

Audubon Sugar Institute 2012-2014 Report



Sugar and Bioproducts Research
for Louisiana



Audubon Sugar Institute

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Audubon Sugar Institute Goals

- To enhance the productivity and profitability of the Louisiana sugar industry and other sugar-processing-related industries.
- To improve the practice of sugar manufacturing through education and technology transfer.
- To conduct research toward a diversified sugar-processing industry.
- To attract, retain and develop a world-class staff to serve our stakeholders.
- To encourage use of low environmental impact technologies in sugar processing.

Front cover photo: Audubon Sugar Institute, LSU AgCenter, with a Squier mill from the original Audubon Sugar Factory on display.
Back cover photo: The milling tandem at Alma Plantation, LLC, in Lakeland, Louisiana.



Dr. John Russin

Once again it is a pleasure to provide overview comments for this issue of Audubon Sugar Institute report. ASI remains an important part of the LSU Agricultural Center and a key partner in our state's sugar industry.

Sugarcane remains a very strong part of Louisiana's agricultural economy. Data from the 2013 Louisiana Summary identify sugarcane as the fifth most valuable agricultural commodity following forestry, poultry, feed grains and soybeans. While down in value from previous years (primarily due to lower prices), the recent crop yielded the most recoverable sugar per acre in recorded history, making it our sweetest crop ever.

The production and refining processes for Louisiana sugarcane rely heavily on several engineering fields. ASI was fortunate to hire two new faculty members who bring the unique combination of engineering backgrounds coupled with sugar production experience. Dr. Daira Aragon, a Colombia native, earned the Ph.D. in Chemical Engineering at LSU and now works in sugar process engineering, material and energy balances, modeling and simulation and optimization and control. Dr. Franz Ehrenhauser, a native of Austria, also earned the Ph.D. in Chemical Engineering at LSU and works to develop new technologies for the sugar industry and separation technologies for production of syrups.

For the past three years, ASI also has taken a lead role in a \$17.2 million award from the USDA NIFA entitled "A Regional Program for Production of Multiple Agricultural Feedstocks and Processing to Biofuels and Biobased Chemicals." The main goal of this five-year project is to expand the agricultural sector in the southern region by developing production and utilization systems for "dedicated energy crops" that USDA had determined were optimal for our region – energy cane and sweet sorghum. The project has four main objectives: 1.) Evaluation and improvement of energy crops for sustainable production in low-input marginal systems; 2.) Development of pilot and industrial facilities for syrup and biomass production that will support fuel and specialty chemical production; 3.) Identifying regionally appropriate business, logistic and supply chain models; and 4.) Offering practical training for students, supply chain participants and the public.

Scientists at ASI, indeed scientists throughout the LSU Agricultural Center, have greatly expanded their programs to accommodate this project to build upon and add value to the existing sugarcane harvesting and processing infrastructure in south Louisiana. A one-of-a-kind pilot scale facility is entering its final phases of facility optimization at ASI and will serve the existing sugar industry long after this award is complete. ASI scientists have broken new ground in separation technologies that may enable production of high-value specialty chemicals from syrups. And economists are developing harvest, transport and processing logistic, again using the Louisiana sugarcane industry as a model, for biomass crops that could be produced in our region.

The three-way partnership among American Sugar Cane League, USDA-ARS and LSU Agricultural Center has enjoyed decades of success in jointly serving all aspects of the sugar industry. These shared efforts in variety development, production, harvesting and processing have helped sustain the sugar industry in south Louisiana that has thrived for more than 200 years. We are proud to be part of this team effort and look forward to future advances and success.

Director's Perspective: Sugar Processing in Louisiana



Benjamin L. Legendre, Professor and Department Head, Audubon Sugar Institute, LSU AgCenter, St. Gabriel, Louisiana

Louisiana is the oldest and most historic of the sugar-producing areas of the United States. Sugarcane arrived in Louisiana with the Jesuit priests in 1751 who planted it near where their church now stands on Baronne Street in New Orleans. Sugarcane was planted within the city limits of New Orleans, and in 1795 Etienne DeBoré first granulated sugar on a commercial scale at a site near Audubon Park. Except for disastrous production years during the Civil War, the disease epidemics of the 1920s and from a severe freeze of 10 F during December 1989 affecting the 1990 crop, the Louisiana sugarcane industry has continued to prosper, mainly due to improved varieties, cultural practices, pest control and sugar-processing innovations. The Louisiana sugarcane industry is currently in its third century of uninterrupted sugar production.

The gross farm value of the 2014 sugarcane crop was \$ 438,975,767 for sugar and molasses. The gross farm value represents 60 percent of the value of the sugar and 50 percent of the value of molasses produced. The remaining percentages are for processing and marketing, which amounted to \$ 303,020,834.

Therefore, the total value of the sugarcane crop to Louisiana producers, processors and landlords at the first processing level was \$741,996,601. Using the economic multiplier, the sugarcane crops from 2012-2014 generated more than \$2 billion each year to the State. Sugarcane continues to rank first in value among the state's row crops.

The 2014 sugarcane variety census showed Louisiana producers continued to rely on several varieties thereby spreading their risk. The most widely grown variety was HoCP 96-540, which was grown on 37 percent of the production acres. This was followed by L 01-299 (22%), L 99-226 (13%), L 01-283 (10%), HoCP 04-838 (6 %), HoCP 00-950 (4 %) and L 03-371 (3 %). There has been a trend in recent years to plant less HoCP 96-540 and more L 01-299.

For the years covered by this report, sugar yield (lb. of sugar/acre) at the beginning of the harvest was good considering that growers harvested their older stubble crops and heavy clay land first. Although the Louisiana breeding programs have developed sugarcane varieties that tend to have an earlier maturity and higher sugar yield, the use of the chemical ripener glyphosate by a majority of the growers has helped increase recoverable sugar, especially at the beginning of the harvest, from 10-30 lb./ton, depending upon the variety. All 11 factories take part to various degrees in the use of chemical ripeners by their growers. The 2014 harvest produced record sugar recoveries and variety response to ripeners was some of the best seen in many years. Sugar recoveries were also higher due to the erectness of the crop and a mostly dry harvest season that allowed proper topping and removal of leafy trash along with a minimal amount of field soil entering the factories.

Although nine factories have closed in the last 20 years, the remaining 11 factories have increased their capacity to absorb the cane supply that was crushed at the factories that were closed. In 1994, the average grinding rate was approximately 300 tons/hour, whereas, in 2014, the average grinding rate was 592 tons/hour, an increase of 97 percent. At the same time, pol in cane improved by approximately 13 percent with the 11 factories becoming more efficient, thanks, in part, to the dedication of the faculty of the Audubon Sugar Institute (ASI). Over the last 20 years there has been a gradual increase in imbibition and preparation index at the remaining factories resulting in improved pol extraction from approximately 91.2 percent in 1994 to 94.3 percent in 2014, an increase of more than 3 percent. At the same time, pol losses in bagasse decreased from 3.00 percent in 1994 to 2.23 percent in 2004, an improvement of approximately 35 percent. Also, there have been significant improvements in loss of sugar in filter cake from 3.8 percent (pol%filter cake) in 1994 to 2.1 percent in 2014.

According to ASI associate professor Dr. Harold Birkett, factory improvements in milling have come despite of the increased throughput. Improvements in improved pol extraction have been realized due to the following changes: 1.) Improved cane preparation; 2.) Increased imbibition; 3.) Larger first mills at several factories; 4.) Improved first mill extraction; 5.) Greater attention to individual mill performance; 6.) Use of moisture plots to adjust mills; 7.) Improved welding on rolls; 8.) Universal use of Donnelly chutes; and 9.) Use of L scrapers and Lotus rolls.

Since 2001, ASI has analyzed the molasses provided weekly by each factory. The results of these analyses are used to calculate "target



purity (TP)” and a true purity for the molasses. Using TP calculated for each factory independently and assuming a grinding rate of 12,300 tons/day and a sugar recovery of 190 lb/ton and an average target purity difference (TP subtracted from the true purity) increase of +1.7 points, it is estimated that, on average, the dollar value of undetermined losses could be as high as \$500,000. With this knowledge there have been reduced losses in final molasses through lowering of final molasses purity.

Research by ASI faculty has shown that in most factories one or more mills in the tandem perform poorly, indicated by increasing moisture percent bagasse and/or low individual mill extraction. At the same time, it is interesting to note that many factories have curtailed washing cane altogether or infrequently, especially under favorable weather conditions. ASI research has shown that washing cane, especially billeted cane harvested by the cane combine, can cause a sugar loss exceeding 10 pounds per ton of cane processed. Also, improvements in factory performance have come from installation of new equipment. The ASI staff has made a concerted effort to visit each of the factories during the off season and again during the milling season to assist in design and installation of new equipment and provide consultation in their areas of expertise such as losses resulting from mill yard storage, cane preparation, milling, boiler operations, steam balance, clarification, crystallization and exhaustion of final molasses. These visits are well-received. Further, many research projects conceived by ASI faculty are funded by the industry through recurring appropriations and the Dedicated Research Funding of the American Sugar Cane League and conducted onsite at the factories.

The Audubon staff have undertaken numerous research initiatives during this report period. These include: large-scale ripener evaluations; impact of freezing temperatures on cane and processing quality; removal of suspended solids from filtrate and results from the demonstration plant located at Sterling Sugars; performance of conventional drum vs. belt filters at several Louisiana factories; starch analyses of cane juice and raw sugar at all 11 factories; use of CILAS particle size analyzer to determine crystal size; improvements of raw sugar quality with double purge of C-masseccutes; application of process modeling; and simulation in Louisiana’s sugar factories and capacity of continuous centrifugals.

In 2011, the LSU AgCenter and ASI were awarded an USDA-NIFA-AFRI-Cap Grant for \$17.2 million. ASI Professor Dr. Vadim Kochergin served as its original principal investigator (PI) until March 2013 when ASI Professor Dr. Donal Day was named PI. In 2014, LSU AgCenter Vice Chancellor Dr. John Russin assumed the duties of PI. To administer the grant, the Sustainable Bioproducts Initiative (SUBI) was created by the LSU AgCenter. SUBI involves a team of university and industry partners led by the LSU AgCenter, studying the regular production of biomass for economically viable conversion to biofuels and bioenergy using existing refinery infrastructure. Through new and existing industrial partnerships, this project uses energycane and sweet sorghum to help reinvigorate the Louisiana sugar and chemical industries. This is a multistate, multidisciplinary grant with a goal of expanding the Southern Regional Agricultural Sector by utilization of energy cane and sweet sorghum to produce butanol, gasoline, isoprene and byproduct chemicals. This multidisciplinary regional

consortium of agricultural scientists, biotechnologists, technology and engineering companies, economists and educators addresses multiple aspects associated with conversion of energycane and sweet sorghum into a portfolio of biobased fuels and chemicals. The projective objectives under the direction of ASI are as follows: 1.) To evaluate selected energycane and sweet sorghum crops and improve their production through utilization of low-input, sustainable systems to ensure an uninterrupted supply of carbohydrates and fiber to biofuel production facilities; 2.) To utilize existing pilot and industrial facilities, incorporating multiple crops and cutting-edge processing technologies to demonstrate butanol, gasoline, isoprene and specialty chemicals; 3.) To develop regionally appropriate business-marketing models and integrate biobased fuels and products into existing logistics and supply chain infrastructures based on inputs from agricultural research and techno-economic analyses; 4.) To expand 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.

Finally, ASI faculty and staff would like to express appreciation to the American Sugar Cane League and the Louisiana sugar industry for their continued moral and financial support. The outcome of the research outlined in this report should result in further improvements in efficiency as well as lower the cost of production while introducing the production of multiple agricultural feedstocks and processing to biofuels and biobased chemicals, thus allowing the Louisiana sugar industry to remain competitive in a global economy.



Giovanna M. Aita

Dr. Giovanna M. Aita is an associate professor at the Audubon Sugar Institute with adjunct appointments in the Department of Food Science and the Department of Biological and Agricultural Engineering at the LSU AgCenter. She holds a B.Sc. degree in Biological Sciences and M.Sc. degree in Food Microbiology from Clemson University and a Ph.D. degree in Food Microbiology from Louisiana State University. Dr. Aita's research focuses on the development and establishment of processes capable of demonstrating operational production of green fuels and chemicals from lignocellulosic biomass. She is a member of the Society for Industrial Microbiology and Biotechnology and the American Chemical Society.

Research Studies at the Renewable Fuels and Byproducts Laboratory

Biorefineries are processing facilities that use renewable plant materials (carbohydrates, protein, oil, lignin) as biomass. These plant materials are converted in the biorefinery into higher-value products. This opportunity can become available in Louisiana if biomass feedstocks are grown on fallow or marginal land. The USDA Roadmap on Biofuels calls for the production of 13.4 billion gallons of advanced biofuels from grassy crops with the Southern region expected to supply 50 percent of these crops. Energy cane and sweet sorghum have similar gross structure to sugarcane and are considered the most appropriate crops for this area. They could be handled by traditional sugarcane harvest and delivery systems and processed into green fuels and chemicals without interfering with the sugarcane season in a modified sugar mill operation. Progress has been made in overcoming some of the technical barriers for the conversion of biomass into higher-value products, but much still remains to be done. Some of the barriers that must be overcome on the technical side include: 1.) biomass handling, storage and distribution; 2.) biomass recalcitrance and pretreatment; 3.) cellulosic enzymes and microbial development; 4.) separation technologies; and 5.) process integration. Presented below are the research projects conducted to address some of the above mentioned technical barriers in the 2012-2014 period. (G. Aita.) ■

Use of Ionic Liquid for the Pretreatment of Energy Cane Bagasse

Lignocellulosic biomass appears to be a prospective renewable resource that can be used for the generation

of biofuels and bioproducts. The major concern in lignocellulose conversion is overcoming biomass recalcitrance through pretreatment while still maintaining a green, energy-efficient and cost-effective process. Pretreatment aims to break down the carbohydrates-lignin complex to make cellulose and hemicellulose more susceptible to enzyme degradation. Long residence times, high energy demand, harsh processing conditions, high processing costs and environment pollution remain as challenges in current biological, physical, chemical and physicochemical pretreatment methods.

Energy cane is a promising energy crop with high fiber content, cold tolerance and less fertilizer and water input requirements compared to conventional sugarcane. Ionic liquids (ILs) are promising solvents for the pretreatment of lignocellulose as they are thermally stable, environmentally friendly, recyclable and have low volatility. This study assessed the use of ionic liquid 1-ethyl-3-methylimidazolium acetate ([EMIM][OAc]) as a solvent during the pretreatment of energy cane bagasse and its effect on the chemical composition, surface morphology, cellulose crystallinity and enzymatic hydrolysis of the pretreated biomass (Figure 1).

