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Product Parameters Optimization for Double Purge of C-Magma Integrated with Conventional Three-Boiling Crystallization Scheme to Improve Raw Sugar Whole Color

Luz Stella Polanco
Louisiana State University and Agricultural and Mechanical College

PRODUCT PARAMETERS OPTIMIZATION FOR DOUBLE PURGE OF C-MAGMA INTEGRATED WITH CONVENTIONAL THREE-BOILING CRYSTALLIZATION SCHEME TO IMPROVE RAW SUGAR WHOLE COLOR

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Luz Stella Polanco
B.S., Universidad del Valle, Cali-Colombia, 1989
M.S., Louisiana State University, 2009
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ABSTRACT

Efficient crystallization in raw sugar production is the result of a combination of factors and responses that meet specific criteria. Matching a sugar quality standard, maximizing sugar yield, maximizing crystallization equipment capacity and minimizing heat requirement are all necessary optimization criteria. A double centrifugation or purge of C-magma is intended to improve raw sugar whole color by the reduction of color recirculation with the sugar crystal. Combining a three boiling crystallization scheme with a second centrifugation after the last crystallization stage required optimizing the input/output product parameters (purities) and the location of the liquid recycle component (double purge molasses) for efficient integration. Sugars™ and JMP® were software programs used to solve this multiple-response optimization problem using a multistage approach. The optimization strategy went from actual data correlations, experimental design, computer simulations and surrogate models to a desirability approach that transformed the problem from multiple-objective functions into a single objective function, the overall desirability.

The implementation of the double purge system by three Louisiana sugar factories confirmed the reduction of whole color of raw sugar, up to 47%, and the linear relation between double purge magma purity and raw sugar whole color. An overall desirability expression was applied to find the optimal input/output product parameters of the double purge system for different scenarios using JMP® optimization algorithm. Optimal control parameters and recycle determined by this strategy matched the average settings used during actual operation at the 2013 sugarcane crop season. In addition, surrogate models were used to evaluate the importance of the random and control parameters for each response and for each syrup purity scenario. This optimization strategy offers an approach not only to evaluate the importance of several product parameters or the optimal settings, but also offers a better understanding for sugar factory processes and for troubleshooting specific undesired responses. In the future this optimization strategy may be applicable for integrating additional equipment and for improvements of existing process stages at any sugar facility.

CHAPTER 1 INTRODUCTION

1.1. Integration of a Double Purge of C-Magma System to Improve the Whole Color of Raw Sugar – Project Scope

The integration of a second centrifugation (double purge) of C-magma at the last stage in a traditional three-boiling crystallization scheme in order to improve whole color of raw sugar requires definition of product parameters (input and outputs) and recycle point(s) for the double purge molasses. A greater separation efficiency of crystals from surrounding molasses at the last crystallization stage is achieved by a double centrifugation, normally, dedicating the first centrifuge to separate final molasses with low purity (sucrose % soluble solids) and the second centrifuge (double purge) to produce a high purity magma (Chou 2000). Product parameters, such as the C-magma purity coming from the first centrifugation and the double purge magma purity out of the second centrifugation, and the point of recirculation for the double purge molasses are related to the raw sugar whole color. The recirculation of impurities to previous stages also affects sugar losses to final molasses, energy requirements and processing volumes (Figure 1.1). A double purge system for C-magma has to be assessed as a multi-objective or multiple response optimization and the criteria to define the system settings must be based on the desired raw sugar whole color as well as the overall performance of the boiling house. This double purge magma optimization project covers modelling, factory trials, full implementation, metamodelling, variable importance assessment and multiple response optimizations for different syrup quality scenarios.

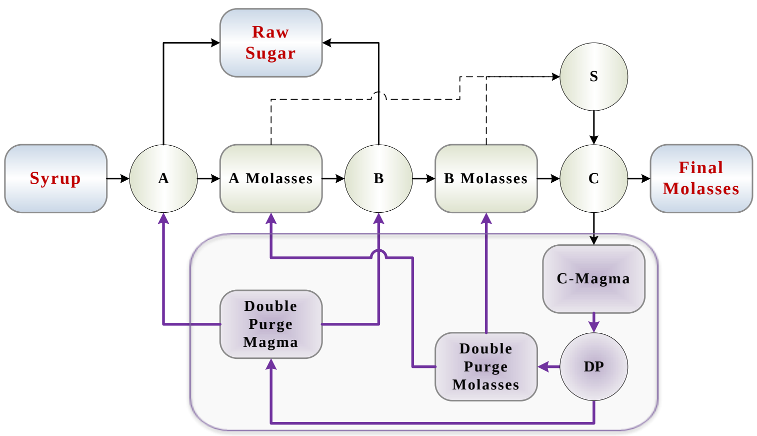


Figure 1.1 Block diagram of a three-boiling crystallization scheme (A, B, C) integrating a double purge (DP) or double centrifugation of C-magma to improve the whole color of raw sugar. Double purge magma is used as a crystal seed on A and B crystallization stages. Double purge molasses can be recycled to A or to B molasses

1.2. Raw Sugar Importance and Quality

The U.S. Food and Drug Administration defines raw sugar from a sugarcane factory as an intermediate food product (not for human consumption) which requires further refining, since it contains impurities that have to be removed (FDA 1980). Consequently, raw sugar is the raw material for the sugar refinery where through mainly decolorization and recrystallization different quality (food or chemical uses) white-sugars are produced. From juice extraction at mills to the crystallization of high purity sucrose crystals in the refineries, sugar manufacturing requires the elimination of complex mixtures of non-sucrose compounds and colored impurities. Refineries, practically, have been responsible for purification and color removal for many years but, refineries are not energy autonomous, everyday more of these tasks are being transferred to the energy self-sufficient sugarcane factories. Worldwide, refineries are facing escalating manufacturing costs and higher product specifications (refined sugars) to satisfy their markets (Eggleston 2010). The sustainability of both refiners and raw sugar producers is based on two important requirements, low sucrose loss and low economic cost (Chou 2000). Fluctuating contract prices for raw sugar (Figure 1.2) and high variable production costs (Figure 1.3) limit the ability of the sugarcane industry to invest in high capital, long term projects. At a local level, Cargill, Inc. and Louisiana Sugar Growers and Refiners, Inc. (a cooperative of sugar growers and seven mills in Louisiana) started in 2011 the operation of a new "state-of-the art" refinery (Louisiana Sugar Refining, LLC – LSR). On an energy basis, the new refinery required higher quality raw sugar than it was previously demanded. This scenario, together with premiums and penalties on color in the contract

prices has encouraged the raw sugar manufacturers in Louisiana to raise their standards for polarization and color to approach HP (high polarization, 98.0 – 99.3 °Z) and VHP (very high polarization, > 99.6 °Z) raw sugar. Table 1.1 shows the quality standards for these raw sugars in the world market.

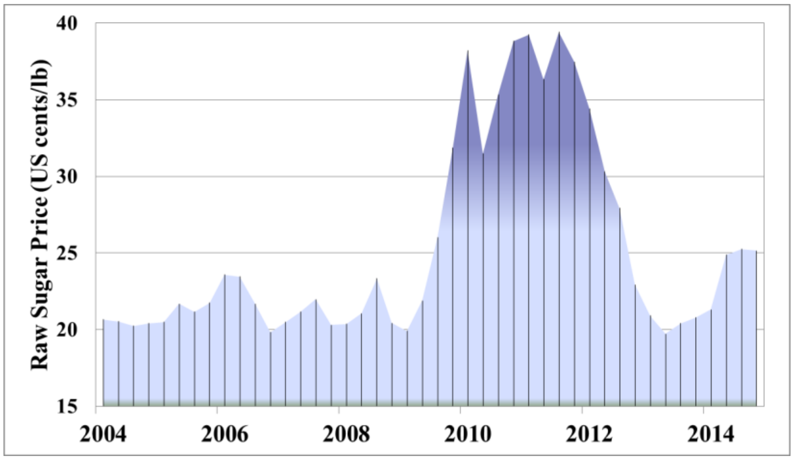


Figure 1.2 Raw sugar price fluctuations in United States from the first quarter (Q1) of 2004 to the fourth quarter (Q4) of 2014, contract # 14/16 New York (Haley 2015)

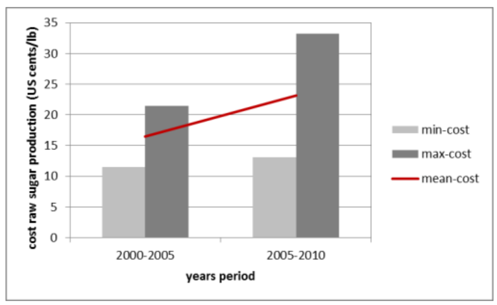


Figure 1.3 Raw cane sugar maximum and minimum production costs for producers in the NAFTA area: United States – Mexico. Fuel, chemicals and fertilizers (79%), labor (36%) and capital (8%) were responsible for the increment on the raw sugar production costs (Haley 2011)

Table 1.1 High grade raw sugar specifications, color @ pH 7.0 in ICUMSA Units IU (Rein 2007)

	Polarization (°Z)	**Color (IU)**	**Producers**
V-VHP (Very, Very High Pol)	> 99.6	300 – 700	Brazil (VVHP), Australia (QHP)
VHP (Very High Pol)	> 99.3	1100 – 2000	South Africa (VHP), Australia (IHP)
HP (High Pol)	98.0 – 99.3	~1800	South Africa(HP), Australia(Brand1)

In Louisiana, raw sugar production is third in the top commodities produced in the State, having a total value in 2013 of $ 771 million (Westra 2014). In 2013, the United States produced 70% of the sugar that was internally consumed (~ 12 million short tons), distributed between beet – 58 % and sugarcane – 42 % with 94% of the produced sugar used for food and beverage purposes. In the same year, Louisiana produced approximately 3.7 million short tons of raw sugar being the second raw sugar producer (~ 43%) in the U.S. while Florida was the first producer (~ 48%), other US raw sugar producers are in Texas and Hawaii (Haley 2014). Raw sugar production is an important industry not only for Louisiana but also for the United States economy.

