (<55°F) temperatures at the second sampling during late grinding. Most factories, with the exception of one, applied amylases to either the evaporators or syrup tanks. However, none of the factories were able to provide accurate information on the concentrations of amylase used. Therefore, it appears as if the discrepancies observed in starch concentrations in raw sugar could be due to differences in processing methodologies applied at each mill in addition to those observed in cane juice.

## ACKNOWLEDGEMENTS

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

Table 1. Starch analysis of first expressed juice during early grinding.

Sampling Date	Factory	Starch (ppm/Brix)	Cane Origin		Weather Conditions
			Cane Yard	Fresh Cane	
OCT 17 2012					
	1	212		X	Precipitation/Warm
	2	157		X	Overcast/Warm
	3	162		X	Sunny/Warm
	4	314		X	Sunny/Warm
	5	120	X		Sunny/Warm
	6	202	X	X	Sunny/Warm
	7	253		X	Sunny/Warm
	8	138		X	Sunny/Warm
	9	218	X		Sunny/Cool
	10	155	X		Sunny/Warm
	11	190		X	Sunny/Warm
OCT 24 2012					
	1	262		X	Sunny/Cool
	2	214		X	Sunny/Warm
	3	240		X	Sunny/Warm
	4	280		X	Sunny/Warm
	5	245	X		Sunny/Warm
	6	254	X	X	Sunny/Warm
	7	160		X	Sunny/Warm
	8	230		X	Sunny/Warm
	9	327	X		Sunny/Cool
	10	265	X		Sunny/Warm
	11	172		X	Sunny/Warm

Table 2. Starch analysis of raw sugar during early grinding.

Sampling Date	Factory	Starch (ppm/Brix)	Amylases	
			Addition	Location
OCT 17 2012				
	1	357	No	N/A
	2	302	Yes	Evaporator
	3	216	Yes	Evaporator
	4	199	Yes	Syrup Tank
	5	217	Yes	Evaporator
	6	286	Yes	Evaporator
	7	230	Yes	Evaporator
	8	388	Yes	Evaporator
	9	245	Yes	Evaporator
	10	305	Yes	Syrup Tank
	11	181	Yes	Evaporator
OCT 24 2012				
	1	413	No	N/A
	2	318	Yes	Evaporator
	3	189	Yes	Evaporator
	4	240	Yes	Syrup Tank
	5	178	Yes	Evaporator
	6	290	Yes	Evaporator
	7	203	Yes	Evaporator
	8	255	Yes	Evaporator
	9	317	Yes	Evaporator
	10	267	Yes	Syrup Tank
	11	258	Yes	Evaporator

N/A= Not Applicable

Table 3. Starch analysis of first expressed juice during late grinding.

Sampling Date	Factory	Starch (ppm/Brix)	Cane Origin		Weather Conditions
			Cane Yard	Fresh Cane	
DEC 5 2012					
	1	176		X	Precipitation/Cool
	2	152		X	Overcast/Cool
	3	151		X	Overcast/Cool
	4	166		X	Precipitation/Cool
	5	174	X		Precipitation/Cool
	6	202	X	X	Sunny/Cool
	7	147	X		Precipitation/warm
	8	202		X	Precipitation/warm
	9	217	X		Precipitation/Cool
	10	218	X		Sunny/warm
	11	220	X		Precipitation/warm
DEC 12 2012					
	1	116		X	Sunny/Cold
	2	140		X	Sunny/Cold
	3	195		X	Sunny/Cold
	4	162		X	Sunny/Cold
	5	182	X		Sunny/Cold
	6	239	X	X	Sunny/Cold
	7	159	X		Sunny/Cold
	8	106		X	Sunny/Cold
	9	231	X		Sunny/Cold
	10	182	X		Sunny/Cold
	11	135	X		Sunny/Cold



Table 4. Starch analysis of raw sugar during late grinding.

Sampling Date	Factory	Starch (ppm/Brix)	Amylases	
			Addition	Location
DEC 5 2012				
	1	152	Yes	Syrup Tank
	2	222	Yes	Evaporator
	3	133	Yes	Evaporator
	4	149	Yes	Syrup Tank
	5	173	Yes	Evaporator
	6	209	Yes	Evaporator
	7	167	Yes	Evaporator
	8	207	Yes	Evaporator
	9	162	Yes	Evaporator
	10	181	Yes	Syrup Tank
	11	182	Yes	Evaporator
DEC 12 2012				
	1	154	Yes	Syrup Tank
	2	133	Yes	Evaporator
	3	240	Yes	Evaporator
	4	170	Yes	Syrup Tank
	5	195	Yes	Evaporator
	6	248	Yes	Evaporator
	7	209	Yes	Evaporator
	8	142	Yes	Evaporator
	9	257	Yes	Evaporator
	10	238	Yes	Syrup Tank
	11	181	Yes	Evaporator

# IMPROVEMENTS OF RAW SUGAR QUALITY USING DOUBLE PURGE OF C-MASSECUITE

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## INTRODUCTION

The color in raw sugar is influenced by many factors (Chen & Chou, 1993) such as: quality of the cane processed (type of soil, variety, ripeness, deterioration level and trash content); juice clarification (excess of lime salts and bagacillo in the juice); and evaporation and crystallization systems (temperature and molasses recirculation). The main factors that can be controlled insuring high raw sugar quality and maximizing sugar recovery are: (Rein, 2007; Chou, 2000)

- Controlling pan operation (to avoid uneven and agglomerated grain, and overheating)
- Guaranteeing the highest obtainable crystal content on each strike (reducing the recycle of materials)
- Reducing remelt to a minimum
- Proper centrifugal operation for high and low grade massecuites (avoiding excessive washing to minimize molasses production)

Chou (2000) and Bento (2008) stated that the best way to achieve a good sugar quality (low color and low ash content) at the boiling house is insuring, independent of the boiling scheme, a good C-magma quality (the seed for the first and/or the second strike).