IL-treated energy cane bagasse resulted in significant lignin removal (32.1 percent) with slight glucan and xylan losses (8.8 percent and 14.0 percent, respectively) and exhibited much higher cellulose and hemicellulose enzymatic digestibilities (87.0 percent, 64.3 percent) than untreated (5.5 percent, 2.8 percent) or water-treated (4.0 percent, 2.1 percent) energy cane bagasse, respectively (Table 1). The enhanced digestibilities of IL-treated biomass can be attributed to delignification and reduction of cellulose crystallinity as confirmed by Fourier Transform Infrared Spectroscopy (FTIR) and X-Ray Crystallography (XRD) analysis (Table 2). When pretreating energy cane bagasse with recycled IL, enzymatic digestibility decreased as the number of pretreatment cycles increased (Qiu, Z. and Aita, G., 2013). Decreasing the pretreatment temperature from 120° C to 100° C and extending the residence time from 30 minutes to 2 hours brought significant improvement to the pretreatment efficiency of recycled [EMIM][OAc] on the biomass. However, response surface methodology model indicated that a higher glucose yield of IL-treated biomass could be obtained at higher pretreatment temperatures with shorter residence times. The optimal processing conditions were pretreatment of energy cane bagasse at 131.9° C for 28.1 min at 8.4 percent solids loading resulting in a final glucose yield of 35.96 grams

of glucose per 100 grams of native biomass (Qiu et al., 2014). The results obtained demonstrated that [EMIM][OAc] can be used as a potential solvent for the pretreatment of lignocellulosic biomass such as energycane bagasse. Furthermore, the sugar yields obtained post pretreatment have great potential as building blocks in the production of renewable fuels and chemicals. (G. Aita, Z. Qiu.) ■

Figure 1. Scanning electron microscope images of (A) untreated (B) water-treated and (C) [EMIM][OAc]-treated energycane bagasse at 1000X Magnification (Qiu, et al., 2012).

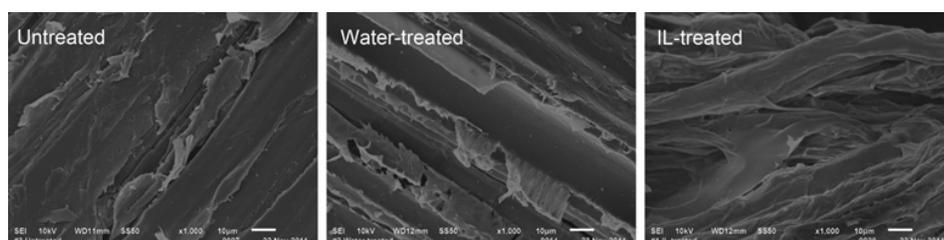


Table 1. Chemical composition of untreated, water-treated and [EMIM][OAc]-treated energycane bagasse (Qiu, et al., 2012).

Biomass component (% dry weight basis)	Untreated	Water-treated	IL-treated
Ash	1.44 ± 0.01	1.10 ± 0.01	1.39 ± 0.01
Extractives	1.49 ± 0.04	1.44 ± 0.14	1.52 ± 0.09
Acid soluble lignin	3.98 ± 0.08	4.15 ± 0.14	5.54 ± 0.36
Acid insoluble lignin	20.83 ± 0.22	21.10 ± 0.14	14.31 ± 1.06
Total lignin	24.81 ± 0.14	25.25 ± 0.01	19.85 ± 1.45
Glucan	40.87 ± 0.22	43.41 ± 0.27	43.89 ± 0.21
Xylan	20.82 ± 0.10	21.85 ± 0.18	21.10 ± 0.33
Arabinan	1.53 ± 0.05	1.59 ± 0.06	2.05 ± 0.27
Mannan	ND	ND	ND
Recovered solids	100	95.99 ± 1.67	84.89 ± 2.32

ND: none detected

Table 2. Crystallinity index of untreated, water-treated and [EMIM][OAc]-treated energycane bagasse (Qiu et al., 2012).

Sample	LOI ¹ (1426/896 cm ⁻¹)	TCI ² (1373/2917 cm ⁻¹)	CrI ³ (XRD)
Untreated	0.9593	0.4057	0.5628
Water-treated	0.8174	0.3747	0.5338
IL-treated	0.3718	0.1937	0.2452

¹LOI: lateral order index or crystallinity index based on FTIR; ²TCI: total crystallinity index based on FTIR; ³CrI: crystallinity index based on XRD.

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Studies on the Dilute Ammonia Pretreated Energycane Bagasse and Sorghum Bagasse Enzyme Hydrolysate Liquors for Syrup Production

Pretreatment methods can cause the same lignocellulosic biomass to have different compositions entering into enzymatic hydrolysis. Enzymatic hydrolysis of lignocellulose has long been studied as a way to breakdown pretreated biomass into fermentable sugars for conversion into biofuels and chemicals. Bagasse pretreatment by chemical processes yields glucose and xylose as the major monomeric sugars and arabinose, mannose and galactose as the minor ones, in addition to microbial inhibitors and other degradation products. In general, these inhibitors are classified into three major groups – organic acids, furan derivatives or furaldehydes and phenolic (mono- or polymeric) compounds. The downstream negative effects of these degradation or pretreatment byproducts (nonsugar) have been reported, thus the need to remove or reduce them to non-inhibitory levels is a prerequisite prior to converting lignocellulosic sugars into syrup.

Sweet sorghum (*Sorghum bicolor*) bagasse (SSB) and energycane (Ho 02-113) bagasse (ECB) were pretreated with dilute ammonia (DA) at 1:0.5:8 biomass, ammonia to water ratio at high temperature and pressure (160° C, 140-160 psi) (Aita et al., 2011; Salvi et al., 2010). The DA pretreated SSB and ECB were combined with various mixtures of commercially available enzymes, Spezyme® CP, Novozyme 188, Accelerase XY, and Accelerase 1500. A mix of monomeric and oligomeric sugars in the SSB and ECB enzyme hydrolysate was observed. Monomeric sugars do not only include glucose but xylose, arabinose and mannose. The oligomeric sugars (made up of two or more monomeric sugar units linked together) still have to be fully characterized. For both DA pretreated SSB and ECB, varying enzyme loadings did not affect the nature of the monomeric sugar components identified. Organic acids (i.e. formic, lactic, acetic, citric, aconitic, levulinic), furaldehyde groups (furfural and 5-Hydroxymethylfurfural) and phenolic acids were detected in the enzymatic hydrolysate. These components can hamper or cause changes in subsequent processes and thus need to be removed through detoxification. A detoxification strategy is essential prior to sugar syrup production.

Based on our initial identification and characterization of the groups present, several SSB and ECB enzymatic hydrolysate detoxification strategies were evaluated.

These strategies aimed at achieving maximum sugar recoveries. Recovery of the nonsugar components was also investigated to be processed as value-added chemicals. Detoxification strategies included ionic liquids liquid-liquid extraction (LLE), ionic-exchange resins (acidic/basic), polymeric adsorbents/flocculants, activated charcoal and calcium salts. The successful detoxification strategies included ionic liquid LLE and ion-exchange resins, as evidenced by the removal of inhibitors (Table 3). The ionic liquid LLE detoxification strategy resulted in sugar recoveries in the range of 60 percent to 87 percent. Some of the detoxification strategies were affected by the nature of the hydrolysate. Ionic liquid LLE proved to be a route that will be explored further (i.e. recyclability). The fermentable monomeric sugars obtained after detoxification can be converted into lignocellulosic sugar syrups which can be stored for future use. This lignocellulosic syrup can be converted to biofuels or introduced into processes to make chemicals. The nonsugar components can also be recovered and converted into useful chemicals. (G. Aita, S Oladi.) ■

Table 3. Summary of nonsugar components from dilute ammonia pretreated sweet sorghum bagasse and energycane bagasse enzyme hydrolysates removed by various detoxification strategies.

Detoxification strategy	Nonsugar Components*	
	Sweet sorghum	Energycane
Ionic liquid LLE	-	-
Ion-exchange resin	-	-
Activated charcoal	-	-
Molecular sieve	+	+
Calcium salts	-	-

*Nonsugar components: Organic acids, Furaldehydes and Phenolic acids; +/-: present/absent.

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Synthesis of Novel Polyesters Scaffolds from Sugarcane Industry Byproducts for Use in Skin and Bone Tissue Engineering

This research focused on the synthesis of nontoxic, biodegradable polyesters of aconitic acid, cinnamic acid and glycerol from byproducts of the sugarcane industry as scaffolds for skin and bone tissue engineering. Utilizing the byproducts, molasses and sugarcane bagasse not only adds value to the cane industry, but also paves a path for synthesizing novel biobased materials from the isolated specialty chemicals.

Aconitic acid is an unsaturated, tribasic organic acid that exists as two isomers (trans- and cis-). Trans-acetic acid is the favored form. In sugarcane, the amount of aconitic acid (free or as the Ca²⁺/Mg²⁺ salt) varies widely. Molasses contain amounts of aconitic acid ranging from 0.9 percent to 5.5 percent (on dry solids basis). Extraction of aconitic acid from molasses was studied in detail as part of this work. The yields of recovered aconitic acid varied from 25 percent to 69 percent depending on the extraction conditions (Table 4). Under all the conditions, the purity values of extracted aconitic acid were higher than 99 percent.

Table 4. Percent yield and purity of aconitic acid extracted from molasses (Kanitkar et al., 2013)

Time	Temperature					
	30°C			40°C		
	1h	3h	6h	1h	3h	6h
% Yields	34.44±1.84	43.40±0.67	49.91±0.63	43.30±0.53	62.95±1.79	68.81±0.53
% Purity	99.85±0.18	99.62±0.06	99.53±0.36	99.68±0.17	99.89±0.18	99.62±0.25

Subsequently, polyesters of aconitic acid, glycerol and cinnamic acid were synthesized (Table 5). Different compositions of polyesters were characterized to determine their mechanical properties, porosity, mass loss in stromal medium, ability to support growth and proliferation of human adipose derived mesenchymal stem cells (hASC) (Kanitkar et al., 2014; Kanitkar et al., 2015).

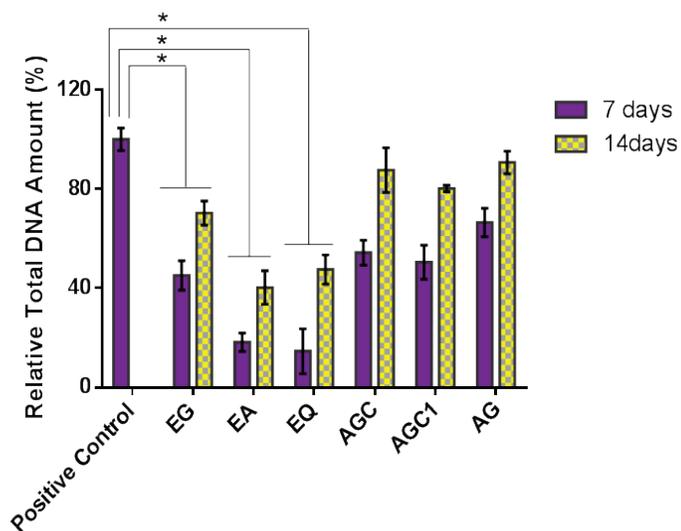
It was observed that the amount of free –OH and –COOH groups present in the polymer samples influenced their cytotoxicity and biocompatibility properties. These polyesters were tested as scaffolds for growth and viability of hASC, and it was observed that the amount of cinnamic acid present in the polyester samples affected the growth and proliferation profiles of hASC. Overall, based on the metabolic activity assays by alamarBlue® analysis and total DNA content as evident by picogreen assay, it was concluded that these polyesters hold potential as scaffolds for tissue engineering applications (Figure 2). The scaffolds were tested for their potential application in wound healing by adding bFGF to the hASC culture medium and studying the amount of collagen synthesized on each scaffold type by the cultured hASC. At the end of 14 days, the highest collagen amount was observed on hASC cultured on AG scaffolds (306 µg/ml) (Kanitkar et al., 2015). At the end of 14 days, significantly lower amount of collagen synthesized and DNA quantification was observed for AGC scaffolds as compared to PCL and AG scaffolds. Based on the collagen and total DNA amount synthesized, these polyesters hold great potential as scaffolds for tissue engineering of skin, particularly in wound healing.

Table 5. Polymer compositions tested (Kanitkar et al., 2015).

	Glycerol	Aconitic Acid (mol %)	Cinnamic Acid
Excess Glycerol (-OH)(EG)	60	30	10
Excess Acid (-COOH) (EA)	40	50	10
Equal -OH and -COOH (EQ)	40	30	30
Low Cinnamic Acid (AGC)	63	31	6
Low Cinnamic Acid (AGC ₁)	64	27	9
No Cinnamic Acid (AG)	63	37	0

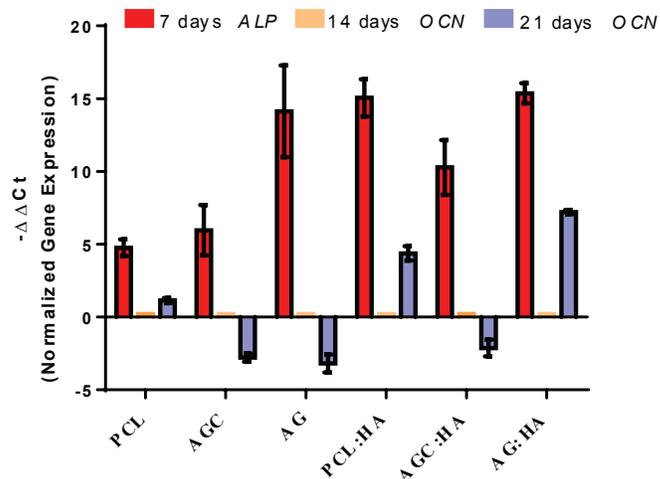


Figure 2. Relative total DNA amount for hASC cultured on different polymers for 7 and 14 days. All the results are normalized to positive control. *Indicates statistical significance between the two groups [EG: Polyester having excess glycerol (-OH groups); EA: Polyester having excess acid (-COOH groups); EQ: Polyester having equal moles of -OH and -COOH groups; AGC: Polyester of aconitic acid, glycerol and cinnamic acid; AGC1: Polyester of aconitic acid, glycerol and cinnamic acid and AG: Polyester of aconitic acid and glycerol (no cinnamic acid)] (Kanitkar et al., 2015).