Double centrifugation or double purge of C-magma to improve sugar quality was a common procedure during the first half of 20th century (van Hengel 1983; Jullienne 1989). Then, with a more limited technological scenario – respect to process automation and centrifugation, the system was tried in Louisiana sugarcane factories, operating with a three-boiling crystallization scheme. As sugar factories expanded and there were no standards for

color in the early 20th century, this system fell out of use. However, published system settings (Hugot 1986) and new models (using Sugars™ software) give a glimpse on expected results and process stream flows to build modern double purge systems. Existing material and energy balance spreadsheets or specific software for the sugar industry, like Sugars™, can be used to create a deterministic model – using both historical data and sample analysis, to determine the flows of each stream and the required energy. However, there are no published reports that combine process parameters (e.g. temperature, pressure, recycle) and/or product parameters (e.g. purities or concentrations) for the whole crystallization station to optimize the production of raw sugar. This research utilizes specific important factors (main effects and interactions) to set product quality, yield and process efficiency in order to integrate double purge of C-magma to the boiling system, a multiple objective optimization.

1.3. Project Outline

The double purge of C-magma project involved model creation, trials, full implementation, sampling, chemical analysis, statistical analysis, model validation, experimental design for computer simulation and analysis; creation of surrogate models and application of desirability functions for multiple response optimization (Figure 1.4). The Sugars™ (2014) software was used for modeling and simulations and the JMP(R) (2014) software was used for statistical analysis, experimental design, main effects, interactions and model fit of selected responses (surrogate models) and, for multiple response optimization.

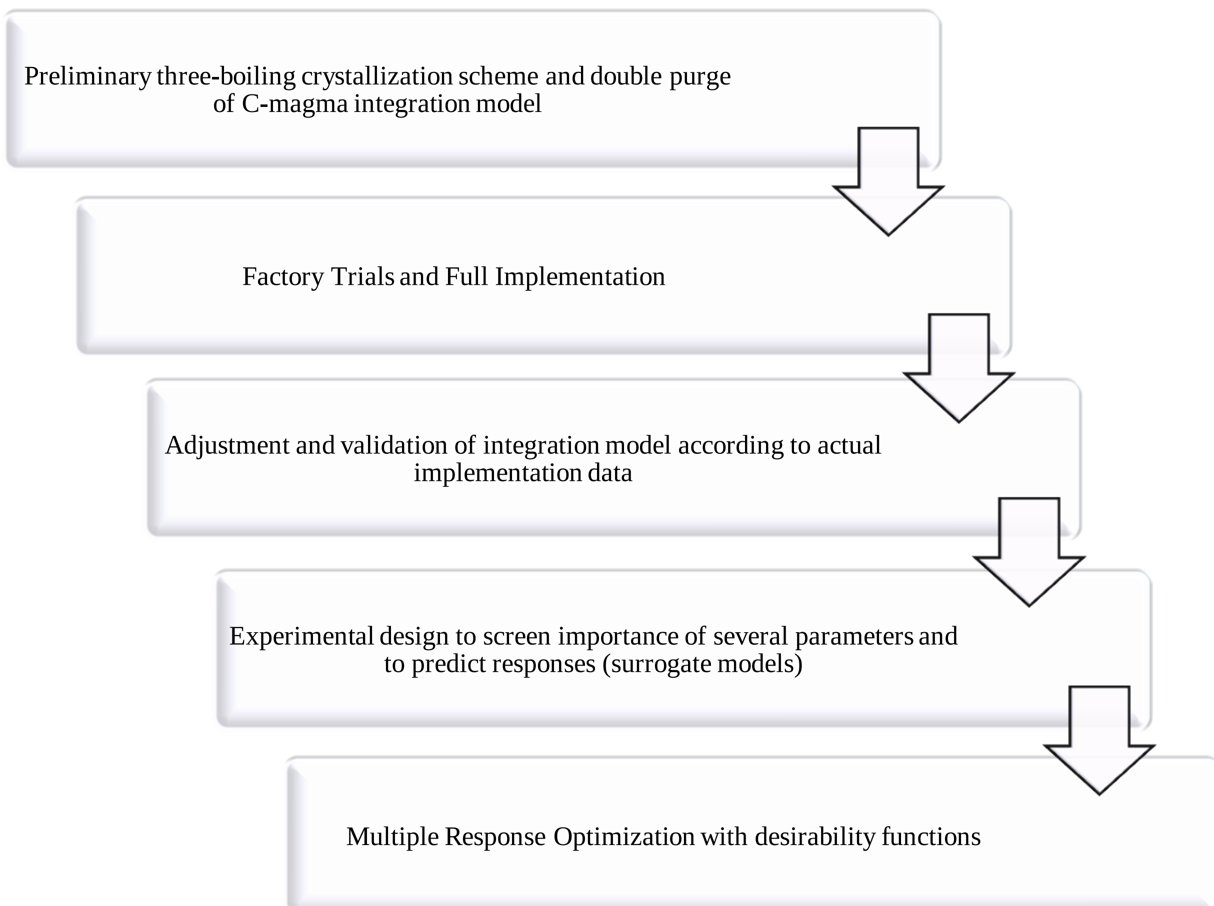


Figure 1.4 Block diagram for the multiple response optimization strategy for a three-boiling crystallization scheme with double purge of C-magma

1.4. Project Outcomes

The improvement of the raw sugar polarization and color normally requires costly modifications of the crystallization scheme like a double magma or a VHP (very high pol) crystallization system (van Hengel 1983; Wright 1989; Broadfoot 2006). Double purge of C-magma offers an easily implemented alternative for improving raw sugar color with minimal capital investment (Jullienne 1989; Madho and Davis 2008). There are few recent publications which mentions the use of double purge of C-magma (Perez, Moreno et al. 1997; Bourzutschky 2005; Iswanto 2007), but there is a paucity of information about its implications on boiling house operation and the final color in solution measured specifically at 1.2 µm and 8.5 pH (US sugar refinery contract price specification). The scope of this project covers from the initial integration model, full scale implementation to the application of desirability function for multiple response optimizations. The main achievements of this project are:

- A significant raw sugar color reduction (~47%) compared to the color of raw sugar produced with a single purge and, in general terms a better raw sugar quality
- Full implementation of the system by three raw sugar factories in Louisiana
- Measurement and modeling of color profiles for Lula Sugar Factory
- Parameter importance for raw sugar whole color, heating requirements and boiling house processing capacities responses
- Metamodeling (surrogate modeling) approach to approximate the boiling house responses to the variation of controlled double purge parameters combined with boiling house random parameters and scenarios
- Desirability functions approach to the multiple response optimization of the double purge of C-magma system

CHAPTER 2 LITERATURE REVIEW

2.1. Introduction

In the past, sugar refineries ‘blued’ the sugar crystals during production of white sugar by injecting an emulsion of blue ultra-marine at the graining point during crystallization to neutralize the natural ‘yellowish’ coloration to improve the visual “whiteness” of the sugar crystal – “contenting the eye of the purchaser” (Geerligs 1909). Indeed, color (appearance) is an important control parameter for refineries. Color in solution is a standard specified to trade sugar on the market (ICE 2008). This literature review will help the reader understand the nature of color in cane raw sugar, analytical procedures applied for color measurement, causes and prevention of color formation, feasible color reduction technologies for raw sugar production, advantages of double centrifugation or double purge of C-magma, challenges and recommended system settings for efficient integration to a three-boiling crystallization system at a raw sugar factory. There is also a review of some publications on the application of modeling and simulation of the sugar manufacturing process in order to evaluate the effects of changes in plant configurations. Multiple-response optimization is still a new research field for the sugar industry.

2.2. Raw Sugar Color

Color in cane sugar is the result of mixtures of colored compounds with different molecular weights (Table 2.1). Some originate in the sugarcane plants and some are formed during processing (Chen and Chou 1993; Bento 2008). During sucrose crystallization, macromolecular impurities (high molecular weight colorants and polysaccharides) present in sugar massecuites have high affinity for the crystals, affecting not only the color, but also purity, growth rates and the shapes of the resultant sucrose crystals (van der Poel et al., 1998). The impurities (non-sucrose) in raw sugar crystals are found on: 1) the surrounding mother liquor layer on the crystal surface, 2) absorbed on the crystal faces or bound to the crystal surfaces – inclusion and, 3) trapped between crystal layers due to rapid crystal growth – occlusion (van der Poel et al., 1998).

Table 2.1 Sugar colorants by groups according its molecular weight – MW: 1- original plant pigments or color precursors and- 2- Color molecules formed during the process (Bento 2008)

VHMW	HMW	MMW	LMW
> 200,000 Da	**12,000 – 50,000 Da**	**2,500 – 12,000 Da**	**< 2,500 Da**
Phenols-polysaccharides[2]	Melanoidins[2*]	Hexoses Alkaline Degradation Products – HADP[2]	Flavonoids[1]
Enzymatic Browning Products[2] of Cell Wall Polysaccharides – CWP[1]	Phenols Browning Product (melanin)[2*]	Hydroxy-methyl-furfural – HMF Polymers[2*]	Phenolic Acids[1]
		Caramels[2*]	Phenols-Iron complexes[2*]
H: High; VH: Very High; M: Medium; and L: Low * Sugar colorants formed at the boiling house			

Bento (2008) reviewed the research that had been done on the nature of colorants and its location in the crystal for raw cane sugar production. Table 2.2 shows the distribution of sugar colorants inside and in the external syrup layer on the crystals. Some relevant facts about color in raw sugar are (Bento 2008):

- the weight % of colorants in raw sugar with 98.5 % sucrose content (~1 % non-sucrose content) is about 0.16 – 0.20 %;
- 70 % of the colored compounds are high molecular weight (>12,000 Da);
- 73% of the color material is located on the surface layer of the sugar crystal;
- 60 - 70% of the total color can be removed by affination (washing) at the refinery,
- During storage, color of raw sugar can double, depending on the type of colorants present, impurities in the external syrup layer, and storage conditions (storage time, temperature, relative humidity);
- Color inside the crystal is affected by the color of the C sugar used as seed for crystallization.