The implementation of double purge to a three-boiling scheme does not involve significant changes to the boiling house but will upgrade the quality of the seed used in the A and B strikes. A lower recirculation of final molasses with the magma will improve not only the quality of the raw sugar produced but also the quality of the first (A) and second massecuites. Improvement of A and B strike quality will be due to a lower recirculation of non-sugars from the low grade massecuites, reducing color and viscosity of the mother liquors. This may also benefit the sugar yield from the first massecuites by lowering the purities of the A and B molasses.. The system can be implemented, with minor changes to the traditional 3-boiling scheme, by addition of a second centrifugation stage after the centrifugation of C-massecuite (Figure 1). Little or no water is applied at the first stage, obtaining magma of 79 – 81 purity. In the second centrifugation, the magma is affined producing 92 – 94 purity. The 64 to 67 purity wash molasses can be blended with either B or A molasses. The higher purity magma becomes the footing for A and B massecuites.

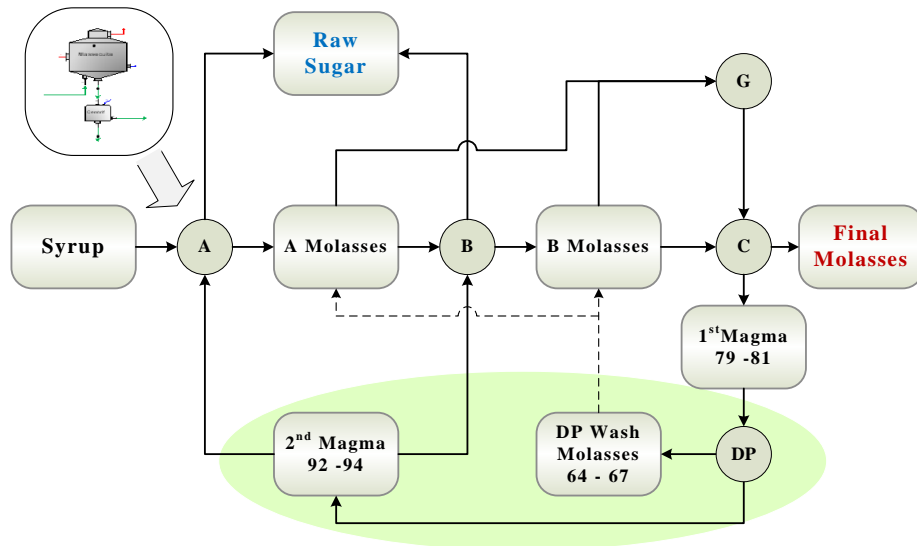


Figure 1. Three-boiling scheme + double purge of C-magma showing the materials going in and out for each crystallization stage

## IMPLEMENTATION OF C-MAGMA DOUBLE PURGE AT THE LULA FACTORY

A double purge system for Lula factory was designed and integrated to the traditional boiling scheme in 2012. Figure 2 shows the details of the design.

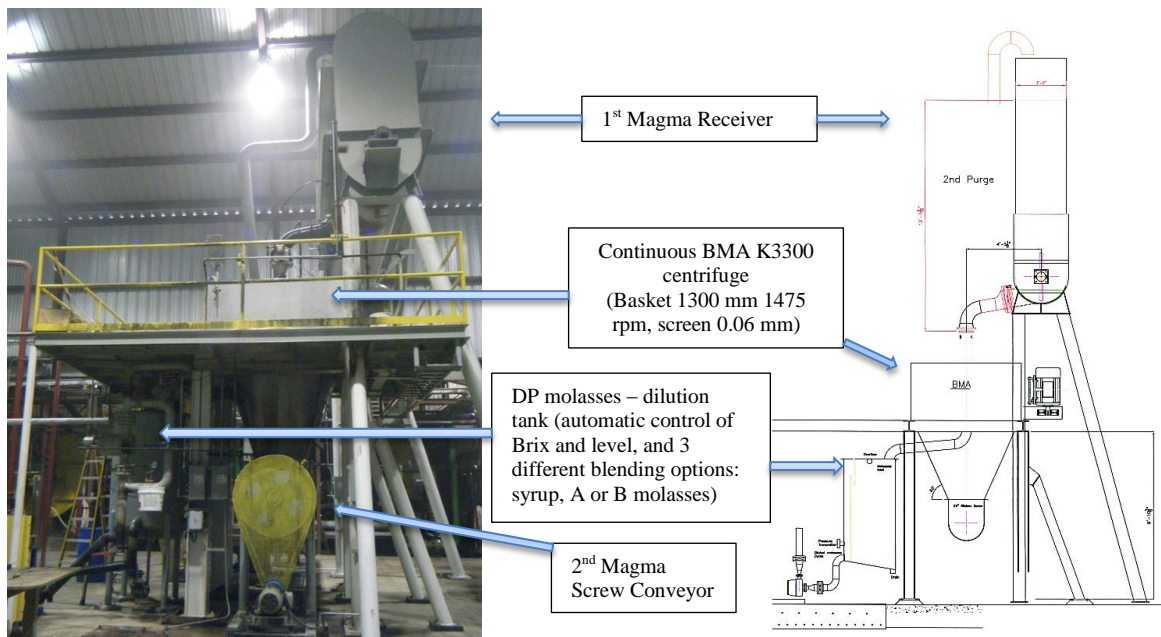


Figure 2. Design and implementation of the double purge system at Lula Factory (Eng. Gregory Carline)

During the 2012 crop season, the double purge system was operated continuously, stopping only for liquidation at the end of the season. Because of the high purity of the double purge molasses, this stream was routinely sent to the A molasses tank. Analyses of the two new streams – 2nd magma and double purge wash, were added to the lab work load. The expected purity range for these parameters were defined by the Audubon Sugar Institute staff and closely supervised by Lula personnel. The results of the analysis of raw sugar from the double purge system at Lula factory were compared to those from Westfield factory, which also operates a traditional 3-boiling scheme and had equivalent quality of cane (common sources).