Composites of AG and AGC with and without hydroxyapatite (HA) (20 percent by weight) were synthesized and characterized for their potential use in bone tissue engineering (Kanitkar et al. 2014). Polycaprolactone (PCL) scaffolds were prepared and used as control. These materials were utilized as bone scaffolds where hASC were induced to undergo osteogenesis and analyzed for mineralization and osteogenic target gene expression over a 21-day period (Figure 3). Based on calcium deposition results, alkaline phosphatase (ALP) and osteocalcin (OCN) expression data, it was concluded that these scaffolds synthesized by the monomers obtained from sugarcane industry byproducts hold promising potential as tissue engineering scaffolds for bone.

Figure 3. qPCR analysis of ALP (7-day) and OCN (14- and 21-day) expression from hASC on PCL, AGC, AG, PCL:HA (80:20), AGC:HA (80:20) and AG:HA (80:20) scaffolds. PCL: Poly (caprolactone) scaffold; AGC: Polyester of aconitic acid, glycerol and cinnamic acid; AG: Polyester of aconitic acid and glycerol; PCL:HA (80:20): Poly (caprolactone) with 20 percent (by weight) HA; AGC:HA (80:20): Polyester of aconitic acid, glycerol and cinnamic acid with 20 percent (by weight) HA; AG:HA (80:20): Polyester of aconitic acid and glycerol with 20 percent (by weight) HA (Kanitkar et al., 2014).



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Daira Aragon

Dr. Daira Aragon joined the Audubon Sugar Institute in 2012. After being a postdoctoral researcher and assistant professor-research, she has been an assistant professor on tenure track since October 2013, with an adjunct appointment in the Department of Biological and Agricultural Engineering. Her research interests involve process modeling and simulation and devising novel strategies for process optimization, control and intensification in sugar processing and biofuel production. Additionally, she conducts research on feedstock harvesting practices that would allow optimal utilization of sweet sorghum and energy cane for conversion into biofuels and other biobased products. She also participates in extension activities, primarily supporting the sugar industry in Louisiana with laboratory analysis, performance assessments and factory simulations.

Modeling Sugar Production Processes in Louisiana

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

Factory models for raw sugar manufacturing processes can be used in combination with plant data during the grinding season or in the downtime to assist plant managers and engineers to develop mechanisms to maintain and/or improve process operations. Simulation, control and optimization methodologies based on mathematical process models can be used to increase efficiency in milling, clarification, evaporation and crystallization operations. Specific applications include balancing sugar, steam and energy consumption, evaluating changes to equipment, evaluation of changes in operating conditions and finding temperatures, pressures and other operating conditions to minimize boiling time, maximize sugar recovery or obtain a desired grain size, among others.

Efforts are being made to develop factory models in the software SUGARS™ for all Louisiana sugar mills. During 2012 and 2013, complete factory models were developed for the Lula, Leighton and Louisiana Sugar Cane Coop. (LASUCA) factories. Additionally, the diffuser section of the Enterprise factory and models of the boiling house for Sterling and Westfield factories were developed. Equipment capacities and operational conditions such as temperatures, steam pressures, cane throughput, flowrates, pol and Brix values are required. (D. Aragon, V. Kochergin, S. Polanco, M. Granovsky, C. Lohrey.) ■

Double Purge of C-Masseccutes

Because of the incentives towards production of very high pol (VHP) and very low color (VLC) sugar, Louisiana factories continue looking for viable alternatives. Double purging (or affination) of C-masseccute has been tested as a low-cost process that requires minimal operational changes. In 2013 Louisiana sugarcane crop season, three factories integrated double purge of C-magma to their three-boiling crystallization schemes, corroborating color improvements on raw sugar up to 50 percent when comparing to a factory using single centrifugation. The double purge system was run controlling purity of the first magma around 82 and purity of the affined second magma between 90 and 92. The purity of the second wash molasses ranged between 63 and 67, and it was sent to A or B molasses tanks. Like with a three-boiling scheme, A and B masseccutes were seeded with footings, prepared from the affined high purity magma, returning a high polarization (~99.2°Z) and low color raw sugar (1,000 – 2,000 CU). One factory reduced color approximately 50 percent while the other two factories reduced color about 36 percent. Double-purge system and boiling-house configuration and settings differed slightly between factories. Indices of C-masseccute production, sugar lost on final molasses and sugar recovery does not distinctly show a positive or negative effect for the integration of the double purge system. (V. Kochergin, S. Polanco, D. Aragon.) ■

Filtrate Clarification

A Very Short Residence Time Clarifier (8 minutes) has been designed and implemented for filtrate clarification to avoid recirculation of the filtrate into the clarifier. Louisiana Low Turbulence (LLT) Technology has proved to be robust for different clarification applications. Preliminary data from 2012 Alma factory field trials of new filtrate clarification process were promising. The quality of the obtained clear filtrate was good. The quality of filtrate was similar to factory clarified juice with removal of suspended solids greater than 84 percent. Factory trials were fully scalable – a quarter of factory filtrate has been processed in continuous tests. A new LLT clarifier was modified to use a flash trough instead of a flash tank and was tested at Sterling Sugars during the 2013 crop season (Figure 4). The hydraulics of the design was satisfactory. The clear juice from three clarifiers had turbidities very close to the target (10 units of absorbance): New LLT Clarifier, 9.7 Units; LLT Clarifier: 10.7 Units; and SRT Clarifiers: 10.6 Units. LLT clarifiers have lower residence times compared to SRT clarifiers. These results demonstrate the potential for the LLT clarifier, specially is modified with flash troughs. (V. Kochergin, C. Gaudet, S. Grimaldo, D. Aragon.) ■

Simulation of a Biorefinery Processing Sweet Sorghum and Energy Cane

Biomass is currently the only renewable source for production of liquid substitutes of petroleum-based fuels for transportation. To make the biorefinery concept possible, regional scenarios that take advantage of specific growth areas, types of feedstock and available infrastructure must be considered. Energy cane (hybrid of commercial sugarcane, *Saccharum officinarum* and wild sugarcanes) and sweet sorghum (*Sorghum bicolor* L. Moench) are potential crops for conversion into fuels and chemicals due to their low agricultural input requirements, potentially high fiber content and processing similarities with established sugarcane crops.

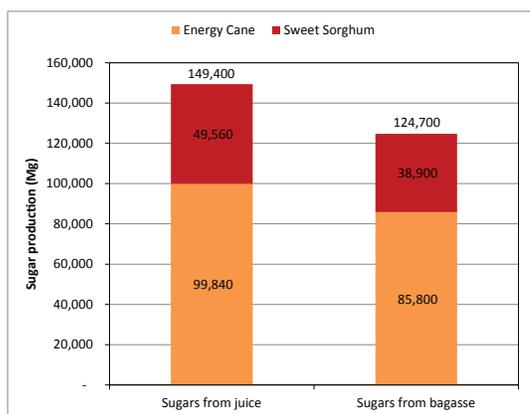
A biorefinery processing 10,000 ton of feedstock per day has been simulated in SUGARS software. Energy cane and sweet sorghum are assumed to be harvested with sugarcane equipment and processed in a raw sugar factory. Juice containing high quantities of fermentable sugars is extracted in a series of roller mills, clarified and later concentrated into storable syrup. This syrup can be used to produce fuels, chemicals and other biobased products by means of fermentation, catalytic processes or thermochemical conversion. The fiber remaining after

Figure 4. Filtrate clarifier demonstration unit installed at sugar mill.



extraction of the juice, called bagasse, is burnt in boilers to produce steam and generate power required by the sugar mill and biorefinery equipment. Additional bagasse available after meeting the facility's steam and power requirements is processed to release lignocellulosic (or second generation) sugars in an adjacent conversion plant. Excess bagasse can produce close to 45 percent of the total fermentable sugars extracted from the energy cane and sweet sorghum varieties included in this work (Figure 5). Alternatively, when excess bagasse is burnt to generate power, close to 390 GWh can be exported during the year. This study demonstrates that the integrated biorefinery approach based on energy cane and sweet sorghum is feasible in terms of fiber availability. (D. Aragon.) ■

Figure 5. Potential fermentable sugar production per year.



Evaluation of Harvesting and Storage of Energy Cane and Sweet Sorghum

Energy cane and sweet sorghum can be grown in Louisiana to supplement the feedstock supply for a year-round biorefinery, taking advantage of the harvesting and processing infrastructure already in place for sugarcane. Studies that include the issues of equipment efficiency; residue management and utilization; transportation costs; and feedstock storage are necessary for the successful integration of these crops into the biorefinery scheme based on cane-type crops. Harvesting tests were performed between August and November 2013 at St. Gabriel, Louisiana, to determine harvesting efficiency and fermentable sugar losses during storage when energy cane and sweet sorghum are harvested with a regular sugarcane combine harvester (Figure 6). Yields, harvesting efficiency and potential sugar losses were measured on the field for extractor’s fan speed settings of 0 rpm, 800 rpm and 1,100 rpm and billet sizes of 6 and 8 inches. (D. Aragon, A. Amaya, I. Tishechkina). ■

Figure 6. Combine harvester.



Harold Birkett

Dr. Harold Birkett has more than 40 years of sugar, ethanol and sugar byproduct experience, all of which represent work with Schaffer & Associates and its predecessor companies. He is a registered professional engineer in Louisiana and was the chief process design engineer and process specialist for Schaffer & Associates for more than 30 years. He holds a B.S., M.S. and Ph.D. in Chemical Engineering. His dissertation was “The Determination of Sugar Cane Quality in Louisiana by the Core Press Method.” He developed computerized material and energy programs for the sugar and process industries that facilitate the design of plants and the analysis of factory operation and laboratory reports. Dr. Birkett works part time as an Associate Professor of Sugar Technology at Audubon Sugar Institute, LSU AgCenter, conducting research and extension in all facets of sugar processing. He is formerly the process manager in sugar factories and ethanol facilities in Guyana and the U.S. He is a member and past president of the American Society of Sugarcane Technologists and the author of numerous publications and presentations.

Bagasse Boiler Performance Summary, 2011

Assistance was given to five factories with boiler compliance testing (DEQ) on seven boilers. In addition, 30 other boiler tests were conducted. The same methods were used as in previous years. Operating conditions such as steam pressure, steam flow and boiler feedwater temperature were noted. Flue gases were analyzed for temperature and oxygen concentration using a Testo 300 XL flue gas analyzer. Preheated air temperature was determined using an Omega handheld digital thermometer and thermocouple probe. Bagasse samples were collected during each test and analyzed for moisture and ash content. Excess air, boiler efficiency and pounds steam produced per pound of bagasse burned were calculated using a program written for a similar study conducted in the early 1990s.

A brief summary of 2011 operating data and results are presented in Table 6 along with boiler operating data from 2005-10 (328 tests) and the early 1990s (320 tests). Preheated air temperature decreased in 2011. Flue gas temperature decreased slightly from that of 2005-10 but was higher than in 1990-92. Moisture percent bagasse shows a slight decrease over time while efficiency is increasing along with pounds steam produced per pound bagasse burned. Many of the tests conducted in 2011 were part of official boiler compliance testing.

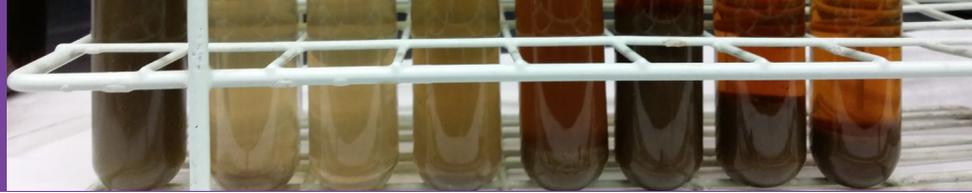


Table 6. Comparison of boiler performance tests in 2011 with that of previous years.

	1990-92	2005-10	2011
No. of Runs	320	328	51
Steam, lb/hr	69,318	107,155	115,258
Preheated air temp, °F	433	465	442
Flue gas temp, °F	418	443	428
Oxygen, %	9.8	8.3	7.9
Bagasse moisture, %	54.48	53.14	53.83
Bagasse ash, %	2.91	4.42	3.37
Excess air, %	107.67	75.38	65.54
Efficiency, %	55.14	56.78	58.47
Lbs steam/lb bagasse	1.98	2.00	2.03
Bagasse % cane	36.83	32.22	31.52

Boiler efficiencies have been improving over time and currently average about 58 percent with an average flue gas temperature of 428° F. A large increase in boiler efficiency can be achieved by lowering the flue gas temperature to 300° F. The only practical way to achieve this is with an economizer (boiler feed water heater using flue gas). This would increase boiler efficiency to about 65.4 percent and produce 9 percent more steam from the same quantity of bagasse.

Use of a gas analyzer to measure oxygen and carbon monoxide levels, along with a temperature probe, would be useful in determining not only optimum excess air levels but also boiler efficiency. Adjustments to boiler control settings and operations could then be optimized. A proper and steady bagasse feed is also necessary for good boiler operations.

We would like to express appreciation to the American Sugar Cane League and all Louisiana sugar factories for their support of this project. (H. Birkett and J. Stein) ■



Update on Entrainment Losses

Several Louisiana factories experience high levels of entrainment, which results in the loss of recoverable sugar. Results from a preliminary study in 2009 to investigate these losses were enough to warrant continued investigation in 2010 and 2011. Condensate and/or condenser water samples were collected at every Louisiana factory and analyzed for trace sugar using the phenol-sulfuric acid method (Dubois et al 1956; SASTA, 1985).

Table 7 shows the average, high and low sugar concentrations in the condensate leaving the individual evaporator bodies. For evaporators with no leaking tubes, the first effect condensate should be sugar-free because this is condensed exhaust while the condensates from the subsequent bodies generally contain sugar resulting from entrainment. The second, third and fourth body condensates contained an average of 21, 40 and 109 ppm of sugar respectively. The increasing level of entrainment down the evaporator set is expected as the vapor velocity increases at the progressively lower pressures in the effects while the sugar concentration of the syrup is also increasing.

The quantity of evaporator condensate produced is typically in the range of 1,300 to 1,700 lb/ton cane. The average sugar concentration in the evaporator condensates of 53 ppm represents a loss of about 0.08 lb sugar/ton cane.