These observations give insight on the importance of sugar crystallization and centrifugal operation to raw sugar color.

Table 2.2 Location, type and molecular weight in Daltons (Da) of colored compounds (colorants) in a raw sugar crystal (Bento 2008)

Crystal Location	Colorant Type	Molecular Weight (Da)
Inner Crystal	Cell Wall Polysaccharides (CWP) complex	>200,000
	Enzymatic browning products	>200,000
	Melanoidins	10,000 – 50,000
	Neutral phenols	<2,500
External Syrup Layer	Melanoidins	10,000 – 50,000
	HADP	5,000 – 50,000
	Caramels	2,500 – 10,000
	Flavonoids	<2,500
	Phenolic acids	<2,500

It needs to be mentioned that the colorant matter in raw sugar from sugarcane, is similar, to that in sugar beet, but the individual concentrations, molecular weights and the locations of these colorants on the sugar crystals are different. While methods for preventing color formation are similar for both sugar beet and the sugarcane processing, the technology for color removal is different. White sugar can be produced in a single stage from sugar beet, while white sugar from cane requires raw sugar production followed by refining. Figure 2.1 illustrates the color levels by process stage in the sugarcane and sugar beet industry (Godshall, Vercellotti et al. 2002).

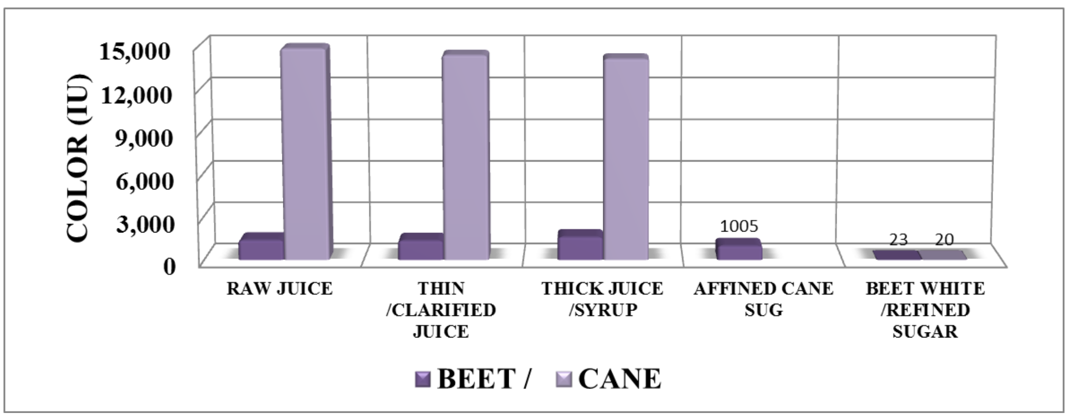


Figure 2.1 Color values (IU-ICUMSA units) for different process stages at the Beet and at the Sugarcane industry (Godshall, Vercellotti et al. 2002)

2.3. Color Measurement

Color can be approached as 'visual appearance' or as a measurement of the impurities that cause it. Color on sugar crystals is related to the light reflected by the crystals, while color of sugar in solution is related to the light absorbed when it passes through the solution (Meade and Chen 1977). There are several dedicated tests to measure color that can be used for "troubleshooting or research purpose" (Clarke 1997). The absorbance of a solution is the best tool to evaluate the formation or reduction of color by process stage (Meade and Chen 1977). Godshall (2005) said that color is "a construct used to evaluate the degree of refinement of raw sugar" and in the same way color measurement is defined as a "set of standardized test parameters" accepted by the raw sugar manufactures and by the refineries to be used both for payment purpose and to evaluate refinery performance (Chen and Chou 1993). Any change in the parameters will produce different results on color measurement.

The colors of solutions of raw sugar range from colorless, to amber or to very dark brown near to black (Meade and Chen 1977; Chen and Chou 1993). Solutions of white sugar have lower absorption in the yellows or reds than solutions of raw sugar. Figure 2.2 shows the relationship between the transmitted color and the variation of the attenuation index for some sugar products in both the ultraviolet and the visible range of the electromagnetic radiation spectrum. Highly colored sugar products absorb more in the yellow region (560 – 600 nm) peaking at 560

nm for dark solutions compared to white sugar samples which peak in the ultraviolet region (<300 nm). The wavelength chosen for color measurement – 420 nm ±10 nm, was because it gives high attenuation index for both white and raw sugars (Chen and Chou 1993).

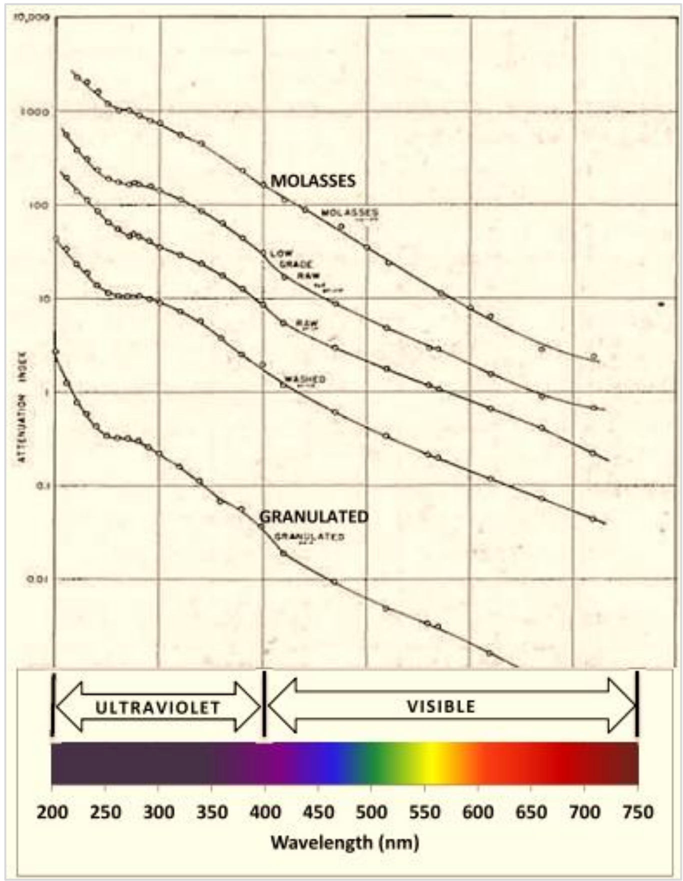
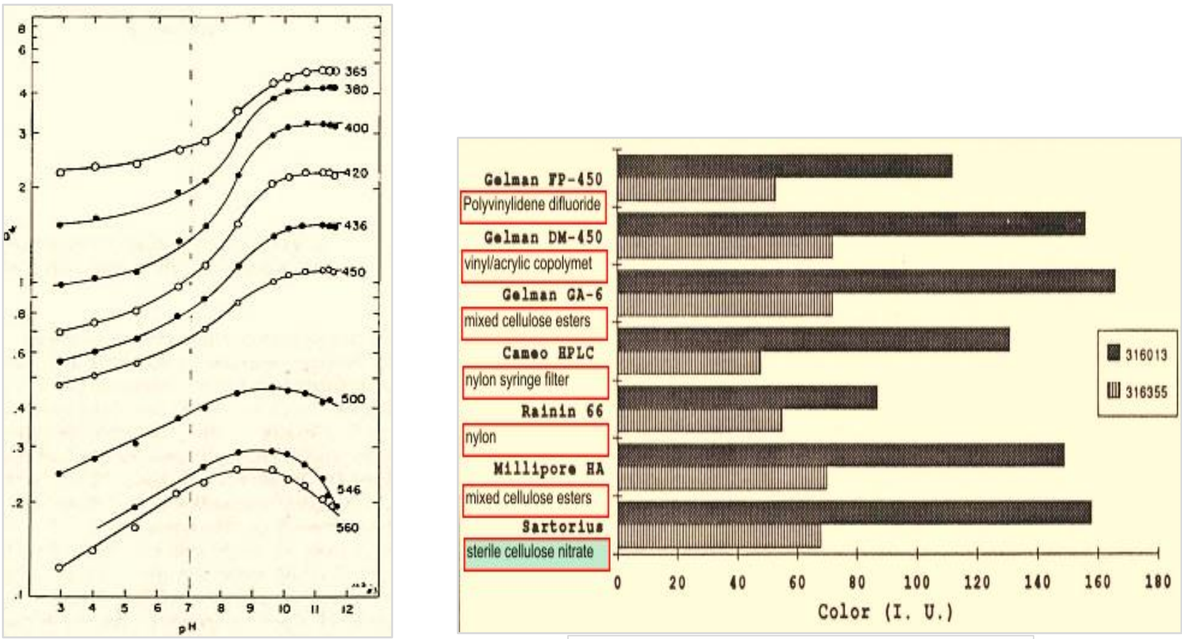


Figure 2.2 Attenuation indices for different sugar products solutions depending on the light wavelength: the lower line – granulated white sugar and the upper line – the darker molasses. At the bottom of the graph is the approximated transmitted color (observed) for each wavelength. Adapted from Carpenter and Deitz (1963)

The acidic nature of some sugar colorants makes color measurement very sensitive to pH (Chen and Chou 1993). Although the sensitivity to pH depends on the nature of the sugar colorant, color measurement of sugar products shows high variation of absorbance around pH 7 – Figure 2.3 (a) (Meade and Chen 1977). Bento (2003) evaluated the influence of the pore size of the filtration membranes on color. He reported that color increased 50% when the pore size on filtration changed from 0.45 μm to 1.2 μm but beyond 1.2 μm the variation is not significant. This incremental change of color measured was attributed to high molecular weight colorants that were retained by the 0.45 μm membrane. It is called "hidden color" (Godshall 2005). Bento (2003) mentions that if membranes with a pore size greater than 0.8 μm are used there will be greater interference in the color measurement due to the turbidity of the solution, but this can be reduced by fixing the Brix (concentration) of the sugar solution. Figure 2.3 (b) shows the influence of the type of membrane filter on color measurement (Kuntz 1993) and Figure 2.4 (a, b) show the effect of pore size on color and turbidity (Bento 2003). Godshall (1997) compiled the research that has been done about color analysis and the factors that affects its measurement.