## RESULTS AND DISCUSSION

Figure 3 shows the daily average purities of the magmas and double purge (DP) molasses throughout the season. Spikes of low purity wash molasses probably were due to leaks of final molasses to the C-magma at the first set of centrifuges (4 SW 4630 – basket 1170 mm, 2000 rpm and screen 0.06 mm).

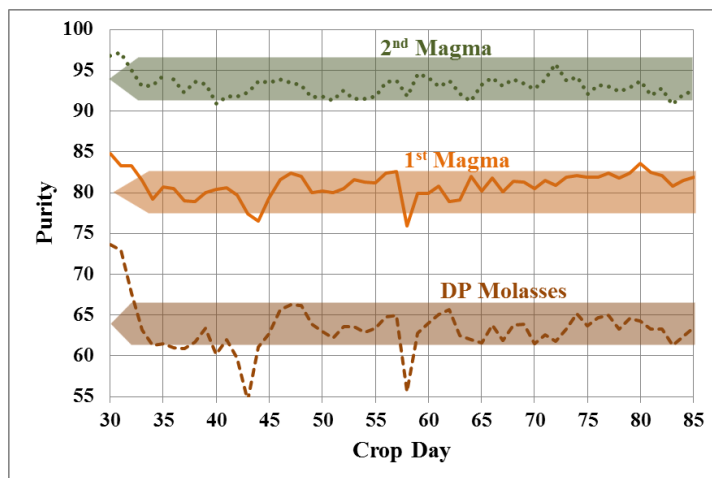


Figure 3. Double purge molasses target parameters (2<sup>nd</sup> magma, 1<sup>st</sup> magma and double purge wash molasses purities)

Figure 4 compares pol and color of raw sugar produced at Lula compared with sugar produced by Westfield. The level of improvement on whole color for a polarization of 99.2 °Z was ~ 40%. Affined color, which is related to the color occluded in the core of the crystal was lowered by ~15% at the same polarization.

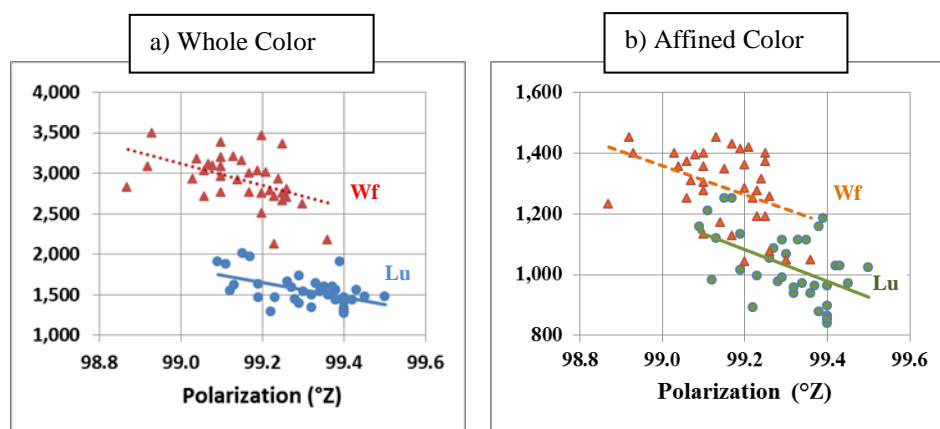


Figure 4. Raw sugar analysis results comparison between Lula (Lu) and Westfield (Wf):  
a)Whole Color (CU 8.5 pH), b)Affined Color (CU 8.5 pH)

Table 1 shows a weighted average of sugar quality parameters evaluated for one month of continuous operation of double purge system (10/23/12-11/26/12). Conductivity ash was 57% lower for Lula raw sugar than for Westfield. Other impurities, i.e. reducing sugars, starch or dextran did not improve with a double purge of C-magma.

Table 1. Comparison of raw sugar parameters (weighted-average) between Lula and Westfield

Parameter	Unit	Lula	Westfield	%Δ
Polarization	°Z	99.3	99.1	-0.2
Whole Color	CU	1552	2923	47
Affined Color	CU	1026	1289	20
Ash (Conduct.)	%	0.07	0.15	57
Reducing Sugars	%	0.15	0.17	
Starch	ppm	188	187	
Dextran	MAU	31	26	

Table 2 shows a summary of color profiles for each strike. Double purging of C-magma produced a 75% color removal. Color ratio's of the massecuite to whole raw sugar color was about 0.04 for A massecuites and 0.03 for B massecuites.

Table 2. Boiling house color profile at pH 8.5 with 0.45 μm Nylon membrane filter

Material	A Strike	B Strike	C Strike
Feed	22590	48590	76220
Seed	20640	21020	70770
Massecuite	19680	38940	73190
Sugar	740	1270	42060
Double Purge			10620

Table 3 compares some factory performance parameters from the last 3 years'. The double purge system had no impact the overall recovery of Lula factory in 2012, even though grinding rates had increased ~20% from 2010. Lula reduced the true purity of final molasses by 8% over this period (ASI – Molasses Survey), however, the target purity difference (TPD) did not show improvement (TPD=9.7) from 2011 to 2012, although it dropped by 10% from 2010. Lula and Westfield had similar overall recoveries (85.8% and 85.5% respectively) during 2012 and same drop in molasses true purity (8%).