Evaporator Body Condensates, ppm sugar					
Factory	First	Second	Third	Fourth	Average
Average	6.74	20.57	58.13	108.42	52.96
High	29.83	139.75	677.92	1653.79	641.43
Low	0.21	1.76	0.92	2.12	2.53

Table 8 shows the average, high and low sugar losses in once-through, closed (with intermittent blowdown) and recirculation systems with continuous blowdown. For the once-through systems the sugar concentration in the condenser water varied from 1 to 1,547 ppm. Assuming 10,000 lb water/ton cane for the once through injection water system, the sugar loss varied from 0.01 to 15.47 lb sugar/ton cane and averaged 1.71 lb sugar/ton cane. This indicates there is considerable scope for reducing sugar losses in the condenser water.

For the closed systems (cooling tower or spray pond) or recirculating systems with continuous blowdown it is not possible to calculate the sugar losses without knowing the quantity of blowdown. The sugar concentration in the

water in the cooling tower and spray pond systems varied from 13 to 7,121 ppm and averaged 2,513 ppm. Assuming that the blowdown from the closed systems is about 2 percent, the average sugar losses at these factories would be 0.86 lb/ton.

For recirculation systems with continuous blowdown the sugar losses varied from 3 to 1,533 ppm and averaged 1.11 lb sugar/ton cane.

	Once-Through Systems	Closed (Assuming 2% Blowdown)	Recirculating With Continuous Blowdown
Average	1.71	0.86	1.11
High	15.47	2.42	3.29
Low	0.01	0.01	0.01

A routine, and preferably continuous, monitoring system for measuring entrainment losses is highly recommended. Entrainment losses from evaporator, vacuum pan and filter condensers are all subject to periodic high levels of entrainment. High levels of sugar in the condenser water at several factories indicate that improved entrainment separators would be highly cost effective.

Sugar losses in condensates are generally insignificant (average 0.08 lb/ton cane). Sugar losses in condenser water are significant, averaging 0.9-1.7 lb/ton cane, but can be as high as 15.5 lb/ton cane.

We would like to thank all of the factories and personnel who participated in the study and for so kindly assisting us with sample collection. (H. Birkett and J. Stein) ■

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Capacity of Continuous Centrifugals

An important factor to consider in the purchase of a continuous centrifugal is its capacity. Two Louisiana factories considering the purchase of Western States continuous centrifugals were interested in determining the capacity of side feed versus center feed machines. The areas investigated included the capacity (cubic feet/day), apparent purity rise and water usage. The centrifugals tested were the Western States Titan CC-1300i and CC-5.

The centrifugal capacity calculation method required the Brix, pol and purity of the massecuite, sugar and molasses along with the molasses flow rate. Table 9 shows the summary of the B strike tests while Table 10 shows that of the C strike tests.

Table 9. Summary of centrifugal capacity processing "B" strikes.

	Titan 1300			CC-5
Feed location	Side			Center
	High	Low	Average	
Massecuite Brix	92.80	90.20	91.50	90.20
Massecuite purity	74.22	71.63	72.93	71.63
Apparent purity rise across machine	2.43	-0.20	0.84	0.35
Capacity, cu.ft./day	17,759	15,270	16,152	8,161

Table 10. Summary of centrifugal capacity processing "C" strikes.

	Titan 1300			Titan 1300			CC-5
Feed location	Side			Center			Center
	High	Low	Average	High	Low	Average	
Massecuite Brix	95.80	94.40	95.00	95.0	94.6	94.97	94.60
Massecuite purity	61.03	54.82	56.81	60.76	56.62	58.36	55.14
Apparent purity rise across machine	6.00	1.48	3.31	3.18	2.01	2.53	5.82
Capacity, cu.ft./day	11,195	3,675	7,440	10,068	4,862	7,518	2,171

Capacity-wise, there was no appreciable difference between side feed and center feed Titan 1300 centrifugals. The Titan centrifugal capacity on B strikes was about twice the capacity of the CC-5 and twice that of the Titan machines processing C strikes. The Titan 1300 when processing C strikes had more three times the capacity of the CC-5. It is important to note that centrifugal capacity highly depends on the massecuite being processed. (H. Birkett and J. Stein) ■

Filter Station Operations

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 were to review filter operations in general and compare belt filters with that of the traditional rotary drum filters.

In an effort to compare belt filters with rotary drum filters several areas of interest were investigated over the 2012 and 2013 crops. These included pol losses, bagacillo ratio (bagacillo percent feed/mud solids percent feed), filter retention (mud in filter cake/mud in feed), and filter capacity (filter cake production and removal of mud). Results obtained are provided in Table 11.

Table 11. Summary of filter results.								
	Drums				Belts			
	High	Low	Average	No. of Tests	High	Low	Average	No. of Tests
Sugar loss, lb. pol/ ton cane	17.29	0.88	5.21	19	6.28	0.22	2.64	11
Pol % filter cake	4.08	1.00	4.08	20	5.86	0.46	2.36	11
Filter retention, %	97.88	60.75	89.30	18	98.38	38.89	87.00	11
Bagacillo/ton cane	12.60	1.80	5.32	18	6.20	1.80	3.89	11
Bagacillo ratio	26.40	7.73	16.48	17	23.33	9.41	15.77	11
Filter cake production, lb./hr./sq.ft.	103.68	2.59	34.84	59	85.98	24.75	52.35	14
Mud removal, lb./hr./sq.ft.	20.93	0.45	7.61	59	21.49	7.98	14.48	14

In general, filter operations are highly variable with much scope for improvement. The average filter retention of 88 percent is generally very good. Most of the bagacillo is obtained from the underflow with very little coming from bagasse screening. The bagacillo ratio (bagacillo/mud) of 16 percent is very low. Filter cake production by belt filters averaged about 50 percent more than that of drum filters. Mud removal by belt filters was approximately twice that of drum filters. Belts typically require 100 gpm of belt wash water per filter. The belt wash water, which contains some sugar, is currently ditched. Ideally, a mechanism for utilizing the belt wash water in the process would be advantageous.

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. (H. Bikett and J. Stein) ■



Donal F. Day

Dr. Donal Day is the A. Wilbert and Sons Endowed Professor for Biofuels. He has spent 42 years in process research, 35 of them at the Audubon Sugar Institute, specializing in applying microbial physiology and industrial microbiology to research and development (laboratory-, bench- and pilot-scale) for sugar production, diagnostic and pharmaceutical industries. Dr. Day has published more than 200 technical publications and has 18 patents. He has expertise in biofuels, fermentation, new product development and industrial application, carbohydrates, polysaccharides, enzymes and cell culture. Recently he has been working on an Institute program on conversion of sweet sorghum and energy cane to ethanol and butanol with the goal of expanding opportunities for the sugar processors of Louisiana.

USDA-Funded AFRI-CAP “Multiple Feedstock Program”

The rationale for this project is to drive the expansion of biofuel crops throughout the Southeast United States by development of two crops suitable for this region. This is a region of great agricultural productivity but much in desperate need of a new economic base. By the same token, the biofuel industry has been limited by a shortage of readily convertible feedstocks (other than corn). This program targets both these problems. The Southern Regional Agricultural Sector can be expanded through production of sweet sorghum and energycane, for the manufacturing of biobased fuels and byproducts. These crops are capable of being produced in this region (Figures 7 and 8). However, significant knowledge gap exists regarding the economic feasibility and sustainability of growing and

processing these crops. A regional multidisciplinary consortium headed by ASI, of agricultural scientists, biotechnologists, technology and engineering providers, economists and educators, with the help of biofuel companies trying to secure sufficient carbohydrate and fiber feedstock to meet production goals, was formed to facilitate production and conversion of these crops. This program includes: improving agronomic production of energy crops through breeding for selected parameters; screening using NIR; and harvesting trials for energycane and sweet sorghum using modified sugarcane harvesters. The program utilizes a pilot facility to process selected feedstocks into fermentable sugar syrups for industrial partners to test production scenarios. It supplies input for economic models addressing agricultural and processing and cost structures for feedstock-bioproducts possibilities. It initiates training programs for future biofuels workers and extending information to extension units in multistate areas on the potential for agricultural development using these target crops.

The working scenario for the project is to produce shippable, fermentable sugar syrups at locations near production fields. The syrups can be then sent to centralized conversion facilities for production of fuels and byproducts. It is also predicated on having feedstock(s) available year round, though use of crops with staggered harvest times. A brief summary of project accomplishments through 2013 follows:

Feedstock development has the objectives of optimizing yields, expanding diversity and range of cultivation and reducing inputs through breeding for both sweet sorghum and energycane. Cross-pollination was achieved between cultivated sugarcane and two wild related species as well as miscanthus. These crosses are now being evaluated for biomass yield and several quality traits in diverse locations, primarily outside of the sugarcane belt, across the state of Louisiana. This work is being conducted at the LSU AgCenter Sugar Research Station. For energycane, the enhancement of cold tolerance and optimization of its production potential under temperate climate regimes were studied at the USDA Station (Houma). Energycane plots were established at the Macon Ridge Station in North Louisiana.

Figure 7. Energycane.



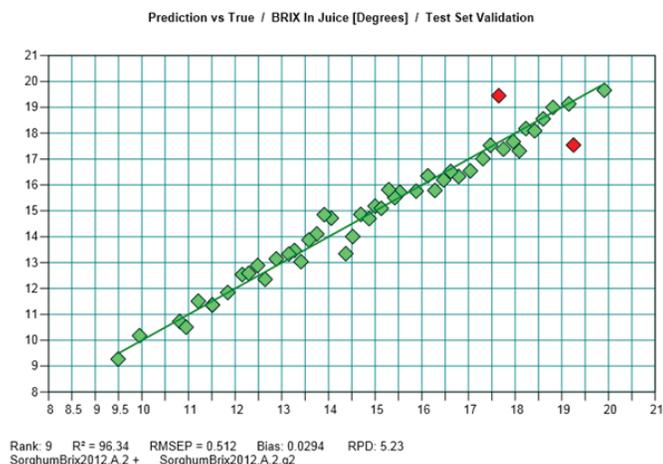
Figure 8. Sweet sorghum plot – St. Gabriel.



For sweet sorghum, the evaluation of geographic zone of adaptation, inclusive of its ability to maintain juice quality into the fall season, production of commercial yields on marginal soil and response to low-input sustainable production practices are addressed. Baseline soil nutrient information gained from pretreatment sampling is used to identify any pretreatment biases and serve as a basis for observing changes in soil fertility and carbon resulting from establishment and management of these cropping systems. Sweet sorghum plots were successfully established at four experimental sites in the state. Varieties were supplied by CERES Corp.

As part of both the breeding program and the processing program, models were constructed and are being validated for using Near Infrared Spectroscopy (NIR) as a rapid analytical tool for each of the experimental crops (Figure 9). Parameters include the

Figure 9. Sample of NIR Calibration-Sweet Sorghum Brix.



range of standard factors (Brix, sugars, etc.) and the lignocellulosic components, cellulose, hemicellulose and lignin.

Feedstock Logistics and Preprocessing is involved with the assessment of harvesting biomass, transportation to the plant, storage, treatment by milling or diffusion and bagasse drying for future usage. Harvesting trials were conducted in collaboration with John Deere. Energycane and sweet sorghum crops were planted at the St. Gabriel Station in sufficient quantities to allow milling and storage trials to be conducted in 2013. These trials included effect of various blower speeds on yields. Storage trials allow evaluation of losses of simple sugars during harvesting, transportation and preprocessing storage.

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

A model has been developed on SUGARS™ software, based on a sugar mill model, which allows calculation of heat and material balance for the primary processing (syrup) 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. Our model assumes a primary processing plant operating on biomass harvested from 50,000 acres at 10,000 tons wet biomass per day. The input parameters to calculate energy and mass balances will be defined by the crop's composition. The goal is to determine the amount of biomass that can be made available for lignocellulosic conversion from each crop while fueling the primary processing plant with biomass.

The pilot plant was installed that replicates part of the proposed primary processing plan (Figure 10). The first segment provides shredding and milling of feedstock to extract simple sugars. The second part is required for purification and evaporation of the simple sugars into syrups. The plant was used in 2013 to prepare test syrups for our industrial supporters and to investigate processing problems from the different experimental crops. This plant will also be used as part of the Chemical Engineering laboratory course at LSU.

Research Reports

Figure 10. Audubon pilot plant.



Conversion of the biomass to sugars and the conversion of sugars to second generation biofuels is an underlying need of this program. Second generation fuels are drop-in, non-ethanol fuels produced from lignocellulose rather than “food” crops. The fuel focus is on production from sugars of jet fuels (already demonstrated by a commercial partner) and butanol. Conversion of lignocellulose to sugars starts with a chemical deconvolution of the plant material to make accessible the carbohydrate components (cellulose and hemicellulose). Ammonia treatment has been settled on as one of the more promising ways of deconvoluting biomass. Ammonia treated sugarcane bagasse material was hydrolyzed using various combinations of cellulases and xylanase, which resulted in at least 60 percent glucose yield and 75 percent xylose yield with up to 70 percent ammonia recovery. This method has been applied to sweet sorghum and is being tested on energycane.

The substitution of a higher BTU, drop-in fuel (butanol) for ethanol will result in increased gas mileage in vehicles and easier distribution of renewable fuels (via pipeline) throughout the United States. An immobilized cell column using a strain of *C. beijerinckii* supplied by Optinol™ was constructed and has operated on a synthetic media with a variety of glucose containing carbon sources (Figure 11). The concentration of butanol was low, as is common with this fermentation. Research is proceeding extraction methods for the butanol as well as on use of recycle streams to enhance the viability of the butanol fermentation system.

Through sensitivity analysis using our LCI model of energycane and through analysis of extant life cycle inventories of biocommodity fuels and chemicals, we have found that the environmental impacts of feedstock

Figure 11. Continuous butanol fermentation using immobilized cell production columns.



production are likely to dominate the overall life cycle effects of any economically successful biocommodity product. This conclusion allows developers of biocommodity products and policy to recognize the importance of developing and incentivizing feedstock production systems with minimal environmental impact.

The Economics team has evaluated the costs of producing energycane and sweet sorghum as a biofuel feedstock. The Education task force 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. The Extension task force 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 publication of surveys, guides and articles in trade journals.



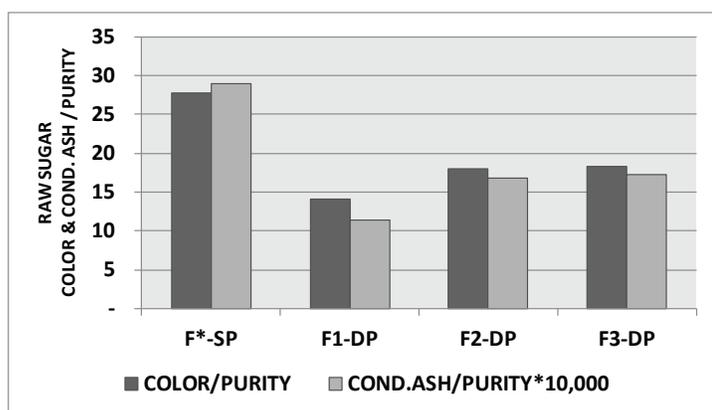
Table 12. Double-purge system average purities and raw sugar purity and color achieved during the 2013 Louisiana sugarcane crop season for each factory.