ICUMSA (International Commission for Uniform Methods of Sugar Analysis), which standardizes the analytical procedures for the sugar industry, established as official the method GS1/3-7(2002) to measure color of raw sugars and colored syrups. This method defines as the parameters for color measurement: solids concentration (Brix) and cell length depending on the color level of the sugar solution, wavelength of 420 nanometers (nm), a cellulose nitrate membrane filter with a pore size of 0.45 μm and pH adjustment to 7.0 ± 0.1 (ICUMSA 2007). In the United States, the method adopted for payment purpose on the raw sugar contract, ICE 16, is the denominated "ICUMSA Method 4 (1978) Modified" which measures color for whole raw sugar on a 25 Brix sugar solution, at

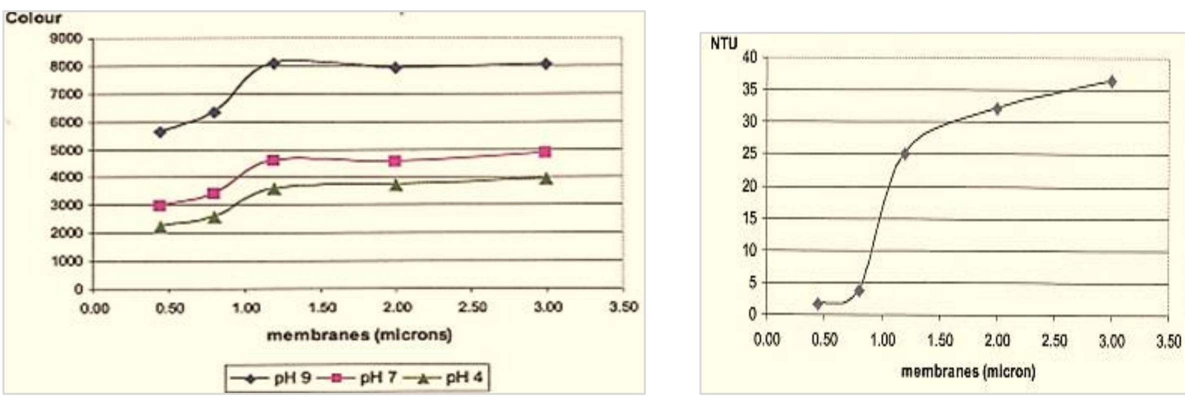
wavelengths of 420 nm and 720 nm (turbidity correction), cell length of 10 mm, glass fiber filter with a pore size of 1.2 μm and a pH adjustment to 8.5 ± 0.1. This method also includes the affination and color measurement on 50 Brix affined sugar solutions (ICE 2008). Godshall (1997) underline that even though the color methods are validated and standardized, they create controversy because of their incorporated variability and empirical foundation.



a) Attenuation index by pH and light wavelength

b) Type of membrane filter and color measurement on ICUMSA units (IU)

Figure 2.3 a) Sugar solution attenuation index variation with pH and with light wavelength (Carpenter and Deitz 1963), b) Color of sugar solution color dependence on type of membrane filter with same pore size (0.45 μm) for high color (316013) and for low color (316355) granulated white sugar (Kuntz 1993)



a) Color (IU) measurement by pore size membrane filter and by solution pH

b) Turbidity on nephelometric units (NTU) and pore size of the membrane filter

Figure 2.4 a) Color variation with pH and membrane pore size and, b) Turbidity variation with membrane pore size (Bento 2003). Courtesy of L.S. Bento

Other specialized methods and analytical instruments have been used to identify the type and/or the molecular weight of color compounds or to evaluate compositional variation due to color formation or removal. Some reported parameters and methods, are (Lionnet 1986; Godshall 1997):

- total phenols have been measured using the Foulin-Ciocalteu method (ppm cafeic acid on cane),
- amino-nitrogen compound by ninhydrin method (ppm aspartic acid in cane),

- color and color precursors by UV (ultraviolet) optical absorbance < 300 nm,
- iron-phenol complexes by addition of ferric chloride or chelating agents such as phosphoric and citric acid
- color in the crystal by the ratio of affined color/whole color
- type of colorant by Indicator value – IV = ratio of color-pH 9.0 / color-pH 4.0 (Table 2.3)

Table 2.3 Indicator values according to type of colorant (Clarke 1997)

Colorant type	Indicator Value (IV)
Melanoidins	1.0-1.2
Caramel	1.0-1.5
Hexose Alkaline degradation Products HADP	1.5-3.0
Phenolics and flavonoids	5.0-14

Gel permeation chromatography – GPC (ultraviolet, refractive index, Evaporative light Scattering detectors), ultrafiltration – UF, Millipore membrane filtration, dialysis, fluorescence spectroscopy and also gas chromatography-mass spectrometry – GC-MS have also been used to determine molecular weight and to identify colorants (Godshall 1997; Bento 2003; Bourzutschky 2005).

2.4. Controlling Color in Cane Sugar Manufacturing

A pure sucrose crystal is colorless (to blue), however the color seen in both raw and white sugar is due to differing concentrations of colored material trapped inside the crystal, bonded to crystal surface or part of the non-sucrose components of the external syrup layer. Observed sugar color is the result of a complex mixture of colorant compounds, with different molecular weights and chemical structures, which may be attached to other compounds, such as polysaccharides. The measurement of sugar color and ways of removal or prevention has been a topic of research for more than one hundred years (Honig 1963; Meade and Chen 1977; Smith, Paton et al. 1981; Hugot 1986; Chen and Chou 1993; van der Poel, Schiweck et al. 1998; Godshall 2005; Rein 2007; Bento 2008).

2.4.1. Sugar Manufacturing Process – Color Prevention and Formation

The manufacture of raw sugar from sugarcane starts with cane harvesting and delivery to the factory. At the factory, the juice is extracted from the sugarcane stalk by milling or diffusion. The extracted juice is clarified (large removal of non-sucrose matter – impurities) and then the clarified juice is concentrated (syrup) by evaporation under vacuum. The syrup is pumped to the boiling house, where by several crystallization stages the bulk of the sucrose is crystallized and then removed by centrifugation from the exhausted mother liquor (molasses). In this sugar making process, part of the colorants comes from the sugarcane plant but some are formed in processing from color precursors like reducing sugars (glucose and fructose), amino acids, enzymes (polyphenol oxidases), cell wall polysaccharides and iron compounds (Clarke 1997; Bento 2003). This color formation is favored by process conditions (Temperature, pH and Brix), and the amount of amino acids present and it can be catalyzed by some components in the ash (iron, copper), by intermediate reaction compounds like 5-hydroxy-2-methyl furfural – HMF or also by some of the colorants produced by the color formation reactions (Paton and McCowage 1987; Paton 1992; Clarke 1997; Mersad, Lewandowski et al. 2000). There are numerous factors that influence the final color in raw sugar, ranging from the variety and freshness of the cane processed to process conditions such as pH and temperature and by material recirculation (Chen and Chou 1993). Figure 2.5 illustrates how sugarcane raw juice color increases depending on the type of "trash" delivered with the sugarcane stalks and with sugarcane varieties. According to Paton (1992), flavones (flavonoids –natural colorants in sugarcane) contribute with 20 – 30% to color in the sugar crystal while high molecular weight colorants formed in the process (e.g. melanoidins and caramels) contribute 70% approximately. Clarke (1997) pointed out that there are two rules to keep color down in sugar: fast removal of impurities (non-sucrose components) and reduced recirculation.

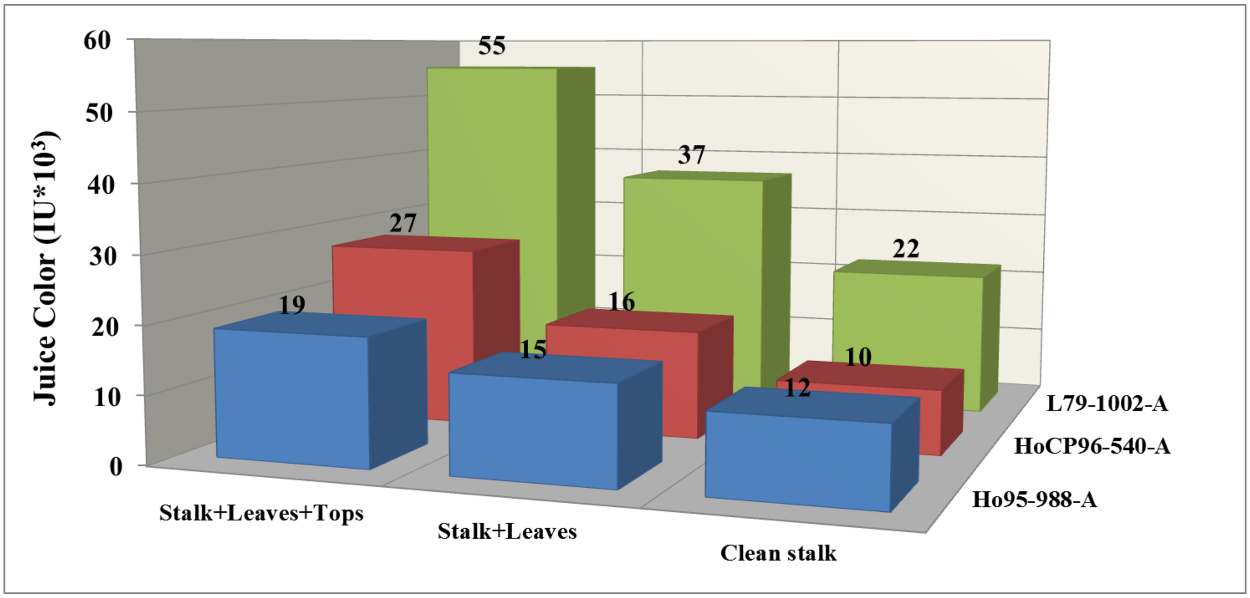


Figure 2.5 Color measurements on raw juice extracted from clean stalk, stalk with leaves and stalk with leaves and tops, for two commercial sugarcane varieties Ho95-988-A and HoCP96-540-A and for an energy-cane variety L79-1002-A. Color values taken from Richard, McKee et al. (2010)