Table 3. Comparison of 3 years of some performance parameters from Lula's factory reports and from Audubon Sugar Institute's "molasses survey"

<b>Parameter</b>	<b>Units</b>	<b>2010</b>	<b>2011</b>	<b>2012</b>
<b>Ground Cane</b>	<b>Tons/day</b>	9,168	10,604	10,886
<b>Syrup Purity</b>	<b>%</b>	85.9	85.6	86.4
<b>Overall Recovery</b>	<b>%</b>	85.6	85.3	85.8
<b>Molasses True Purity</b>	<b>%</b>	46.4	45.1	42.5
<b>Molasses TPD</b>	<b>%</b>	11.9	9.7	9.7

## CONCLUSIONS

A comparison of results obtained during the 2012 season for two Louisiana factories, processing cane from the same sources, showed that the double purge system improved the raw sugar color approximately ~50% (1,500 compared to 3,000 CU at pH 8.5). The ash content was reduced ~60% and the affined color was reduced ~20%. Other impurities in raw sugar were not affected by this modification of the boiling scheme. There was no detectable effect on factory performance even though grinding rates were higher than in previous seasons. Still there is room for improvement in factory performance at Lula. The lessons learned in 2012 have given insight into methods to enhance performance using double purge of C-magma.

## **ACKNOWLEDGEMENTS**

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- The American Sugar Cane League
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# THE MOLASSES SURVEY

*C. Verret, D. Dorman, & S. Lu*

## INTRODUCTION

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

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

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

The TP is subtracted from the true purity to give a target purity difference or TPD. The TPD is used by the factories to determine how well they are recovering sugar from their massecuite (which is reflected by residual sugar in the molasses). “True purity” is the sum of the non-crystallizable sugar and that which was crystallized, but was lost across the centrifugals. 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 2011 survey season stretched from 09-25-11 until 12-25-11 and the 2012 survey season stretched from 09-23-12 until 01-06-13. A total of 182 samples were analyzed in duplicate for the 2011 season and 208 samples were analyzed in duplicate for the 2012 season. Including standards, this totaled 532 samples for 2011 and 608 samples for 2012. Analyses included:

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

\*Because we measure sugar using HPLC, we perform a direct polarization of molasses clarified using Octapol<sup>TM</sup> (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 2012 season was longer than the 2011 season. The 2011 season operated for 14 weeks and the 2012 season operated for 16 weeks. The 2011 & 2012 seasons TPD weekly averages started at their maximum value of 14.1 & 10.5. As the seasons continued the TPDs took it usual downward trend towards the end of the seasons. The 2011 and 2012 seasons demonstrated little variation. Industry average TPDs for 2011 and 2012 was 7.8, respectively. (Figure 1)

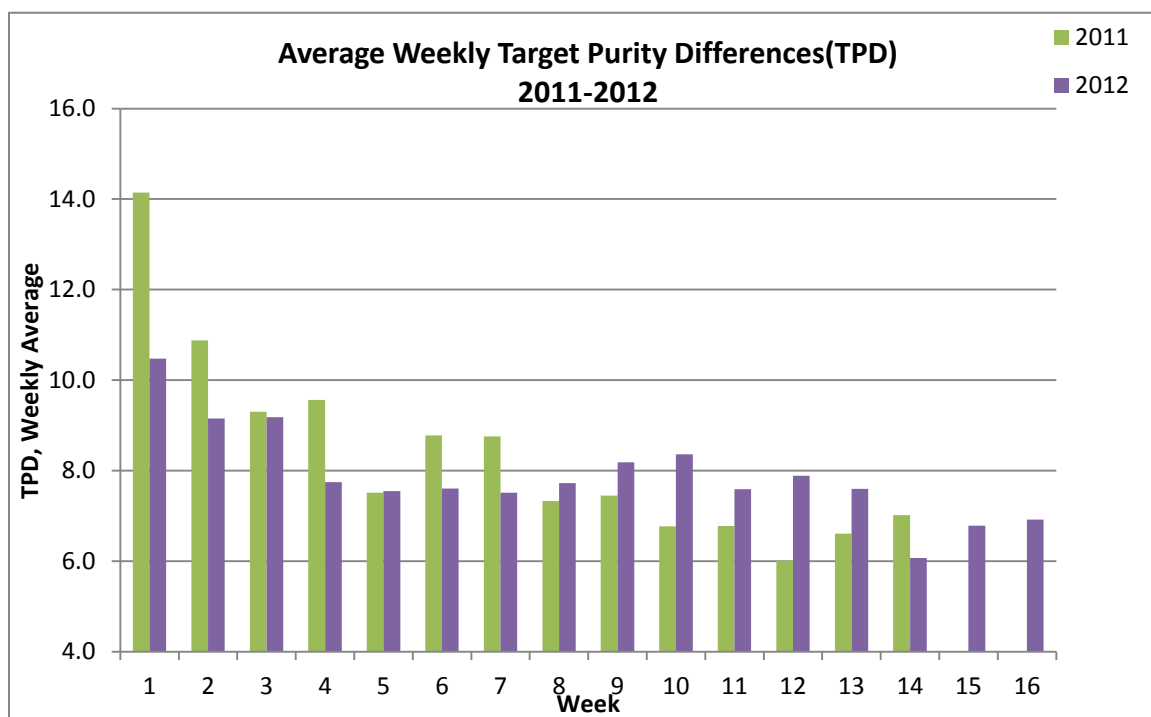


Figure 1. 2011-2012 Average Weekly Target Purity Differences

The conductivity ash component for the 2011 & 2012 seasons were statistically consistent. They both started their seasons at their minimum values of 11.5 and 12.1. As the seasons continued the ash increased to their maximum values of 17.8 & 17.3. (Figure 2)