Material	Parameter	Factory-1	Factory-2	Factory-3
1st Magma	Purity	81.3	81.4	81.2
2nd Magma	Purity	92.3	90.0	90.5
2nd Molasses	Purity	63.1	65.7	64.9
Raw Sugar	Purity	99.3	99.4	99.5
	Whole Color*	1,164	1,536	1,675
	Affined Color*	709	666	

*Color @ 8.5pH & 1.2 μ m

The double-purge system proved that can be used to improve raw sugar quality without affecting sugar recovery at the boiling house. It was noticed that the goal polarization (99.2 °Z) was easily reached (Table 12). At the same polarization the whole color and the conductivity ash content were approximately 30 percent to 50 percent lower after the integration of double purge of C-magma (Figure 14).

Figure 14. Raw sugar whole color and conductivity ash – purity ratio for the three factories with double-purge (DP) of C-magma compared to a traditional factory (F*) with single purge (SP).



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Franz Ehrenhauser

Franz Stefan Ehrenhauser, an Austrian native, joined Audubon Sugar Institute in April 2013. He holds a Diplom Ingenieur degree (2009, MS equivalent) from the Johannes Kepler University Linz, Austria in Wirtschaftsingenieurwesen Technische Chemie (technical chemistry/economics) and a Ph.D. in Chemical Engineering from Louisiana State University, Baton Rouge (2011). He has collected previously experience in the sugar industry as a research intern at Danisco Sugar and sweeteners in Nakskov, Denmark, addressing sucrose crystallization rates. His current research interests center around purification and separation technologies for the sugar and biobased chemical industries, i.e. sugar crystallization, advanced separation technologies such as membranes, electrodialysis and chromatography, and improved clarification operation.

Size Matters – 2013/14 Season Crystal Size Analysis for Louisiana Sugar Mills

During the 2013/14 season, more than 300 different samples were obtained from Louisiana sugar mills and analyzed. The Cilas 1800 particle size analyzer provides the ability to assess the entire size spectrum of crystals

ranging from 0.04 μm to 2,500 μm . Typical samples analyzed were seed slurry, A-, B-, and C- sugars and massecuites. The following presents a short summary giving an overview of some aspects of the obtained samples.

Seed Slurry Analysis

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

Figure 15 shows the particle size histograms of several sampled slurries during the season. Figure 15a and 15b show powdered sugar slurries. Interestingly enough, not every powdered sugar is identical. Powdered sugar is prepared by dry-milling granular sugar in a (hammer) mill and adding up to 5 percent (m/m) starch as anticaking agent. The label 10X denotes no specific size but only the times the sugar is sent through the mill. Usually, there exists an upper specification on the particle size, but there is no control limit on the small, finer particles.

Figure 15. Crystal size distributions of (a) Domino Sugar powdered (10X) sugar sold in 1-lb. box, (b) Domino Sugar powdered (10X) sugar sold in a 2-lb. bag, (c) ball-milled granulated sugar and (d) ball-milled powdered sugar.

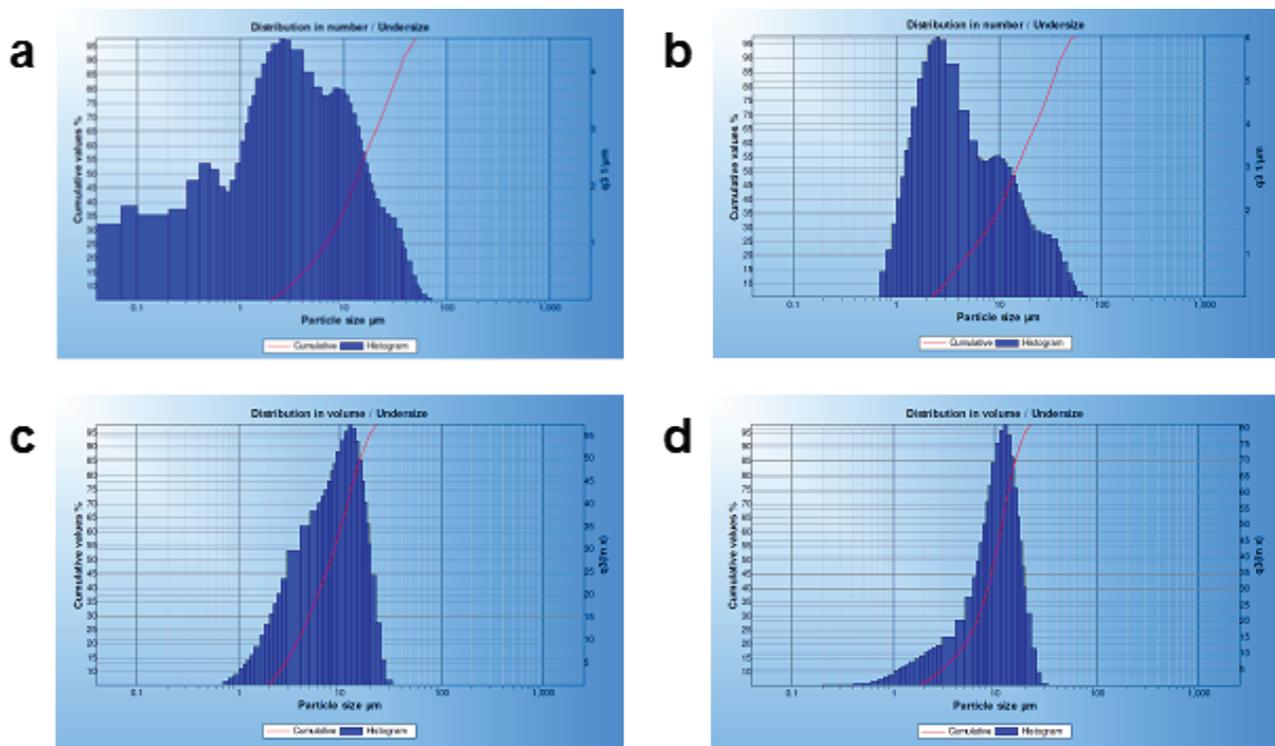


Figure 15a shows the slurry obtained from Domino’s powdered 10X sugar as it is sold in a 1 lb. box, whereas Figure 15b shows also Domino’s powdered sugar sold in a 2-lb. bag. Clearly, the distribution is quite different. The boxed sugar has a substantial fraction of very small crystals, whereas the bagged sugar exhibits a more narrow size range, without the ultrafine crystals. Other powdered sugars (Western Sugar and Milliana) were also measured, and their particle sizes exhibited different characteristics. From these samples we can conclude that the selection of the powdered sugar is relevant. The sustainability of the particle size distribution during the season is especially challenging, and, as such, the powdered sugar for the entire season should be bought from the same production lot. Storage of powdered sugar is also the key, as Louisiana’s climate causes caking and the sugar should therefore be stored in a dry place (e.g. closed drum).

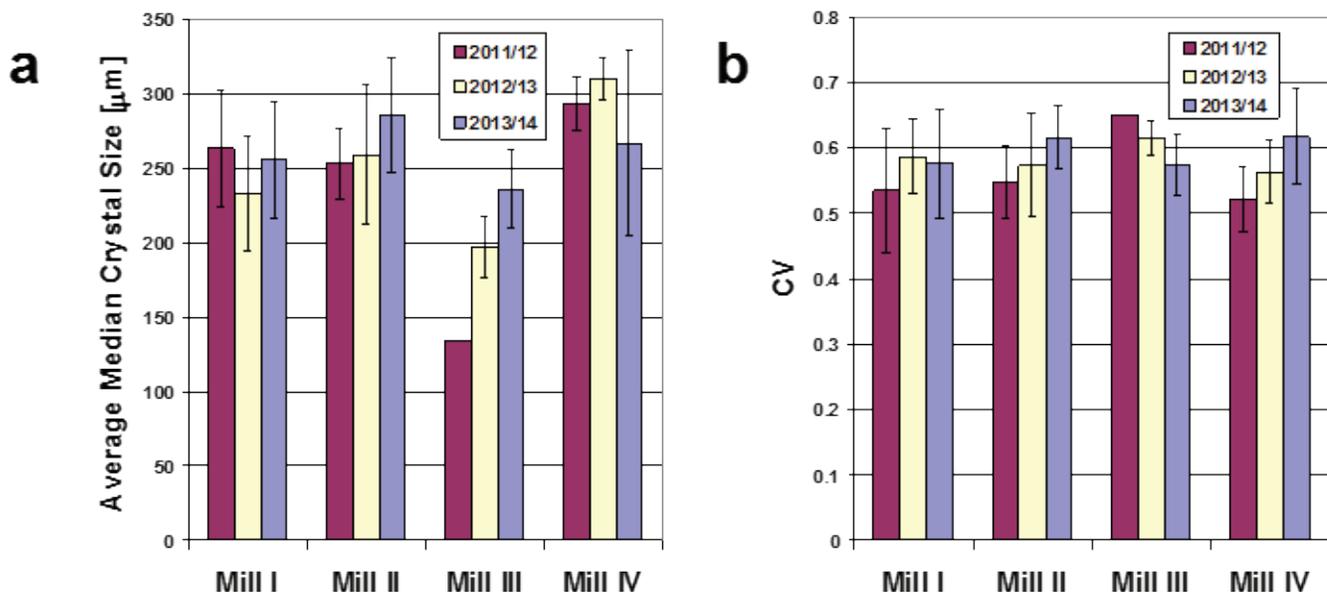
Figure 15c and Figure 15d show ball-milled seed slurries. Figure 15c shows a seed slurry which has been produced from granular sugar. The particle size is comparable to the one of the ball-milled powdered sugar (Figure 15d). The choice of the seed slurry and preparation method is challenging and will vary from mill to mill. A narrow (small CV) particle size distribution is the key feature of any good slurry. The desired particle size range depends on the configuration of the pan floor and the graining procedure. As such for some mills, ball-milled fine slurry material is ideal, whereas others prefer

powdered sugar. The most important feature for any slurry preparation is reproducibility throughout the season to provide consistency.

C-Sugar Analysis

Four mills have regularly sent samples (from 1 up to 49 samples per mill and season) of C-sugar from different C-sugar centrifugals for analysis during the last three seasons. Figure 16 shows an overview of the measured samples. Figure 16a shows the average median of the obtained C-sugars, which centers around 250 μm . The error bars in the graph denote the standard deviation of the median of the measured sugars and are a measure for the reproducibility during the season. Mill III has the least deviation in the particle size of their C-sugar for the 2013/14 season. CVs for the C sugar can be found in Figure 16b. The values range between 0.5 and 0.65. A more narrow distribution and therefore lower CV might be desirable, but difficult to achieve. Generally, it is difficult to establish common trends for the shown values. Particle size rises for mills II and III, whereas for mill I and IV no trend can be determined. CV values are rising for mills II and IV. For mill I no clear trend in CV is recognizable and for mill III a decrease can be seen. Generally, larger median crystal sizes and lower CVs are desirable since this should improve recovery and purgability at the centrifugals.

Figure 16. (a) Average median crystal size, and (b) CV of C-sugar for mills I-IV during the 2011/14 seasons.

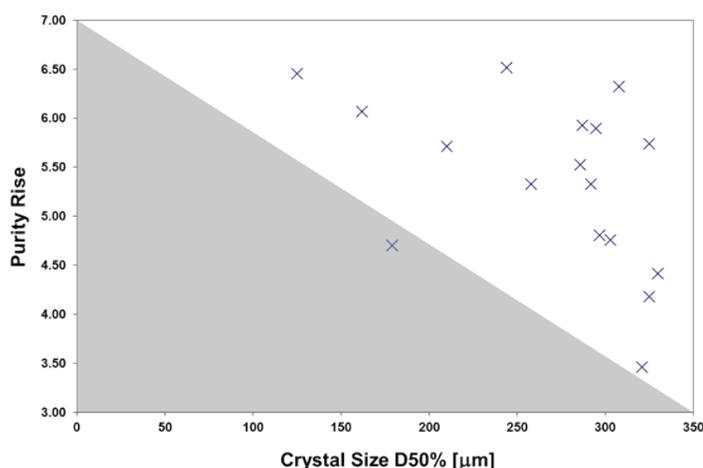


Centrifugal Performance

A unique evaluation of the centrifugal performance was possible at one mill by evaluating nutsch purity and comparing with actual (centrifugal) molasses purity. As the nutsch filter is more effective in capturing the crystals of the massecuite, while the centrifugal recovery is subject to losses. The purity rise, i.e. the difference between the purity obtained by nutsch filtration and the actual purity of the centrifugal molasses, is a measure of the experienced sugar losses to molasses.

Figure 17 shows the purity rise plotted versus the median crystal size (D50%). There is no clear trend between the purity difference and the particle size recognizable. Even though a trend is not visible, the absence of small purity differences for small particle size distributions (grey shaded area) indicates that by increasing crystal size the potential to minimize losses is given.

Figure 17. Purity rise in dependence of the median (D50%) crystal size.



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

The measurement of the crystal size distribution enables the assessment of the crystallization process, the centrifugal operation and seed slurry preparation. Seed slurry should be prepared in a consistent manner in order to provide a stable basis for further optimizations. C-sugar crystal size distributions in the four measured mills show

no common trend; however, best potential centrifugal recovery can be achieved by aiming at larger crystal sizes and uniform (low CV) crystal size distribution. (F. Ehrenhauser, I. Tishechkina, and D. Aragon).

The authors would like to thank the American Sugar Cane League and the Louisiana sugar mills for their continuing support and contributions for this research. (F. Ehrenhauser, I Tishechkina, D. Aragon) ■

Benjamin Legendre

Dr. Benjamin Legendre is the Denver T. Loupe/ ASSCT Sugar Heritage Professor and has 46 years of research and Extension experience in state and federal organizations in the genetic improvement of sugarcane, selection of improved sugarcane cultivars, including “energycane” and cane and juice quality. He is recognized for his work on plant growth regulators as chemical ripeners. He is recognized as an international authority on sugarcane breeding and sugarcane quality, specifically on factors that affect postharvest deterioration. He has visited and/or accepted invitations to consult in 20 foreign countries on variety improvement, germplasm enhancement, cane and juice quality, cane deterioration and the use of chemical ripeners.