Table 2.4 adapted from Bento (2008), compiles the information given by several researchers about process stages, the colorants formed and factors that prevent or favor color formation, including additional information and the sugar storage as other process stage. Preventive actions on one stage may reduce color formation in the next stage(s). It has to be emphasized that manufacturing practices to prevent color formation will drive the ultimate goal of the raw sugar factory, 'Sugar Recovery' (Clarke 1997).

2.4.2. Color Removal in Raw Sugarcane Manufacturing

From the 1980's the trend of refineries around the world has been to transfer the costly decolorization process to the raw sugar factory, up to the level of requesting VLC (very low color) sugar (Bourzutschky 2005). Refineries needs and price premiums, internal market demand for direct-consumption or industrial sugar and, competition among producer countries have forced sugarcane factories, mostly in countries other than United States, to change to a more energy intensive and costly boiling scheme and now they are evaluating one or a combination of technologies for color removal (Bourzutschky 2005; Madho and Davis 2008). Godshall (2005) mentions that the clarification stage removes approximately 2/3 of the polysaccharide-colorant complex (from enzymatic reactions) but the other 1/3 has to be removed by other color removal techniques. Color removal technologies can be applied from extraction through the last stage of the raw sugar crystallization process. Many of the color removal technologies that have been successful at the sugar refinery or in the sugar beet industry cannot be implemented at the sugarcane raw factory because of the higher load and different composition of the impurities and colorants in the sugarcane juice (Madsen 2009). The choice and justification of any color removal technology(s) has to be assessed considering productivity aspects such as (Clarke 1999):

- Type or group of colorants to be removed (e.g. molecular weight, polarity)
- Target level of color improvement (e.g. low color, very high pol VHP, very low color VLC)
- Constraints due to volume and/or to physical chemical composition of process streams (e.g. equipment size, dosage, scaling compounds, input and output flow rates)
- Capital investment and maintenance cost (e.g. return on investment – ROI, time)
- Operability and labor requirements (e.g. easy automation, easy integration, low labor)
- Robustness of technology to keep the target quality (capability)
- Short and long-term productivity impact (e.g. sugar recovery)
- Environmental, safety and health considerations (e.g. wastes, emissions, hazards and food regulations).

Table 2.4 Sugar manufacturing process stages and color source, formation and prevention (Smith, Paton et al. 1981; Paton 1992; Clarke 1997; Godshall 1997; van der Poel, Schiweck et al. 1998; Bourzutschky 2005; Godshall 2005; Bento 2008)

	SUGARCANE →	EXTRACTION →	CLARIFICATION →	EVAPORATION →	CRYSTALLIZATION →	STORAGE
Process Products	- Sugarcane Stalks	- Mixed Juice - Bagasse	- Clarified Juice - Filtrate Juice - Filter Cake	- Syrup	- Massecuites - Molasses - Magmas and Grain - Remelt - Raw Sugar	- Raw Sugar
Process conditions	- Weather (Temperature and moisture) - Cut-crushing time: 2 – 60 hours	- Low Brix (10-20) - Low pH (5-6) - Medium Temp. (50-70C)	- Low Brix (10-16) - High pH (7-9) - High Temp. (100-115C) - Residence time (0.5 – 3 hours)	- High Brix (60-70) - Medium pH (6-7) - High Temp. (65-100C)	- High Brix (60-98) - Medium pH (6-7) - Medium Temp. (60-70C) - Residence time (2 – 40 hours)	- Temp - Rel. Humidity - Time: days, weeks, months
Cause of color formation	- Excess of nitrogen and salts in the soil - Reducing sugars and polysaccharides (maturity and variety of sugarcane) - Cut-crush time - Burning - Leaves and tops – trash - Weather	- Lack of cleanness - Long or frequent stop times - Death zones	- Excess of Lime salts (high pH) - High retention time, poor circulation - Long or frequent stop times - Overheating - Mud recirculation with filtrate juice	- Death zones - Long or frequent stop times - Overheating - Low Brix (increases strikes time on crystallization)	- Temperature and seeding control - Death zones - Long or frequent stop times - Overheating - Crystallizers cooling profile - Non-sucrose recirculation	- Molasses layer (color, invert sugars, amino acids, ash) - Initial high sugar temperature > 45 °C - pH < 7 - Long storage time - Safety factor > 0.25
Formed colorants	- CWP (Cell Wall Polysaccharides) - Flavonoids - Phenolic acids - Precursors (glucose, fructose, amino acids, enzymes and colorants attached to CWP)	- Enzymatic Browning Products	- Hexoses alkaline degradation products HADP - Melanoidins - Caramels - NOTE: 60-70% HMW natural color removal	- Melanoidins - Caramels NOTE: Reducing sugars, temperature and time are the factor for color formation	- Melanoidins - Caramels NOTE: Brix, temperature, time and amount of Amino acids and reducing sugars are the factors for color formation	- Melanoidins
Prevention of color formation	- Sugarcane varieties - Soil Fertilization - Reduce cut-crush time - Reduce yard storage time - Reduce Trash - Eliminate cane burning	- Sanitation and cleaning - Steady State operation (no stops) - Reduce death zones - Reduce bagacillo in juice - Control formation of reducing sugars	- pH control - Type, preparation and dosage of flocculant - Phosphate content - Proper flashing - Clarifier design - Steady state operation - Mud filtration - Temperature control on heat exchangers (flashing)	- Vacuum and steam control (Temperature profile) - Evaporator level - Evaporator design - circulation and death zones - Evaporator cleaning - heat transfer coefficient (retention time) and Brix profile - Steady state operation	- Vacuum control - Pan design (circulation and death zones) - Pan cleaning - heat transfer coefficient - Steaming between strikes (batch pans) - Crystal content - Good centrifugal separation - Reduce recirculation - Surfactants	- Safety Factor <0.25 - Sugar Temp: < 45 °C (spread sugar - slinger) - Reduce initial color - Reduce ash and reducing sugars - Neutral pH (add sodium Carbonate-sugar) - Low storage Temp - Not Temp or RH fluctuation

Color removal methods that can be applied in the sugar industry can be separated into two groups: addition of decolorization agents (inhibition of color precursors and oxidation of colorants) and liquid-solid separation treatments (coagulation and flocculation, precipitation, flotation, filtration, membrane filtration, resin absorption, ion exchange, crystallization and centrifugation). Reducing the input of polyphenols and cell wall polysaccharides (color precursors) by elimination of trash with the cane (e.g. dry cleaning) is an indirect approach to color removal prior to juice extraction (Clarke 1997). After juice extraction the approach is the inhibition or complete elimination of color precursors before or on clarification (Madsen 2009). However, the sulfites and bisulfites (inhibitors) that are effectively used in other countries for this purpose are restricted by the US food regulation. Hydrogen peroxide (oxidant) to eliminate color in juice clarification or in syrup increases the costs of sugar production because of the amount required; although there were some promising trials for application of hydrogen peroxide in the wash water at the high grade centrifuges (Saska 2007; Madho and Davis 2008). Other decolorization agents have to be used, together with other separation process because the residues of the chemicals or the decolorization reaction products can affect the crystallization process or get trapped inside the crystal (Clarke 1997). Separation processes such as membrane filtration, resin absorption and ion exchange are affected by the load of impurities and by compounds present in the juice that make it more difficult or expensive to implement (Rein 2008; Madsen 2009).

Madho and Davis (2008) and Jullienne (1989) approached color removal according to the distribution of color in the raw sugar crystal. About 90% of the whole color, in the case of VHP sugar, is equally distributed between the layers of crystal built around the seed or nucleus and the mother liquor surrounding the crystal, and the other 10% of the color is added by the seed. Adapting this information to reduce the color of raw sugar from 3,000 to 1,500 CU (actual requirement), useful strategies or combinations would be (Jullienne 1989; Madho and Davis 2008):

I- Avoid seed grown on low purity and high color molasses
II- Improve quality of the mother liquor for crystallization of raw sugar
III- Improve the procedure for sugar crystallization and recovery
IV- Remove the mother liquor layer around the crystal or reduce color of this mother liquor

Table 2.5 summarizes color removal treatments that have been successfully implemented at the factory level and/or tested, presenting the advantages and disadvantages. Although these treatments are proposed for the quality improvement of a higher polarization sugar – VHP (1,000 – 2,000 IU color range), higher color reduction can also be obtained for raw sugar produced by a three-boiling scheme, which is the scenario for this study.