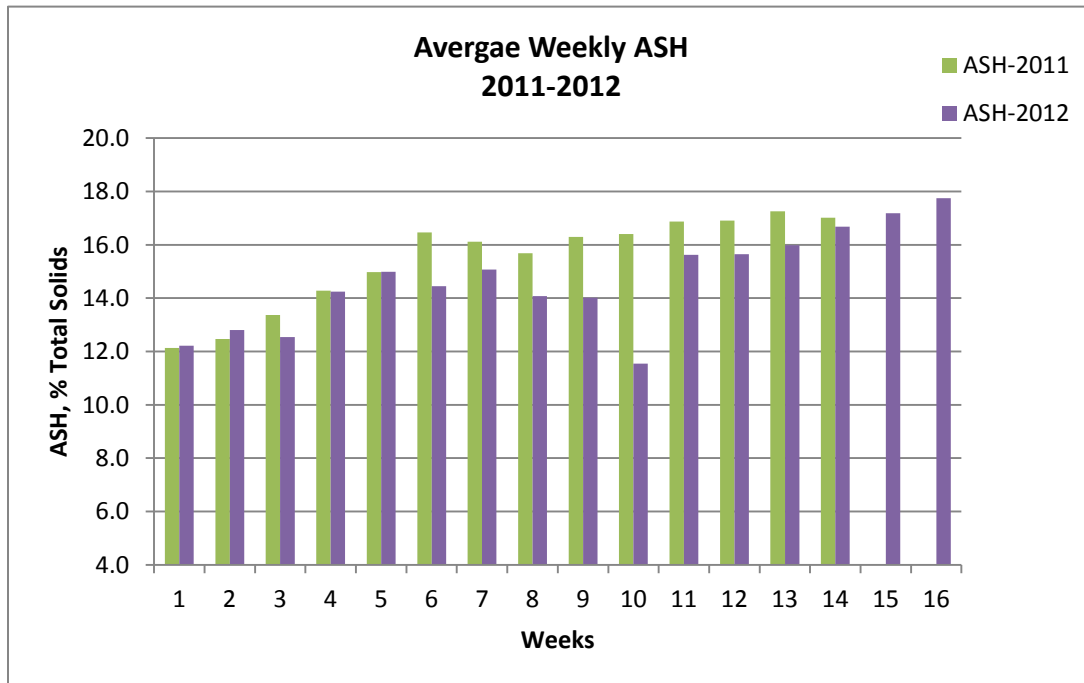


Figure 2. 2011-2012 Average Weekly Conductivity Ash

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

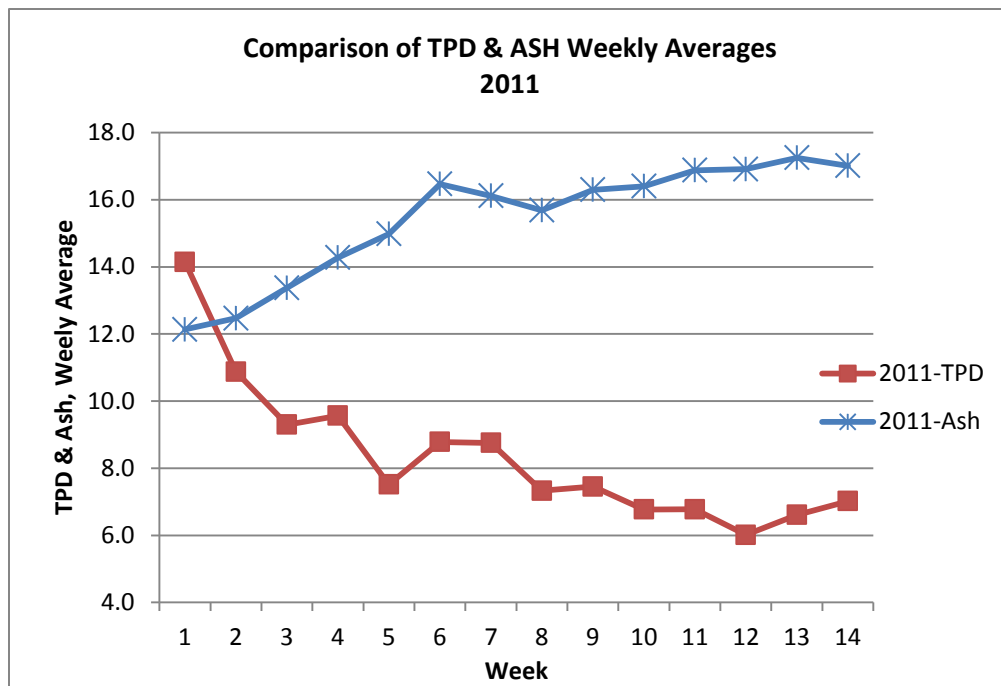


Figure 3. 2011 TPD & Ash comparison

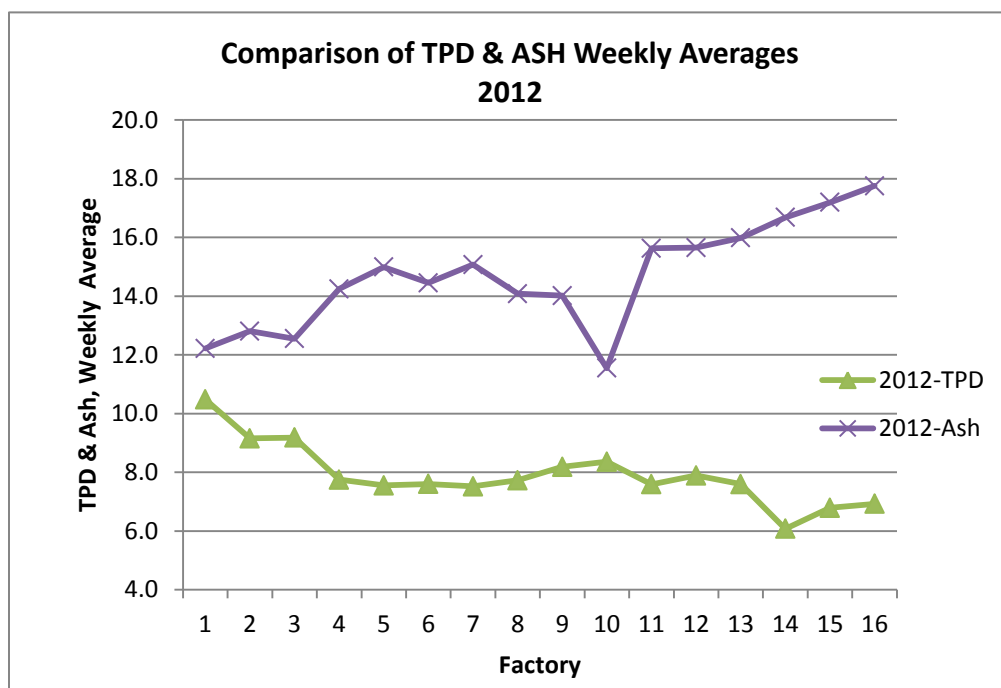


Figure 4. 2012 TPD & Ash comparison

In general, there has been a significant downward trend, relative to time, in the amount of reducing sugar in final molasses. This was attributed to the observation that the mills were grinding more cane and had installed evaporation capacity sufficient to minimize the sucrose inversion that would take place. This is evidenced by the decrease in reducing sugar over time. (Figure 5)