He spent 31 years at the USDA-ARS, Sugarcane Research Unit, Southern Regional Research Center, Houma, Louisiana and served as research leader for 11 years prior to accepting a position with the LSU AgCenter as Professor and Sugarcane Specialist with the Louisiana State University Cooperative Extension Service. In 2008, he was appointed Professor and Head, Audubon Sugar Institute of the Louisiana State University Agricultural Center, St. Gabriel, Louisiana.

Dr. Legendre has authored or co-authored 243 publications and made over 250 presentations before local, national and international audiences. He is a member of honorary societies such as Alpha Zeta, Gamma Sigma Delta and Phi Kappa Phi and is an honorary life member of both the American and the International Society of Sugar Cane Technologists. He has also received numerous awards in recognition of his invaluable contributions to the sugar industry.

Managing Damaging Freeze Events in Louisiana Sugarcane

Exposure of sugarcane to damaging frosts occurs in approximately 25 percent of the sugarcane producing countries of the world, but is most frequent on the mainland of the United States, especially in Louisiana. The frequent winter freezes that occur in the sugarcane



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

In the southern area there was minimal damage to the stalk with only the apical meristem or terminal bud affected. However, in the more northern areas, the freeze events affected the entire stalk. With approximately 40 percent of the crop still remaining in the field, and to study the reaction of commercial and candidate varieties to these early freeze events, the Outfield Variety Test at Alma Plantation (located in Lakeland in the northern area above I-10 and U.S. Highway 190) was chosen for the study. The test included the commercial varieties, HoCP 96-540, L 01-283, L 01-299, L 03-371 and HoCP 04-838 and the candidate varieties, Ho 07-613, L 09-112, HoCP 09-804 and Ho 09-840. Ten-stalk samples were hand-cut at ground level but not stripped or topped from each of three replications for the first of three sampling dates, Dec. 12, 19 and 26, 2013. Another set of samples was cut on Jan. 3, 2014, but this time the samples were hand stripped and tops were removed approximately 30 cm. (12 in.) below the apical meristem (terminal bud). All samples were immediately transported, weighed and processed at the Sugar Research Station at St. Gabriel using the press method of analyses. Juice samples were analyzed for Brix by refractometer and sucrose by polarimetry and bagasse (residue) samples were analyzed for moisture (by drying). The Brix, sucrose, purity and fiber content of the cane were then calculated from these analyses after which the

estimated yield of theoretical recoverable sugar per ton of cane (TRS/TC) was calculated. Juice samples were also analyzed for pH, titratable acidity, total polysaccharides and mannitol. Further, results were compared to actual factory data for daily core juice pH, crusher juice polysaccharides, syrup purities, C massecuite viscosities and sugar yield from Alma Plantation (Lakeland) and syrup purities and sugar yield from the Leighton factory, located in Thibodaux, in the southern area below I-10.

Immediately following the field assessment, the LSU AgCenter issued best management practices (BMPs) to be used in reducing the impact of the freeze events on sugar yield. Those BMPs stressed the need to deliver high quality cane to the factories free of mud, deteriorated tops and leaves and other trash. The BMPs indicated that growers and processors should not panic because the industry had experienced freeze events of this magnitude many times before. Since areas of higher elevation tend to be warmer, the BMPs recommended that growers should harvest fields with lower elevation first. Also, growers were informed that varieties with poor stalk cold tolerance, i.e., L99-226, L99-233 and L03-371, should be harvested first (Figure 18). Other items discussed in the BMPs included standing vs. down cane, topping height and whether to burn. It also warned of overnight sleeper loads that could lead to increased deterioration.

Figure 18. Stalk cold tolerance of Louisiana sugarcane varieties following a freeze event.

Resistant	Intermediate	Susceptible
HoCP 04-838* (9)	LCP 85-384 (29)	L 99-226 (59)
	HoCP 96-540 (24)	L 99-233 (70)
	L 97-128 (23)	L 03-371 (61)
	HoCP 00-950 (50)	TucCP 77-42** (64)
	L 01-283 (34)	
	L 01-299 (39)	

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

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

Data from the Outfield Test at Alma indicated that most of the parameters measured for the samples with tops and leaves, i.e., pH, titratable acidity, total polysaccharides, TRS/TC, remained relatively stable (unchanged) over the sampling period although it became increasingly impossible to clarify juice samples in the lab with aluminum chloride on the Dec. 26 sampling date. The

Alma factory data, however, showed that the core lab juice pH, syrup purity and sugar yield started a slow decline over the same period (Figure 19). On the other hand, total polysaccharides in the crusher juice and C-masseccuite viscosity at Alma showed significant declines after the freeze events with the BMPs in place. For the Leighton factory operating south of I-10, syrup purities and sugar yield actually continued to rise in spite of the freeze events and a wet harvest (Figure 20). In general, ambient temperatures following the freeze events were cooler than normal although there was one record daily high temperature of 29° C (84° F) on Dec. 5. With the BMPs in place, the Alma factory experienced no difficulties in the boiling house without any indication of c-axis elongation of sugar crystals even with the last strike of the 2013-2014, which was processed on Jan. 6 (Figure 21).

Figure 19. Syrup purity for Alma Plantation during the 2013-2014 crop harvest before and after the freeze events of Nov. 28-30, 2013.

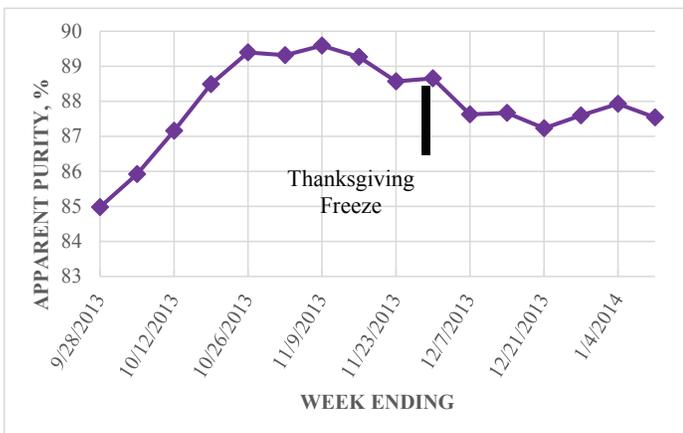


Figure 20. Syrup purity for Leighton Factory during the 2013-14 crop harvest before and after the freeze events of Nov. 20-30, 2013.

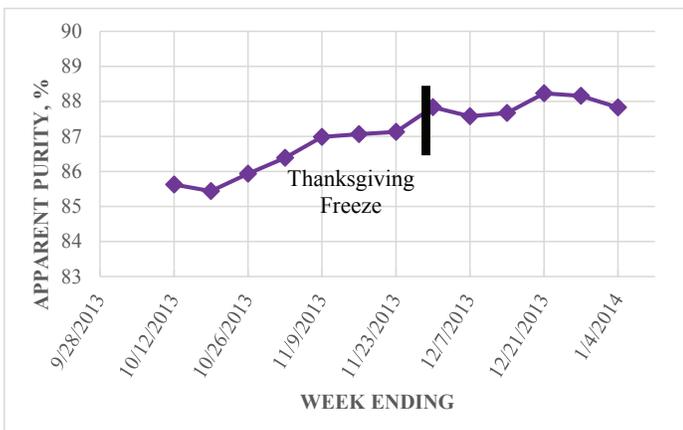
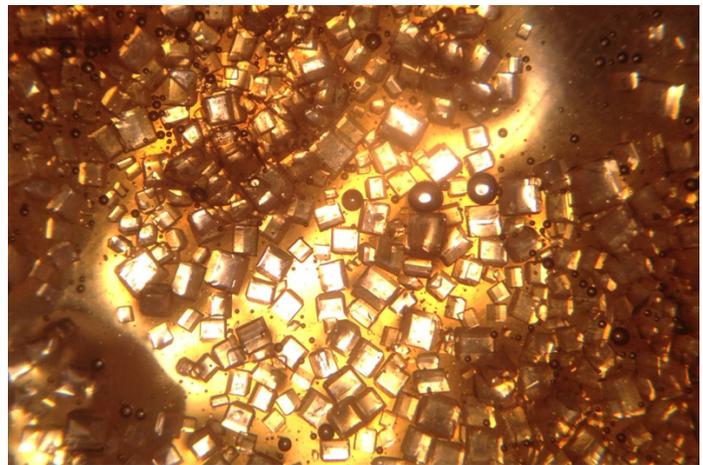


Figure 21. Photo of normal sugar crystals with no c-axis elongation taken on the last day of the 2013-14 crop harvest at Alma Plantation on Jan. 6. (Photo provided by Belisario Montes)



It is interesting to note that on the final sampling date of the Outfield Test, Jan. 3, where tops and leaves were removed, there was no problem in clarifying juice samples in the lab at the Sugar Research Station while at the same time there was a significant reduction in juice pH and total polysaccharides and higher TRS/TC for all varieties in the test from the previous sampling date, Dec. 26. All other parameters remained the same as the Dec. 26 sampling dates when all tops and leaves were not removed. These data showed that the BMPs implemented at the time of the freeze proved to be an effective tool in mitigating the effects of the freeze events of the magnitude that occurred on Nov. 28-30 and that factories could continue to operate with minimal problems in the boiling house so long as the frozen tops and leaves were removed.

Based on an actual field yield of 76.7 tonnes/ha (34.2 tons/ac) and assuming the 6.7 tonnes/ha (3.0 tons/ac) loss, the predicted yield for the 2013 crop should have been 83.4 tonnes/ha (37.2 tons/ac). Further, the reported actual yield of 111 kg/tonne (222 lb/ton) should have been 117 kg/tonne (233 lb/ton) assuming a loss of 5.5 kg/tonne (11 lb/ton) due to freeze. Therefore, the loss for the state sugarcane crop for 2013 due to the freeze events was approximately 1,205 kg/ha (1075 lb/ac) or a total of 200,000 tonnes (221,000 tons) sugar. (B. Legendre, H. Birkett, K. Gravois, H. Waguespack, M. Duet, E. Dufrene, W. Jackson, B. Ball and J. Stein) ■

Chemical Ripeners

Since 1971, 32 processors have closed sugar mill operations. In spite of increases in daily processing capacities of the 11 remaining sugar factories, the total number of days required to process the states crop has increased. Mill managers have shifted the harvest period to begin processing cane earlier in the year to lessen the threat of freezing conditions which often occurs in late December and early January. Shifting of the harvest period into late September or early October has led to a greater proportion of immature sugarcane being processed. Since 1980, glyphosate has been the predominant chemical compound used to enhance early season sucrose. Factors such as crop erectness at the time of ripener application, available soil moisture, plant stress, ripener rate, treatment to harvest interval and sugarcane variety have a compounded effect on the efficacy of glyphosate ripener within a given year. Furthermore, there is interest in evaluating alternatives to glyphosate for use in sugarcane production programs. In 2013, the United States Environmental Protection Agency (EPA) granted registration of trinexapac-ethyl (Moddus 2EC®) as a sugarcane ripener. Utilization of ripeners to increase early season sucrose has proven to a valuable tool for both sugarcane producers and factories.

Early Season Variety Response to Glyphosate

A field experiment was conducted at the Sugar Research Station to measure early season variety response to glyphosate. In 2013, Roundup PowerMax (5.3 oz/A) was applied to second stubble field plots at St. Gabriel, Louisiana. Sugarcane plots were hand-sampled, harvested and weighted 28 days after glyphosate

application on October 3, 2013. Glyphosate statistically increased theoretical recoverable sugar per ton of cane (TRS) for all varieties except HoCP 00-950. Glyphosate increased TRS levels by 18 to 25 percent in HoCP 96-540, L 99-226, L 01-299 and L 03-371, as well as, 12 to 14 percent in L 99-233 and HoCP 04-838 and minimally increased (4 to 8 percent) TRS in HoCP 00-950 and L 01-283 (Figure 22). Averaged across varieties, glyphosate reduced sugarcane yield by 2.1 tons of cane per acre, but increased sugar yield per acre by 746 pounds.

Figure 22. On-Farm Evaluation of Moddus and Glyphosate.

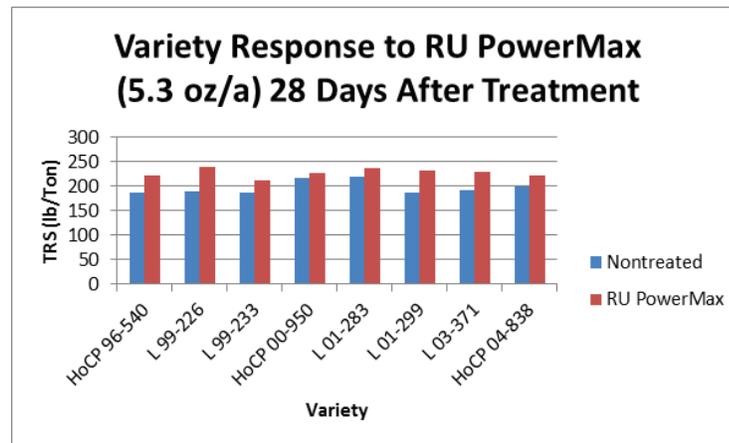


Figure 23. (a) Yield of theoretical recoverable sugar (TRS) per ton of cane (lb/ton) and (b) tons of cane per acre (tons/A) following the application of glyphosate (PowerMax at 5.3 oz/acre and Moddus at 19 oz/acre).