Since the 90's the Australian sugar industry has faced the demand to produce a higher purity - lower color raw sugar. There are several publications about the three-boiling scheme and alternative boiling changes on the production of a higher quality raw sugar. By means of boiling house models and factory trials these researchers evaluated process throughputs; pan, centrifuges and tanks capacity; and the expected costs and profits to determine the most feasible alternatives (Smith, Paton et al. 1981; Wright 1989; Wright, Broadfoot et al. 1995; Wright 1996; Broadfoot and Pennisi 2001; Broadfoot and Pennisi 2002; Broadfoot 2006). The alternatives considered for color reduction in Australia have been to increase wash on the high grade batch centrifuges and variations of boiling schemes from CBA scheme (same double magma boiling scheme) to schemes requiring complete melting of A and B sugars with double purge of C-magma and recrystallization (Wright 1996; Broadfoot 2006). Models which evaluate boiling house capacity requirements depend on the purity of the syrup, which changes from low purity at the start of a sugar crop season, to high purity at the middle and then to low purity at the end of the crop season (Broadfoot 2006). With low syrup (or juice) purity the required capacity (pans and centrifuges) of the low grade (C strike) and intermediate (B strike) crystallization stages are higher because of a higher input of impurities. In the case of high syrup (or juice) purity, the required capacity of the high grade stage (A strike) is higher (Broadfoot and Pennisi 2001; Broadfoot and Pennisi 2002; Broadfoot 2006). Overall, the models show that an incrementally rise of the purity of raw sugar requires increases in the required capacity of pans and centrifuges at the different stages of the crystallization process, since fewer impurities are leaving with the sugar (Wright 1989).

Table 2.5 Treatments for color removal in raw sugar and some observed advantages and disadvantages

Treatment	Strategies	Advantages	Disadvantages	References
Specialized Chemicals or flocculants on juice clarification	II	**- Bentonite** 0.2% juice reduces turbidity on 10-15% **- Hydrogen Peroxide** 1000-7500 ppm Syrup, color removal ~5-16% **- Ozone** 4500 ppm/Bx syrup, color removal 72%. 12500 ppm/Bx mixed juice, color removal 40% **Polyaluminium Chloride**: 90 ppm/Bx mixed juice, 19% color removal	**-Bentonite-** High costs **-Hydrogen Peroxide**: High costs **-Ozone**: 20-24 kW-h/kg – High energy consumption Chemicals increase costs and have environmental, safety or health issues	(Madho and Davis 2008)
Syrup Flotation	II	-5-10% Color Removal -80-95% Turbidity Reduction -25% Viscosity reduction on final molasses -24% Color Raw Sugar Reduction	-Flocculant residues on sugar (when high dosage) -High capital and operational costs -Increase sugar loses in filter cake	(Madho and Davis 2008) (Rein 2007)
Double-magma boiling scheme	II & III	-Raw sugar only from the 1st strike. Better quality -Massecuites purities profile help to achieve molasses exhaustion	-Higher energy compared to 3-boiling scheme -B sugar nucleus is C sugar -Increased pan and centrifugal requirements -High capital costs	(Birkett and Schaffer 1978) (Rein 2007)
Boiling scheme with melting of C and/or B magmas	I, II & III	-Better sugar quality compared to double magma	-Higher energy and equipment capacity requirements than double magma -US RS price premiums are not proportional with the quality improvement	(Madho and Davis 2008)
Increase washing in centrifuges	IV	-Reduce sugar color	1% wash increase, sugar dilution increases 1.5%	(Madho and Davis 2008)
Hydrogen peroxide on wash water in centrifuges	IV	-100-500 ppm/sugar color reduction of 20-30% -No residues in sugar	-Reduces pH of sugar at high concentration (storage) -Destruction of reducing sugars -Corrosive and fire hazard (oxidizer)	(Saska 2007) (Madho and Davis 2008)
Double purge (centrifugation) of C magma	II & IV	-Lower color (50-70%) and lower conductivity ash in raw sugar -Less molasses layer attached and surrounded to the C-nucleus -Less final molasses to the 1st strike -Shorter cycles and less wash on A batch centrifuges	-Increase C massecuite volume -The cost for an additional centrifuge only justified by significant color reduction and sugar recovery	(Meade and Chen 1977) (Madho and Davis 2008) (Bourzutschky 2005) (Jullienne 1989) (Hugot 1986) (Chen and Chou 1993) (Chou 2000)

In summary, changes in the boiling or crystallization scheme and the integration of double purge of C magma to the actual crystallization process are among feasible color removal technologies that can offer significant color reduction in raw sugar for factories in Louisiana which utilize a three-boiling crystallization scheme.

2.5. Crystallization Schemes

The goals of the raw sugar factory's boiling house are the production of sugar of the required quality, the efficient use of energy and the recovery of the practically obtainable maximum amount of sugar from the syrup (Meade and Chen 1977). In contrast, the primary goal of a sugar refinery's boiling house is the maximum elimination of color (Chen and Chou 1993; van der Poel, Schiweck et al. 1998; Chou 2000). Overall, a boiling house contains a more or less flexible arrangement of crystallization stages (boiling scheme or boiling system), selected depending on syrup purity (sucrose % soluble solids), desired sugar quality (polarization and color), available energy and available equipment (van der Poel, Schiweck et al. 1998). Figure 2.6 shows different boiling schemes used for the production of raw sugar. The two-boiling scheme is utilized for syrup of low purity < 82; three-boiling has been the most common boiling scheme for average syrup purity of 85 and raw sugar purity between 98.8 and 99.2; double magma or double Einwurf or CBA (Australia) for sugar purity between 99.0 and 99.4 and the VHP boiling scheme for purities > 99.6 (Meade and Chen 1977; van Hengel 1983; Chen and Chou 1993; Wright 1996; van der Poel, Schiweck et al. 1998; Chou 2000; Broadfoot 2006; Rein 2007). Because of variations of sugarcane quality during or between crop seasons the boiling house has to be flexible in order to process a wide range of syrups (e.g. 80 – 92 purities) (van der Poel, Schiweck et al. 1998).

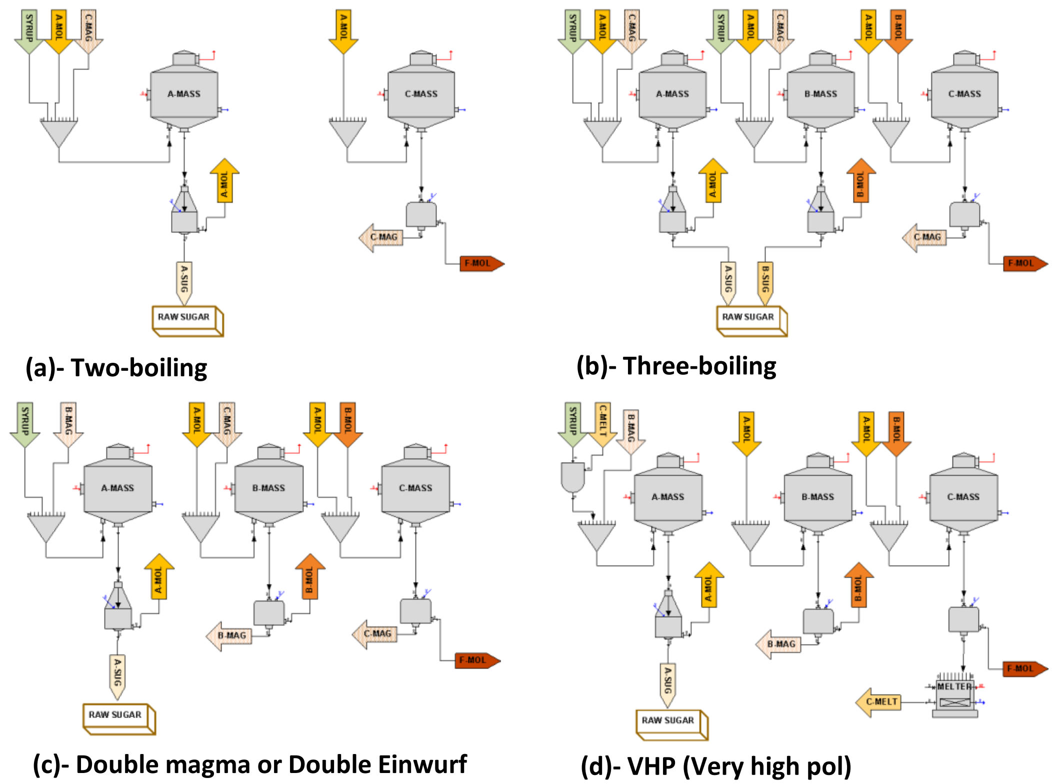


Figure 2.6 Basic boiling crystallization schemes utilized to produce raw sugar. Templates from Sugars™

As a consequence of the rheological characteristics of the materials, the sugar in the syrup has to be recovered in 2 – 4 batch or continuous crystallization stages (strikes), with descending purity, identified by numbers or capital letters (first, second and third or A, B and C strikes); same denomination is applied to the product from each strike (e.g. A, B and C massecuite) and to the streams separated on centrifuges (e.g. A, B and C sugars and molasses). Larger mean crystal sizes (0.7 – 0.9 mm) are produced at higher purity and smaller mean crystal sizes (0.30 – 0.35 mm) are produced at a lower purity (Chen and Chou 1993). Sugar and molasses are separated by centrifugation at each stage, thus the concentration of impurities on massecuites and molasses increases from stage to stage. The final strike which has the highest concentration of impurities and the lowest sugar content produces the

smallest crystals (0.28 – 0.30 mm) and requires the longest crystallization time (2 – 4 hours A and B, 5 – 9 hours C). After the last strike, crystallization by cooling (20 – 48 hours) is used to reduce the loss of sugar on the C or final molasses (molasses exhaustion). The small crystals separated after cooling crystallization can be dissolved or can be prepared (magma) to be used as crystallization nuclei (footing) for the previous stage(s). Footings, instead of nucleation, are used to obtain more homogeneous crystal size distributions, an important consideration for the separation of crystals and mother liquor in centrifuges. The amount and mean size of the crystals nucleus in footing is defined by the amount and mean size of the crystals at the end of each strike, approximately determined by the "d^3 *rule*" equation 2.1 (van der Poel, Schiweck et al. 1998). The equation states that the ratio of mass of crystals produced to the mass of crystals in the footing is equal to the cube of the ratio of the final mean crystal size produced to the mean crystal size in the footing. A failure to introduce the right amount of footing to a strike, may produce not only smaller or larger crystals but also may produce a less homogeneous crystal size distribution, affecting molasses separation on centrifuges and increasing recirculation of non-sugars – color (because of poor separation of molasses). The quality of the raw sugar strongly depends on the quality of the feed and on the quality of the footing used to grow the crystals. Recirculation of colored impurities with the footing affects the quality of the crystal seed and the crystallization medium. The achievement of the maximum crystal yield from the first to the last stage and the efficient separation of impurities on centrifuges are the best strategies to minimize recirculation (Rein 2007).