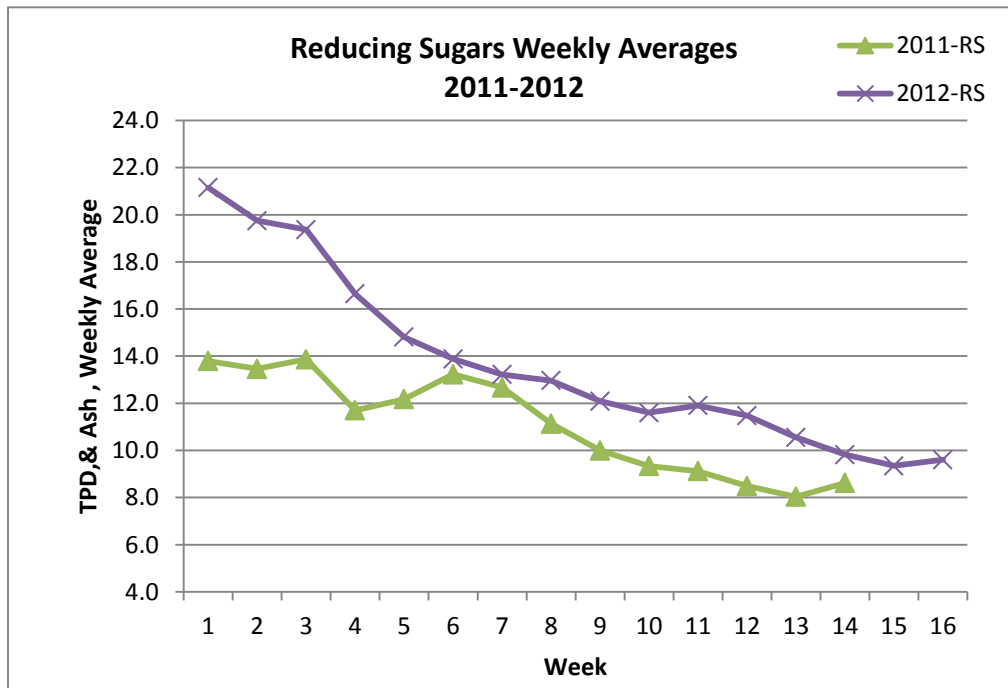


Figure 5. 2011- 2012 Reducing Sugars Weekly Averages

For the 2012 season, the true purity yearly average was 42.4%, the F/G Ratio was 1.63 and the target purity was 34.7%.

Comparing the results from the 2011 and the 2012 seasons to the results from the 2010 season showed the downward trend of the yearly average TPD. This is demonstrated in Figure 6. The 2012 season maximum TPD was 9.7 which was a difference of 1.1 from the previous season and a difference of 2.2 from the 2010 season. The minimum TPD for 2012 and 2011 was 5.8 a difference of 0.8 from the 2010 season. (Table 1)