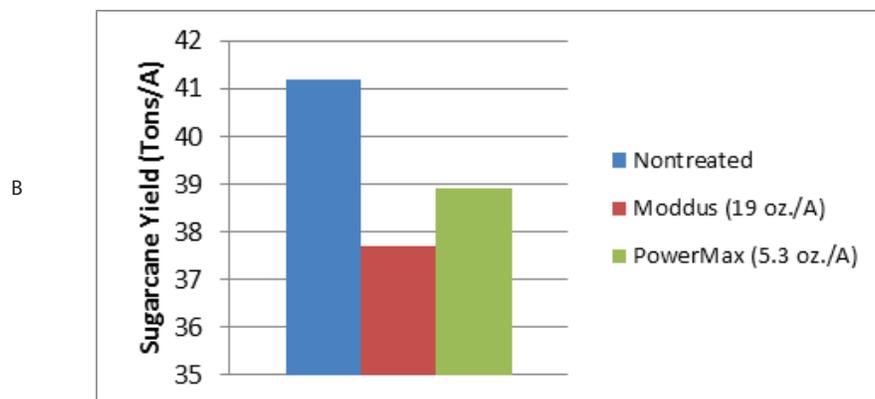
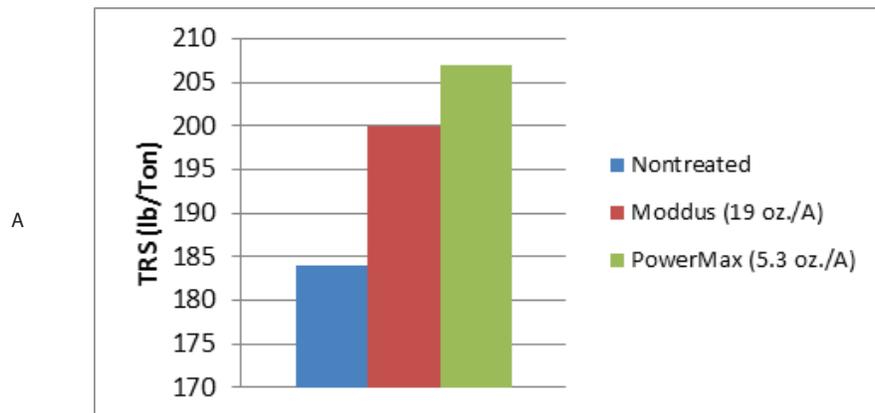
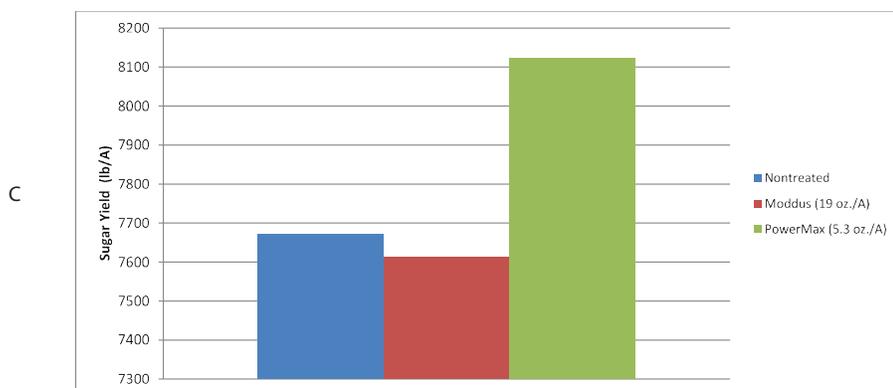


Figure 23. (c) Sugar yield in pounds of sugar per acre (lb/acre) following the application of glyphosate (PowerMax at 5.3 oz/acre and Moddus at 19 oz/acre).



Large-scale, on-farm, evaluations of Moddus (trinexapac-ethyl) and Roundup PowerMax glyphosate were conducted from 2012-2014 on second stubble HoCP 96-540. In 2012, treatments were not replicated within the field, but were applied at three locations (Blackberry Farms, Vacherie, Hebert Brothers, Thibodaux, and Ronald Hebert Farms Jeanerette). The 2013 and 2014 experiments were conducted at Blackberry Farms in Vacherie, Louisiana. Moddus (trinexapac-ethyl) and Roundup PowerMax (glyphosate) was applied aerially at 19 and 5.3 oz/A, respectively. At harvest, Moddus had been applied for 56 days and Roundup Power Max for 28 days. Harvest occurred in mid-October, and two tractor-trailer loads were harvested from each treatment by combine. The harvested area for tractor-trailer loads varied from 0.55 to 0.70 acres. Harvested area and scale weights obtained from Lafourche Sugar Factory were used to calculate sugarcane yield (tonnage). Core sample analyses for obtaining the yield of theoretical recoverable sugar per ton of cane (TRS) were obtained from both front and rear compartments of all trucks that were part of the experiment. Sugar yield (lb of sugar/A) was calculated as the product of sugarcane yield and TRS. Moddus and Roundup PowerMax increased TRS by 8.4 and 12.2 percent, respectively, thus improving early season sucrose concentration (Figure 23a). Moddus significantly reduced cane tonnage by 8.5 percent or 3.5 tons per acre (Figure 23b). Sugar yield for Glyphosate treated cane was 511 lb of sugar per acre greater than Moddus treated cane (Figure 23c). (Orgeron, Griffin, Legendre and Gravois.) ■

Chardcie Verret

Chardcie Verret is graduate of Southern University, where she received her Bachelor of Science degree in Chemistry. She has been employed with the LSU AgCenter-Audubon Sugar Institute for 10 years. She is currently the Analytical Lab supervisor and coordinator. She manages personnel, instrumentation, method development, training and QA/QC protocol for the laboratory. During her time at the Audubon Sugar, she has continued to provide the Molasses Survey Report to the Louisiana Sugar Industry.

The Molasses Survey 2011-2013

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 target purity 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 target purity originates from South Africa (Rein, 2007) and has been confirmed as representative of the Louisiana industry (Saska et al., 2010).

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

The true purity is determined by HPLC and is free of the interferences that can offset polarimetric determinations. The formula for target purity 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).

The target purity is subtracted from the true purity to give a target purity difference or TPD. The target purity difference is used to determine how efficiently sugar is removed from massecuite. “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 purity should be assayed in order to determine how much sugar is lost across the centrifugals. Generally, a lower target purity difference indicates a greater efficiency as it related to the recovery of sugar.



Figure 24. Average Weekly Target Purity Differences(TPD) 2011-2013.

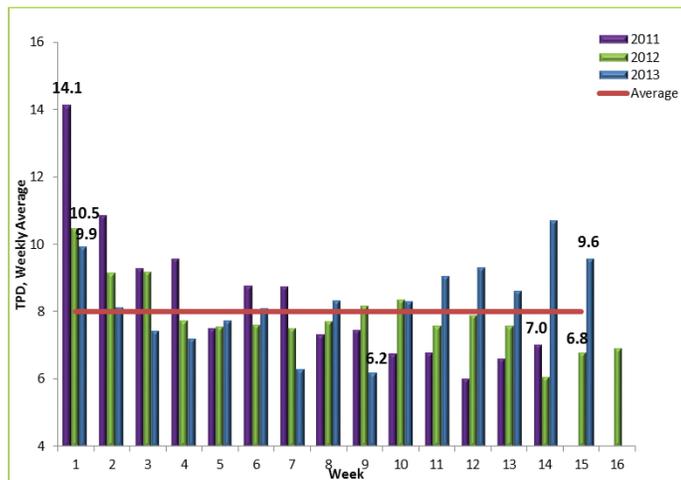
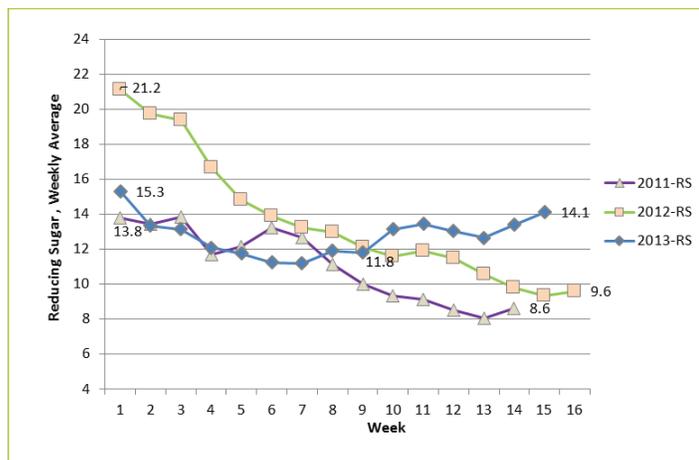


Figure 25. Reducing Sugars Weekly Averages 2011-2013.



Results/Discussion

The 2011-2013 Target Purity Difference weekly (TPD) averages started at their maximum values of 14.1, 10.5 & 9.9. As the seasons continued, the TPDs took it usual downward trend towards the end of the seasons. In the 2013 season the TPDs increased slightly at the end of the season (Figure 24).

In general, there has been a significant downward trend, relative to time, in the amount of reducing sugar in final molasses. For the 2011-2013 seasons, the maximum reducing sugars of 13.8, 21.2 and 15.3 all occurred at the beginning of the seasons. The minimum reducing sugars for the 2011 (8.6) and 2012 (9.6) seasons occurred at end of the seasons. For the 2013 season, the minimum of 11.2 occurred in the middle of the season and slightly increased towards the end of the season (Figure 25).

Additional components of the molasses survey includes the apparent purity, true purity, F/G ratio and

conductivity ash and target purity. From 2011 to 2013, all the components decreased. (Table 13).

Table 13. Additional components of the molasses survey.

Year	App. Purity	True Purity	F/G Ratio	Cond. Ash	Target Purity
2011	37.1	44.0	1.90	15.9	36.2
2012	35.7	42.4	1.63	14.7	34.7
2013	35.7	42.9	1.41	15.0	35.0

During the grinding season, two blind check samples are included with the weekly molasses samples. These samples evaluate the precision and performance of the methods used for the molasses survey by the analytical laboratory. Standard deviations are calculated for key components of the check samples. For the 2011-2013 seasons, the standard deviation for apparent purity decreased from 1.19 to 0.32, which shows the improvement in the analytical laboratory performance (Table 14).

Table 14. Standard deviations of key components of check samples.

	Ref. Brix	App. Purity	True Sucrose	True Purity	Target Purity	TPD
Year						
2011	0.14	1.19	0.42	0.47	0.32	0.45
2012	0.12	0.43	0.43	0.42	0.20	0.38
2013	0.16	0.32	0.40	0.45	0.31	0.38

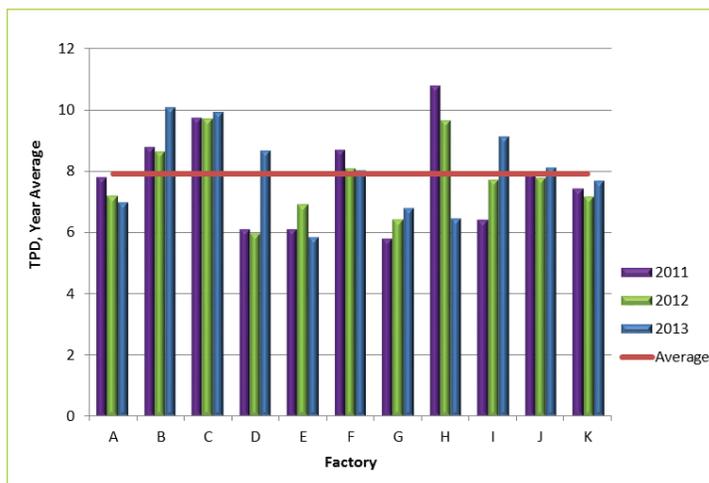
Comparing the results from the 2011, 2012 and 2013 seasons showed a downward trend of the yearly average target purity differences for the factories. Several factories showed a decreased in their yearly target purity difference from 2011-2013 and majority of the factories remain below the yearly target purity difference average of 7.9 (Figure 26).

The 2011-2013 seasons demonstrated little variation. The industry yearly target purity difference averages for 2011 and 2012 was 7.8. For 2013 it was 8.0. The maximum target purity difference for 2011 was 10.8. It decreased slightly for 2012 to 9.7 and increased in 2013 by 0.4 to 10.1. The minimum target purity differences for all three seasons were 5.8 in 2011, 6.0 in 2012 and 5.8 in 2013 (Table 15).

Table 15. TPD Data Summary for 2011-2013.

Year	TPD Minimum	TPD Maximum	TPD Average
2011	37.1	44.0	1.90
2012	35.7	42.4	1.63
2013	35.7	42.9	1.41

Figure 26. Average weekly target purity differences (TPD) 2011-2013.



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

In general, the factories are conscious of their target purity difference and continuing to improve which is an encouraging trend. (C. Verret, D. Dorman and S. Lu)

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Plastic: Formulating Polyester from Cane and Byproducts

Plastic is everywhere, but few recognize its omnipresence in daily life. From polyethylene to polystyrene, polymers come in different colors and transparency depending on the application. The majority of plastic and rubber is petroleum-based, formed of chemicals refined from crude oil. It is imperative to explore biobased polymers as we examine alternative fuel sources in order to envision a balanced energy future where fossil fuels are only part of the industry.

With this goal in mind, the Audubon Sugar Institute began work on polyester in 2010 with a USDA grant awarded for bioplastic. The concept was proven, and a polyester composed of citric acid, glycerin and cinnamic acid was investigated. The work continues with the USDA NIFA AFRI-CAP grant and focuses on extracting aconitic acid from cane with subsequent work on formulating a plastic (Figure 27). Investments in a 3D printer and supplies aim at developing a biobased polymers that could be used to print prototypes or even organ shapes for cellular matrices (Figure 28). (D. Dorman.) ■



Figure 27. Polyester formulated with trans-aconitic acid, the organic acid found in cane plants.

Figure 28. Parts and prototypes printed using 3D printer at ASI using conventional thermoplastic material.



Determining Biorefinery Feedstock Quality with Near Infrared Spectroscopy

In developing an agricultural feedstock for biofuel and biochemical production, it is essential to be able to quantify the potential yield from available components in the feedstock. Near Infrared Spectroscopy (NIR) is an analytical technique that can be calibrated to quantify almost any component quickly with minimal sample preparation. At the Audubon Sugar Institute, the focus is on varieties of energycane and sweet sorghum for use as biorefinery feedstock. These crops have both primary sugars readily available for fermentation in the juice and secondary sugars that require further processing in the fiber. A Spectracane NIR is being calibrated to rapidly quantify these components in both crops. Analysis consists of running 10-15 whole stalks (including leaves and panicles when present) through a hammer mill shredder before running through a conveyer belt system that homogenizes and scans the sample with NIR. The resulting NIR spectra is then input through chemometric software containing the calibration spectra with known quantities of the components of interest. The accuracy of the prediction depends on the robustness of each calibration model.

Models that have been created thus far are presented in Tables 16-19 below for energycane and sweet sorghum respectively showing the predictive ability statistics for the calibration test set samples and an external set of verification samples. The test set samples consist of 30 percent of the total samples available and are used by the chemometric software to optimize the calibration models whereas the external verification set consist of 10 percent of randomly chosen samples completely left out of the calibration process. In identifying a robust model with good predictive ability, the coefficient of determination (R^2) should approach 100 for the test set, the correlation coefficient (r^2) for the external set should approach 1, and the residual prediction deviation for both sets should be above 3 values of 2.5-2.9 acceptable for rough screening.

Continuing work will involve developing calibration models for components in the fiber portion of both crops, including cellulose and hemicellulose. Also, the recent addition to ASI of a multipurpose bench top NIR will allow for separating the juice and fiber prior to scanning for more precise predictive models. (S. Lu.) ■

Table 16. NIR calibration model statistics - sweet sorghum juice components.