$$d_{Cr,1}^3 = \frac{d_{Cr,0}^3 \cdot m_{Cr,1}}{m_{Cr,0}} \qquad \text{(Equation 2.1)}$$

Where $d_{Cr,0}$=Mean crystal size in product
$d_{Cr,1}$=Mean crystal size in product
$m_{Cr,0}$=mass of crystals in footing
$m_{Cr,1}$=mass of crystals in product

2.6. Three-Boiling Crystallization Scheme

For raw sugar production in Louisiana, a three boiling scheme is common practice owing to sugar recovery and energy requirements (Birkett and Schaffer 1978). The primary goal of the three boiling system is to achieve a purity drop around 20 (massecuite purity - run-off molasses purity) in each stage (A, B and C) for a purity of final molasses around 30, Figure 2.7 (van der Poel, Schiweck et al. 1998). Hugot (1986) states that purity drop ranges between 18 – 20 for A strikes, 21 – 23 for B strikes and 22 – 30 for C strikes (after cooling crystallization). The three boiling system performs well when syrup purity is around 84 to 86. But, when syrup purity is higher, A molasses is recycled to the syrup (back-boiling), in order to lower the final purity of the A-massecuite to ~85. The purity of C-magma for a three-boiling scheme is ~ 85. The grain for the C strike is made by blending A and B molasses to adjust the purity to 70, required purity for fast and homogeneous crystal development. The crystal seeds are generally made milling granulated white sugar to reduce the mean size from about 20 – 40 µm to a mean size approximately of 4 – 6 µm. The sugar recovery objective, crystallizing sugar with a three boiling scheme, is easily obtained when the final raw sugar polarization is ~98, but it gets more difficult to keep the purity of the final molasses ~30 when the sugar pol is above 99 because of the additional wash on A and B centrifuges (Wright 1989). Chou (2000) highlights that the main advantages of a three-boiling scheme are that it minimizes the melting of magmas and has the lowest steam consumption compared to other three stage crystallization systems (Double magma and VHP schemes). The results for steam consumption were presented by Birkett and Schaffer (1978).

According to Wright (1996) the traditional three boiling scheme is adequate for the production of raw sugar with a crystal size ~ 800 µm and with purities between 98.8% (~ 4,000 IU color) and 99.2% (~2,000 IU color). For a raw sugar factory with a three-boiling scheme, the shift from producing high color sugar to low color sugar implies a higher amount of impurities passing from the high (A and B strikes) to the lower (C strike) crystallization stages (Wright 1989). Some of the challenges are physical limitations on the maximum crystal content for the higher purity crystallization stages (higher crystal content reduces massecuites flow) and larger impurities recirculation (more capacity requirements for the low grade station). In general, the boiling schemes to produce high pol raw sugar require additional pan and centrifuge capacity and require more steam for water evaporation (Chou 2000). A key to improving quality of raw sugar with a three boiling scheme is to increase the purity of the C-magma (lower recirculation, less color) without affecting the sucrose recovery by the centrifugation of C-massecuite (less washing, less sucrose crystals dilution) (Hessey and Manning 1949; Jullienne 1989; Chen and Chou 1993; Broadfoot, Miller et al. 1994; Chou 2000).

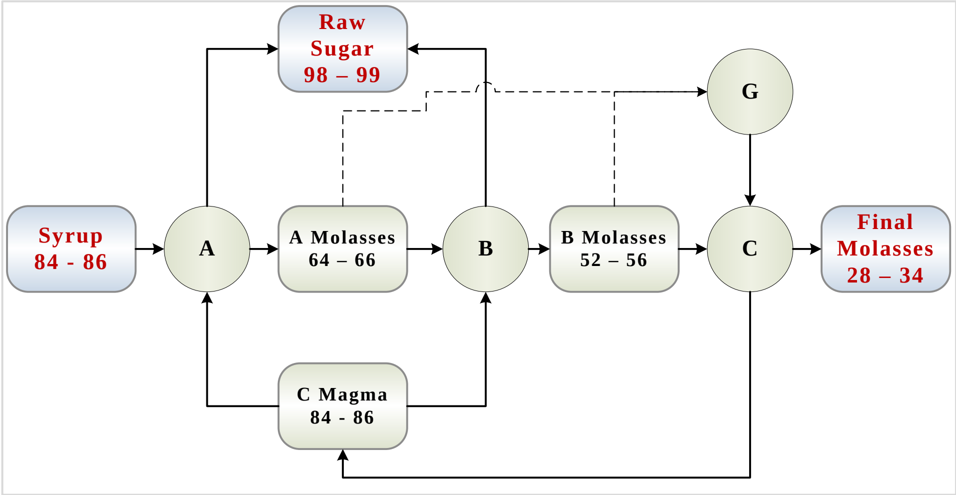


Figure 2.7 Approximated sugars and molasses purity profile for raw sugar production with a three-boiling scheme (A, B, C)

The link between C-magma quality and raw sugar quality has been discussed by some researchers (Baikow 1963; Stevenson 1964; Smith, Paton et al. 1981; Jullienne 1989; Broadfoot, Miller et al. 1994; Wright 1996; van der Poel, Schiweck et al. 1998). Figure 2.8 illustrates the reduction on raw sugar color index (attenuation index) with the increment in magma purity, at laboratory scale, using syrups and magmas from 3 different locations. The tests were performed in a laboratory scale vacuum pan growing C-sugar crystals to about 0.4-0.5 mm under the same pH, Brix, purity, supersaturation, time and temperature conditions. Improvements on ash content and filterability of the sugar crystals produced by a higher purity C-sugar seed were correspondingly observed during the tests (Stevenson 1964).

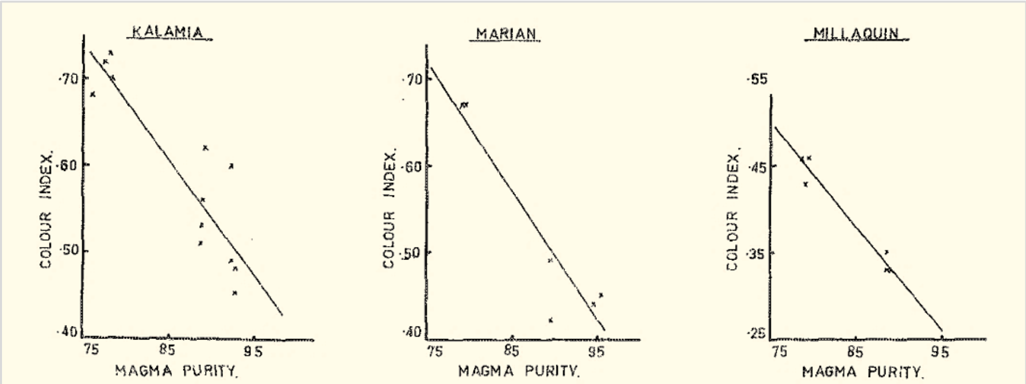


Figure 2.8 Influence of magma purity on affined color index (attenuation index) at 435 µm and pH 11 for sugar crystals produced under similar crystallization conditions at laboratory scale using syrups and magmas from three factories in Australia (Stevenson 1964)

The production of a high quality C-sugar by a single centrifugation is highly constrained by the common characteristics of the C-massecuite; high viscosity, small mean crystal size with a wide particle size distribution (CV larger than 0.3) and, low crystal content (30 – 35%). This difficulty can be understood, as continuous centrifuge requirements for an efficient separation of the crystallites are based on mean particle size, particle size distribution, gravity factor (G-factor), thickness of the crystal layer, viscosity of the massecuite, and washing conditions (Bosse 1993). A low grade centrifugation stage needs to separate the largest amount of viscous molasses (lower crystal content than A and B strikes), requiring high filtration area and the highest possible G-factor (function of revolutions per minute, diameter and layer thickness). Continuous centrifuges replaced batch centrifuges for the separation of C-sugar crystals from low grade massecuites around 1970. Continuous centrifuges allowed the production of low grade massecuites with higher Brix (higher viscosity), lower purity and lower mean crystal size reducing the purity of final

molasses (less sucrose losses) in comparison with prior years (Jullienne 1982; Jullienne 1987). However, put side by side, the purity of molasses separated by a Nutsch pressure filter and the molasses separated by the continuous centrifuges showed a rise in the purity of the molasses from these centrifuges. The purity rise on final molasses (4 points) in South Africa was evaluated considering operational variables on centrifuges like massecuite flow-rate, wash water-to-massecuite ratio, water temperature and steam addition and, massecuite properties like the mean crystal size in the C-massecuite. It was found that changes in these operational variables produced higher purity C-sugar and increased final molasses purity on centrifuges, Figure 2.9.