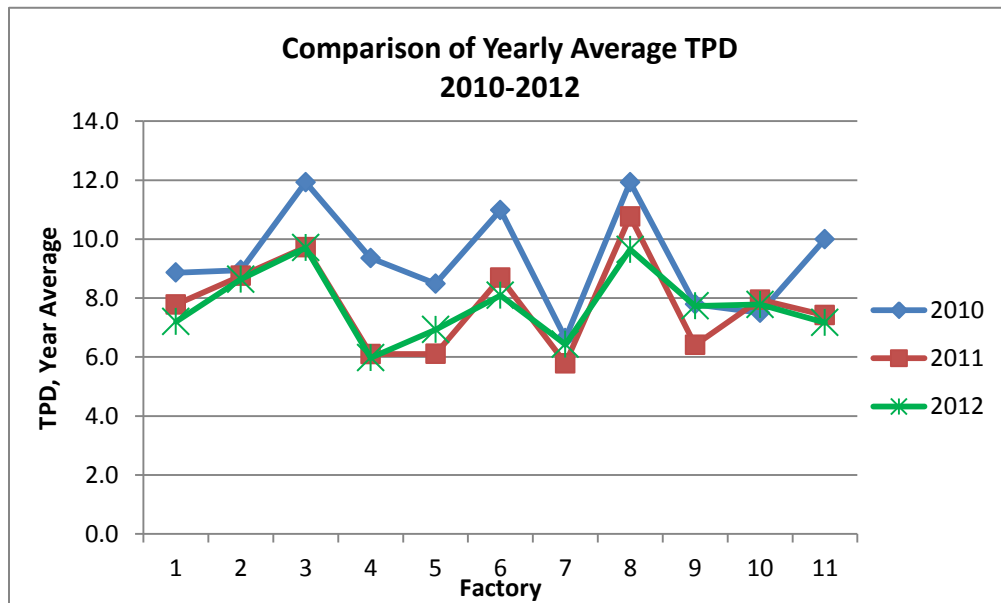


Figure 6. Comparison of TPD 2010-2012

TPD Data Summary for 2010-2012			
Year	TPD Minimum	TPD Maximum	TPD Average
2010	6.6	11.9	9.3
2011	5.8	10.8	7.8
2012	5.8	9.7	7.8

Table 1. Summary of TPD 2010-2012

## CONCLUSIONS

The seasonal average TPD remained the same at 7.8 for the 2012 season. The ash decreased for the 2012 season by 1.2% from the 2011 season which had an ash content of 15.9. The reducing sugars increased for the 2012 season to 13.1% from 10.9% from the previous season. The true purity, target purity and F/G ratio all decreased in the 2012 season.

The differences can be attributed to a wide range of factors which included favorable weather and harvest conditions, the introduction of a new cane variety, cane maturity and increased awareness at the cane delivery/mill level.

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

Historical data suggests that a TPD of five remains a rational and encouraging objective for the 2013-2014 grinding season.

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## **BIOFUELS UPDATE**

*D. F. Day*

In 2011 the LSU AgCenter was awarded an AFRI-Cap Grant for \$17.5 million. This is a multi-state, multi-disciplinary grant with a goal of expanding the Southern Regional Agricultural Sector by utilization of sweet sorghum and energy cane to produce butanol, gasoline, isoprene and by-product chemicals. This multidisciplinary regional consortium of agricultural scientists, biotechnologists, technology and engineering companies, economists and educators will address multiple aspects associated with conversion of energy cane and sweet sorghum into a portfolio of bio-based fuels and chemicals. LSU plans to use energy cane and sorghum to help reinvigorate the Louisiana sugar and chemical industry through new and existing industrial partnerships. Improving biomass cold tolerance and production characteristics can produce a steady stream of biomass to be converted to economically viable sugars using existing Louisiana refinery infrastructure. If successful, the project will contribute significantly to improving rural prosperity and job creation in the region.

### **PROJECT OBJECTIVES**

- Evaluation of selected energy cane and sweet sorghum crops and improvement in their production through utilization of low-input, sustainable systems to ensure an uninterrupted supply of carbohydrates and fiber to biofuel production facilities.
- Utilization of existing pilot and industrial facilities, incorporating multiple crops and cutting edge processing technologies to demonstrate butanol, gasoline, isoprene and specialty chemicals.
- Development of regionally appropriate business-marketing models that integrate bio-based fuels and products into existing logistics and supply chain infrastructures based on inputs from agricultural research and techno-economic analyses.
- Expansion of educational programs at the consortium universities to support a practical training center in biofuel processing linked to an extension/outreach program targeting supply chain participants.

The Sustainable Bioproducts Initiative (SUBI) at the LSU AgCenter is working to expand the Southern Regional Agricultural Sector through the utilization of sweet sorghum and energy cane crops for the manufacture of bio-based fuels and by-products. SUBI is a regional, multidisciplinary consortium of agricultural scientists, biotechnologists, technology and engineering providers, economists and educators that facilitate the development of regional crops into a portfolio of bio-based fuels and chemicals. The project examines the areas of bio-based fuel and chemical production from feedstock development to conversion. The agricultural economics group examines production costs and economic viability of the proposed processes. An education group seeks to develop educational opportunities in the bioenergy field for students at several regional universities and beyond. The extension team plans, organizes and executes events that reach out to stakeholders at all levels to turn research results into actual business development.

## **FIRST YEAR PROGRESS BY TASK**

Feedstock Development for energy cane has the objectives of optimizing yields, expanding diversity and range of cultivation and reducing inputs through breeding. Cross pollination was achieved between energycane and miscanthus. These crosses are now being evaluated for cold tolerance. The goal is to extend the range for energycane to allow it to be grown as far north as Arkansas. Both in order to speed up breeding analysis and develop it as a use for biomass crops Near Infra-red Spectrometry (NIR) is being calibrated for sweet sorghum and energy cane and analysis being expanded outside the traditional brix and sucrose to include invert, cellulose and hemicellulose. Preliminary calibrations have been obtained for these parameters for sweet sorghum and are progressing on energy cane, but will require many iterations to establish reliable databases.