Component	Brix in Juice		Sucrose % Juice		Glucose % Juice		Fructose % Juice	
	Test-Set	External	Test-Set	External	Test-Set	External	Test-Set	External
No. of Samples	60	23	60	23	60	23	60	23
R^2	97.17		92.99		95.62		96.27	
RMSEP	0.459	0.426	0.857	0.87	0.414	0.43	0.345	0.333
RPD	5.96	5.52	3.79	3.59	4.8	4.34	5.23	5.21
Bias	0.0317	0.0338	0.0647	0.00217	-0.0391	0.0647	-0.0487	-0.0062
r^2		0.9849		0.962		0.9736		0.9818

Table 17. NIR calibration model statistics - sweet sorghum stalk components.

Component	Juice % stalk		Fiber % Stalk	
	Test-Set	External	Test-Set	External
No. of Samples	60	23	60	23
R^2	84.98		85.19	
RMSEP	1.82	2.54	1.81	2.56
RPD	2.69	2.35	2.75	2.34
Bias	-0.515	-0.448	0.597	0.473
r^2		0.9115		0.9103



Table 18. NIR calibration model statistics - energycane juice components.

Component	Brix in Juice		Sucrose % Juice		Glucose + Fructose % Juice		Ash % Juice	
	Test-Set	External	Test-Set	External	Test-Set	External	Test-Set	External
Validation Set								
No. of Samples	44	16	44	16	44	16	44	16
R ²	92.78		93.14		75.65		86.76	
RMSEP	0.636	0.406	0.715	0.693	0.285	0.251	0.115	0.0859
RPD	3.73	4.89	3.98	3.55	2.03	2.18	2.79	2.31
Bias	-0.0489	0.0318	-0.201	0.0688	-0.0145	-0.169	-0.019	-0.0349
r ²		0.9803		0.9625		0.8925		0.9182

Table 19. NIR calibration model statistics- energycane stalk components.

Component	Juice % stalk		Fiber % Stalk	
	Test-Set	External	Test-Set	External
Validation Set				
No. of Samples	44	16	44	16
R ²	91.69		91.69	
RMSEP	1.32	1.27	1.32	1.27
RPD	3.47	2.54	3.47	2.54
Bias	-0.0083	-0.116	0.0083	0.116
r ²		0.9272		0.9272





Daira Aragon

Simulation models for two factories were developed in the software named SUGARS. Factories were visited during the grinding season and offseason to gather information related to equipment dimensions, flow properties, plant layout, etc. Results were discussed with factory engineers and models were modified according to the suggestions given at these meetings.

Nutsch purities of massecuites along the C-strike from pan, cooling crystallizers and reheater, as well as purities of molasses from each centrifugal and crystal size of C-sugar from each centrifugal, were measured at the request of one factory. Samples for these tests were collected weekly at the factory.

Technical assistance was provided during the design, installation and test-run of a pilot-scale filtrate clarifier at one of the factories. Samples of clarified juice and filtrate were collected from the pilot unit and the full-size clarifier. Turbidity of samples was measured to evaluate the performance of filtrate clarification. Meetings with factory personnel were held to discuss the results.

Technical assistance and information was provided to the factories using double purge of C-massecuites. Samples of massecuites, magmas and molasses were analyzed for purities and crystal size. Discussions about the performance of the system were held with factory personnel. C-sugar samples were collected from several factories for analysis in the laboratory with a CILAS particle size analyzer.

Harold Birkett and J. Stein

Training and information were provided on a number of issues to several factories and one refinery. Many of the requests were for assistance with analyses to determine starch, color, entrainment losses and dextran. Assistance was also provided to several factories, growers and university personnel seeking information on core lab operations and understanding both core lab and factory reports.

Ten milling tests were conducted at the request of three factories to determine individual and overall tandem extraction. Results provided mill engineers with information necessary to make changes to the mill that would decrease sugar losses.

Seven boiler emissions compliance tests were monitored during the 2011 crop. Five tests each in 2012 and 2013 were also monitored. These tests are required to demonstrate compliance with permits issued by Louisiana Department of Environmental Quality (LDEQ). Assistance was provided by recording operating conditions, checking measured gas flows with calculated gas flows to ensure that reported data was reasonable and reviewing draft reports for accuracy from the testing company before final submission to LDEQ.

Several meetings and/or discussions were held with sugar mills and start-up bioenergy companies. These meetings have helped to educate both on the realities of current and potential energy usage and bagasse availability at the mills.



Extension agents engage children to learn about sugar production and products at AgMagic on the River, held at Docville Farm in Violet, Louisiana.



A lab experiment was conducted at the request of one factory to determine if cold clarification could remove starch and, if so, to quantify the amount removed. The experiment conducted at the factory determined that starch removal with cold clarification was about 40 percent. Starch removal by cold clarification could potentially be an option when juice with very high starch levels cause extreme processing problems.

Juice from several Louisiana sugarcane varieties were collected during the 2011 season for starch analysis in order to provide information to the sugar industry on the varying starch content in current and potential commercial varieties.

Personnel from USDA, Sugar Station, American Sugar Cane League and Audubon Sugar Institute collaborated to conduct cold tolerance tests following freezes in late November, 2013 and early January, 2014. Among variables measured were pH, apparent purity, titratable acidity and polysaccharides. The information obtained is very useful to both growers and factories as it provides the cold tolerance of several varieties and can indicate the degree of deterioration to be expected by the factories.

Franz Ehrenhauser

During the 2013 crop, more than 300 samples of massecuites, seed slurry and A, B and C-sugars were collected and analyzed for their particle size and distribution.

During the 2013 crop, assistance was provided to one mill to aid in the proper FDA classification of mill utilized chemicals.

Prior to the 2014 crop all seed slurry preparation methods were evaluated and communicated to the mills. For factories that use ball mills to prepare the slurry, every single ball mill was evaluated for its performance and the results were presented to the respective factory. The results are available to the factories and can be used to confirm the performance of the ball mill prior to the season.

Based on the request of one mill, several hydrated limes were tested to compare their performance for clarification operation. The evaluated lime samples are very comparable and unfortunately no reliable indicator for performance was found.

The analytical staff currently includes one Associate Chemist, a research associate and a post-doctoral researcher.

ASI currently has three operational HPLC systems. The Louisiana Board of Regents (LaBOR) and the American Sugar Cane League (ASCL) provided the funding necessary to acquire two Agilent 1200 HPLCs (Figure 29). Configured with differential refractive index detectors (DRIs), these isocratic instruments are used for the routine quantification of the carbohydrates commonly found in either molasses or lignocellulosic hydrolysates. These instruments are operated using BioRad Aminex mixed-mode columns (HPX-87) in either potassium or lead-form. The third instrument is an Agilent 1100 HPLC with quaternary gradient pump and a diode array (DAD)/evaporative light scattering (ELSD) tandem. This instrument can be operated in either reversed or normal phase. We have a wide range of columns available (NH₂, C18, phenyl, cyanopropyl, etc.). This system is equipped with an Agilent 1200 automated fraction collector that is software controlled (LC-3D Chemstation). This system is equipped with an autosampler capable of handling 100 samples. All of these systems are automated using Agilent LC-Chemstation software and CAN interfaces via TCP.

The most recent acquisition includes an ion chromatography (IC) a Thermo Scientific Dionex ICS-5000+ HPIC system (Figure 29). The equipment was purchased through a Louisiana Board of Regents Enhancement Grant. The instrument features a temperature controlled autosampler with dual pumps (capillary and microbore) and eluent generators for reagent free operation up to 5,000 psi continuously. With three detectors available for anion, cation or carbohydrate analysis, the equipment can be configured to run any two simultaneously. The capillary channel provides for increased productivity, increased mass sensitivity, expanded capabilities, and improved performance while only using 15ml of water a day. The instrumentation allows analytical testing for more than 15 organic acids, 5 cations, 40 anions including phosphate and carbohydrate detection for up to 8 common sugars.

An Agilent 7890A gas chromatograph (GC) has also been purchased and installed. The 7890A GC has been utilized mainly in analyses of ethanol and fuels. An Agilent 7890 GC equipped with a 5975C inert mass-selective detector (MSD) is also available for use. This instrument is equipped with a turbomolecular pump for minimal downtime and an autosampling robot capable of handling 100 samples. The instrument is controlled via Agilent MS-Chemstation software and is updated to

Analytical Capabilities

include both the NIST and Wiley spectral libraries.

Spectroscopy related instruments include a variety of UV-Vis instruments including Spectronic Genesys model 6 and Beckman-Coulter models DU 730 and 800. A SpectraCane NIR integrated with a Dedini shredder is available for use with whole sugarcane, sorghum and energycane. Additionally, we have recently acquired a multipurpose NIR system. It is the Bruker MPA system. It can be used to analysis liquid or dry samples over a multispectrum range.

A Shimdazu AA-6650 graphite furnace atomic absorption spectrophotometer is available for use. Lamps are acquired on an element-as-needed basis.

In addition, we have a Varian Cary Eclipse spectrofluorometer and a Thermo-Nicolet 6700 fourier transform infrared (FTIR) spectrophotometer. The Eclipse can handle up to four cells for sequential (kinetic) experiments with programmable temperature and in-cell stirring. The instrument is capable of performing experiments involving chemiluminescence, photobleaching and photon-counting. The FTIR is equipped with a single-bounce diamond-anvil ATR, KBr beam splitter (350 cm⁻¹ or 28571 nm) and a gas flow cell with heated lines suitable for interfacing with a thermogravimetric analyzer (TGA).

A Foss Soxtec 2050 and 8000 are automated soxhlet extractors that are available for use. Each can extract up to six samples simultaneously and is equipped for automated solvent addition and recovery. This apparatus is suitable for general use including analytical protocols for the compositional analysis of biomass.



Figure 29. HPLC systems: left, the Agilent 1200 series; right, a new ion chromatography (IC) a Thermo Scientific Dionex ICS-5000+ HPIC system.

A Parr 6200LE semi-automated isoperibol oxygen bomb calorimeter with two 1008 bombs, pellet press and loading station is available for the determination of high heating values (gross calorimetry) and S,N content in both solid and liquid samples.

We have a Mettler Toledo TGA/SDTA851 TGA equipped with an autosampler capable of handling 34 samples.

Other instrumentation includes temperature compensated critical angle refractometers (Bellingham and Stanley) with 0.01 g/100g (brix) resolution, a Rudolph Autopol 880 polarimeter with 0.01°Z resolution (589 or 880 nm) and a conductivity meter with temperature compensation.



Figure 30. Left, Agilent 7890 Gas Chromatography (GC) System; Right, Foss 8000 Soxtec Automated Extractor.

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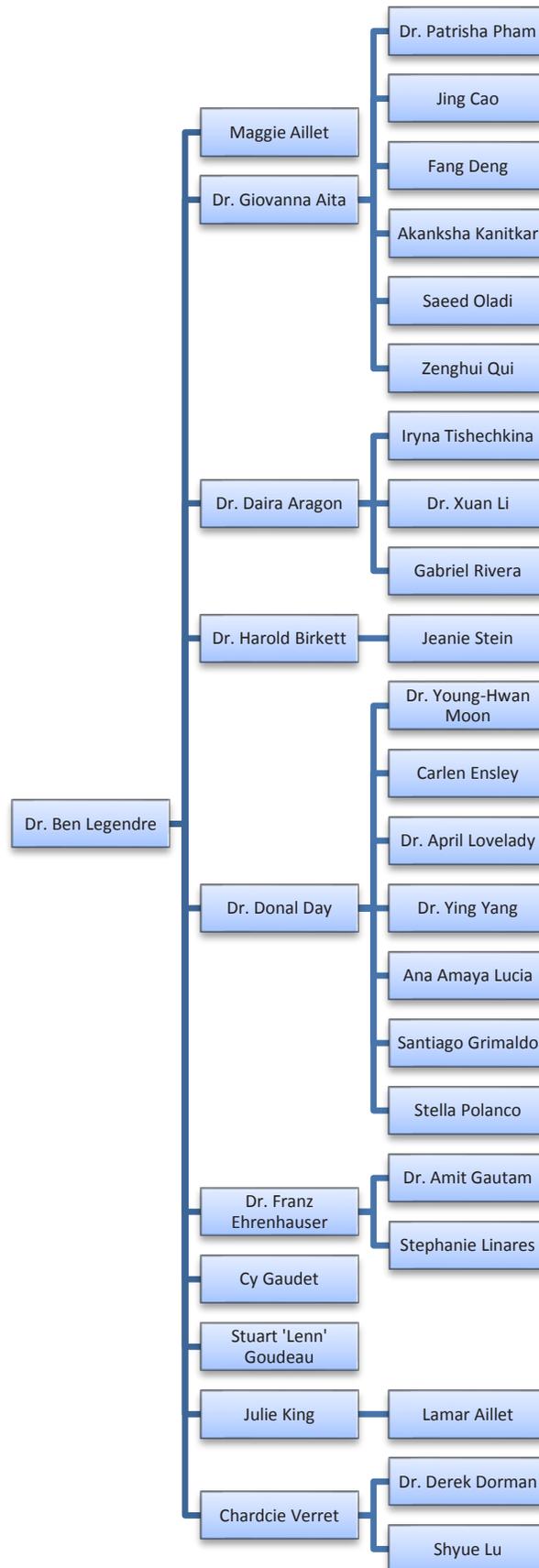
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Pilot Plant



The pilot plant at Audubon Sugar Institute was constructed to produce syrups from various feedstocks, including sugarcane, energycane and sweet sorghum, with equipment similar to an industrial sugar mill. The feedstock (cane or sorghum) is delivered from field and fed into the cane table hopper. The feed passes through two sets of knives and a hammer-type shredder to chop the feedstock into bagasse.



The bagasse then enters the first of four three-roll mills that press the juice from the bagasse. Countercurrent maceration is used so that the juice produced from mill 4 is recycled to mill 3, then to mill 2, which helps increase sugar extraction. The juice that is produced in mills 1 and 2 are sent forward for further processing. The juice is sent through a rotary drum filter to remove any residual bagasse.



The juice then enters the clarification system. The juice is heated to boiling and flashed. Lime is added to adjust the acidity. A flocculent is added, which encourages the field soil and impurities to coagulate. The juice enters the clarifier, where the impurities are pumped out of the bottom and the cleaned juice rises to the top and overflows to a tank for further processing.



The juice is evaporated in a triple-effect plate evaporator system to produce the final syrup.



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