Sustainable Feedstock Production focuses on production of sweet sorghum and energy cane as feedstocks for the biorefinery. For energy cane, the enhancement of cold tolerance and optimization of its production potential under temperate climate regimes are being studied. For sweet sorghum, the input requirements, the evaluation of geographic zone of adaptation, inclusive of its ability to maintain juice quality into the fall season, the ability to produce commercial yields on marginal soil and respond to low-input sustainable production practices are being addressed. The baseline soil nutrient information gained from year one pre-treatment sampling will identify any pre-treatment biases and serve as a basis for observing changes in soil fertility and carbon resulting from establishment and management of these sugarcane cropping systems. Four sweet sorghum plots established across state produced yields ranging from 17-38 wet ton/acre. N and P soil content measured. Ceres Corporation agreed to supply cold tolerant varieties for testing.



Logistics and Pre-Processing involves with the assessment of harvesting biomass, transportation to the plant, storage, treatment by milling or diffusion and bagasse drying for future usage. Experiments have been planned to determine the harvest losses and leaf matter distribution of each of these crops with two types of harvesters. Harvesting trials have been pre-planned in collaboration with John Deere and harvester modifications are being made for sweet sorghum. Protocols for the trials allow for the evaluation of losses of simple sugars during harvesting, transportation and pre-processing storage. Literature has been prepared on bagasse storage methods. The bagasse will be required to supply power to the biorefinery during the off season. Work continued with our industrial partners to develop a use of industrial dryers for bagasse to provide energy efficient solutions for a variety of biomass processing applications.

Fractionation (milling) of sweet sorghum and energy cane samples have been conducted to determine the amount of simple vs. complex (bagasse) sugars from each crop as well as the particle size distribution of stored bagasse. Our operational concept requires year round operation of a primary processing plant operated that produces fermentable sugars that can act as a shippable feedstock for centralized biorefineries. The primary processing facilities of necessity will be biomass (bagasse) powered. A model has been developed based on SUGARS TM software that allows calculation of heat and material balance for the primary processing plants and estimates the required internal power generation and residual bagasse that can be used either for power generation or as a source of additional sugars.

Feedstock Conversion and Refining Part of the task is to produce sufficient quantities of test syrups, in a scalable manner for our industrial partners to develop their processes. The pilot plant area for processing experimental lots of energy cane and sweet sorghum has been expanded to accommodate new equipment designed for simulating a primary processing plant and produce stable sugar syrups for future processing. A new facility capable of 1 ton/hr processing has been completed. It is shown in comparison with the “old” Audubon facility that was on campus. This will be used to produce syrups from the test feedstocks for our industrial partners to try in their facilities. On site work for bioconversion has focused on ammonia pretreatment for lignocellulosic biomass. This process has been optimized. Work with an industrial partner (Optinol) has led to development of a continuous butanol production column around which they are developing a process.



1980  
30 tons/hr



2013  
1 ton/hr



1980  
500 gal Juice per Batch  
50 gal of Syrup



2013  
Continuous Production  
Target 15 gal/hr Syrup

As a starting point, the Economics team evaluated the costs of producing energy cane as a biofuel feedstock for five varieties of energy cane currently being grown by the LSU AgCenter at its Sugar Research Station in St. Gabriel, LA. High energy sorghum cost of production was estimated for the Upper Coastal Bend area of Texas based on yield estimates provided by Texas A&M Soil and Crop Sciences personnel and enterprise budgets from Texas AgriLife Extension Service.

### **Cost of Production of Energy Crops Initial Estimates**

Crop	Production Cost (\$/ton)		Product Value (\$/ton)	
	Crop (wet)	Fiber (dry)	Simple sugars	Fiber
Energy cane	23	96-99	?	?
Sweet Sorghum	?	69	?	?

These numbers are a starting point and will be firmed up as experience is gained with crops, varieties, and yields on processing.

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The Education group focused on developing short courses and educational programs, as well as developing an activity-safety resources map based on existing resources on health and safety in both farming activities and pilot plant operation. Educational programs are being incorporated through existing degree offerings for students and seminars and workshops for teachers and other members of the industrial community. New course offerings have been filed with the appropriate Universities and are currently proceeding towards approval.

- LSU Engineering (Chemical and Biological) and Southern Univ. are involved in setting up programs to interest students in biofuel processes. Programs are being designed and submitted to the respective administrations.
- A workshop and 6 bioenergy presentations to students were made this year

The Extension task takes research and its associated technologies to a broader audience of stakeholders. The goal is to deliver science-based knowledge that initiates creative thinking as ideas transform into business development. Several field day events were held throughout the state. There was also participation in conferences and workshops, and publications of surveys, guides and articles in trade journals. An extension program has been established to familiarize

farmers with these crops and an education program is being established for training people for work in the biofuels industry.

- A National energy cane/sweet sorghum field day was held at the USDA- Houma station.
- A guide to identification of energy cane varieties was prepared and distributed.
- A survey was conducted of growers about their feelings toward growing crops for biofuel production.

The first year has seen the determination that sweet sorghum and energy cane are suitable crops for the production of biofuel and bio-based chemicals in the Southeastern Region, and that the approach of using crops with staggered harvest times is feasible. A pilot plant facility was constructed and should be ready on schedule. Plant breeding programs have made a number of successful crosses which are being evaluated for cold tolerance and range. Preliminary economic analysis has been conducted on the proposed crops and information is being obtained for life cycle analysis. Education programs are being established and research on product utilization is proceeding on schedule

**So what will constitute success of this program?** A successful program will provide new crops that can be produced sustainably in this region and the technology for converting them locally to fermentable sugars, that in turn, can be used in centralized biofuel production facilities.