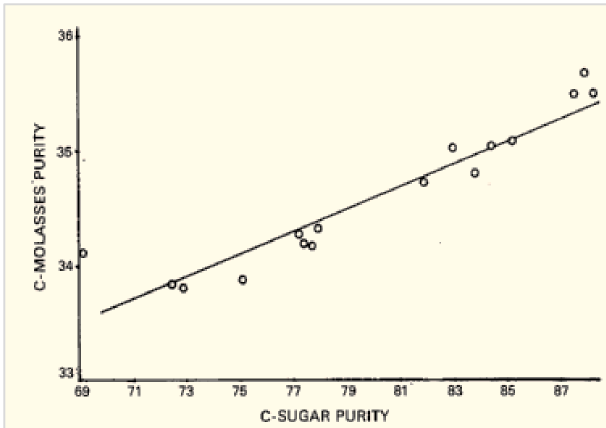
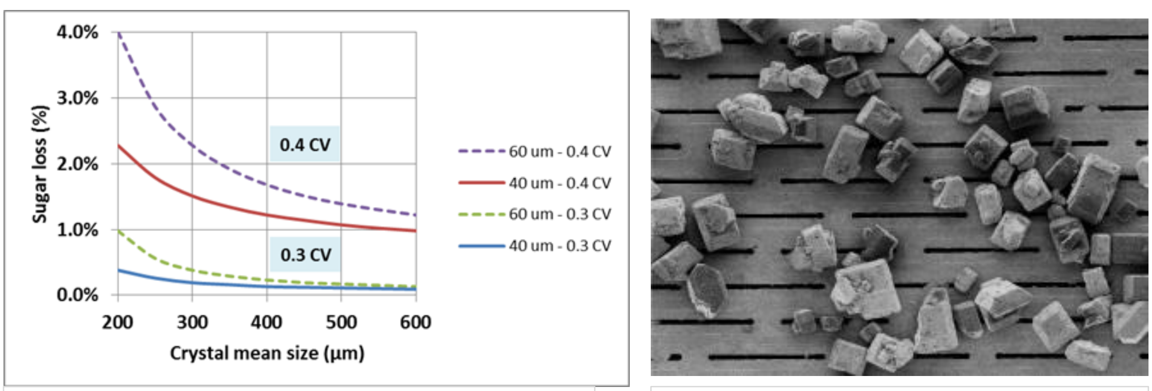


Figure 2.9 Final molasses purity versus C-sugar purity from centrifuge evaluation tests at a South African raw sugar factory (Jullienne 1982; Jullienne 1987)

Mean crystal size was also considered a probable cause of purity rise, for the 4 points of purity rise, 3 points were attributed to the passage of small crystals through the centrifuge screens. Jullienne (1985) stated that for a screen with slot width of 60 µm (popular for low grade massecuites) crystals sizes below 120 µm were considered small. It is noticed that the mean crystal size on C-massecuite reported for South Africa factories (100-130 µm) operating with a VHP boiling scheme is lower than the mean crystal size specified for a three-boiling scheme and double magma boiling scheme (300-350 µm) (Jullienne 1985; van der Poel, Schiweck et al. 1998). Grimwood, Thewlis et al. (2003) highlighted the importance of the combination (assuming Gaussian crystal size distribution) of crystal mean size and coefficient of variation (CV) in terms of sucrose (sugar) losses on C centrifuges, Figure 2.10.



a) Sugar loss (%) versus crystal mean size by slot size and coefficient of variation

b) Slots of centrifuge screens and sugar crystals

Figure 2.10 a) Maximum probable sugar losses on continuous centrifuges for slot widths of 60 and 40 µm depending on crystal mean size and for coefficient of variation (CV) of 0.3 and 0.4. (b) Screen used on continuous centrifuges (90 µm slots width and 10% open area) (Grimwood, Thewlis et al. 2003). Courtesy of Broadbent Centrifuges

Crystal losses on batch centrifuges are lower because the crystal layer is static while the liquid is passing between crystals and through the screen holes. In contrast, in continuous centrifuges the probability of sugar loss is higher as the crystal layer is moving over the screen, exposing more of the small crystals that can pass through the

slots (Chou 2000). The combination of small crystal mean size and a high coefficient of variation impacts crystal (sugar) losses and makes more difficult the elimination of mother liquor (color elimination) because of the reduction in the space between crystals. Batch centrifuges for low grade massecuites, started being displaced by continuous centrifuges around 1960; the thin-layer concept makes continuous centrifugation optimal for the separation of high viscosity molasses (van der Poel, Schiweck et al. 1998). Design and operational features of continuous centrifuges have been upgraded since the introduction of this technology to the sugar industry to correct problems (crystal breakage, crystal dilution, passing of crystals to the green liquor and high massecuite viscosity), to increase massecuite throughput and to automate operation (Kirby and Greig 1986; Bosse 1993; Henpelmann 2006; Bartsch and Geyer 2010). Figure 2.11 shows the Massecuite input, with points for addition of steam or water to improve the flow, water spray to wash the C-sugar crystals and water addition to dissolve or magmatize the C-sugar inside the machine. The molasses (syrup) are forced through the screen holes segregated from the C-sugar crystals.

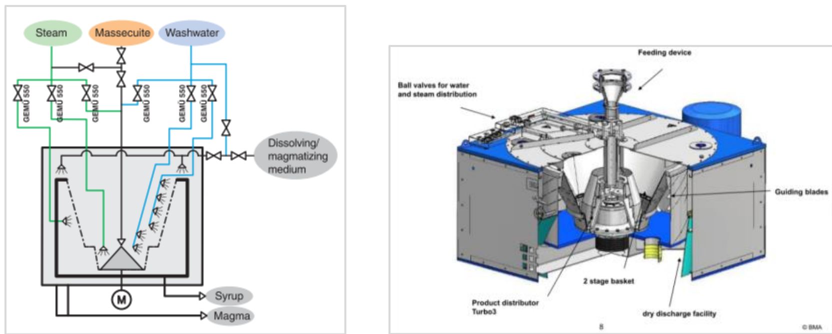


Figure 2.11 Diagram of continuous centrifuge with magma preparation (Geyer and Schmidt 2011; BMA 2014). Courtesy of BMA

2.7. Three-Boiling Crystallization Scheme with Double Purge of C-magma

Figure 2.12 is a sketch of the three-boiling scheme, adding the double purge of C-magma system and the possible points of recycle of the double purge molasses

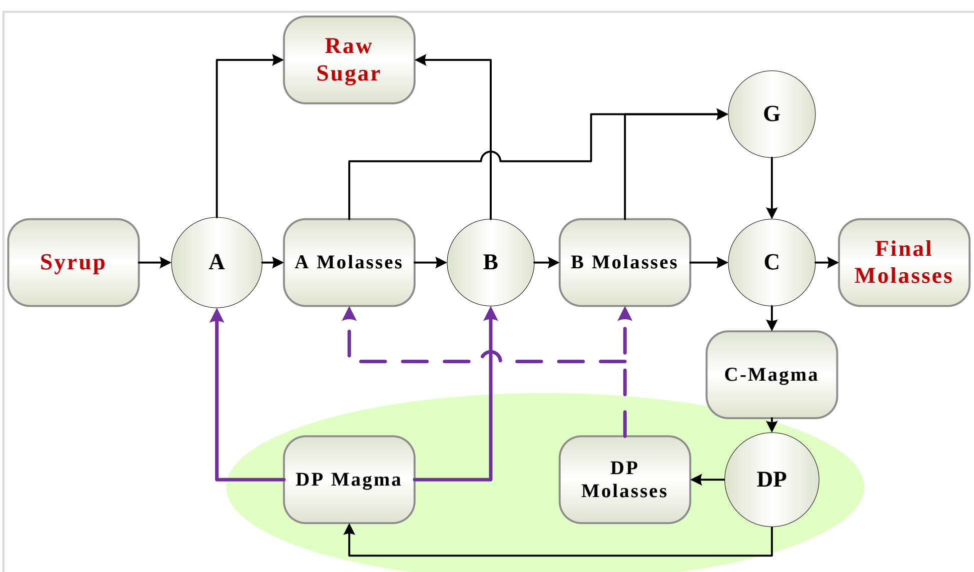


Figure 2.12 Block diagram of a three boiling crystallization scheme with a double purge (DP) system recycling double purge molasses to A or B molasses tanks. The double purge magma is the seed used to grow raw sugar

Chou (2000) describes double purge of C magma as a two stages separation system that reduces non-sucrose recirculation to the high level stage and, that helps to minimize sucrose losses on molasses by reducing washing on the first purge. Double purge of C-magma was first applied in Java (1909) and it has been tried around the world predominantly, in plantation white sugar factories and also in refineries (Geerligs 1909; Meade and Chen

1977; Hugot 1986; Chen and Chou 1993). Jullienne (1989) mentions that double purge using batch centrifuges was a common practice in South Africa until 1970. The system was gradually eliminated when South African factories changed the boiling scheme to produce higher quality sugar (very high polarization – VHP). Large amounts of small crystals in molasses from the second purge machines (crystal lost) were lost, probably a consequence of crystal breakage in the first centrifugation (Carter, Graham et al. 1970). Researchers in Australia and South Africa (Jullienne 1989; Broadfoot 2006; Madho and Davis 2008), recently proposed the implementation of double purge of C-magma for further improvements on higher quality raw sugar like VHP. Models (empirical spreadsheet and software) for color profiles and mass balance have been used to evaluate different boiling schemes and some of them have been used to evaluate the integration of double purge of C magma to particular boiling schemes estimating required equipment capacity and costs (Getaz and Bachan 1989; Smith 1990; Radford 1996; Wright 1996; Peacock 2002; Schorn, Peacock et al. 2005; Broadfoot 2006; Saska 2008).

Hugot (1986) describes the double purge of C-magma using batch centrifuges and highlights that grain size uniformity and mother liquor viscosity are conditions required for a good performance of the second centrifuges. At the first purge, the C-sugar is separated from the molasses with "little or no water" (Meade and Chen 1977). Double purge molasses will be mixed with B molasses or it will be stored independently, but it is not recommended that the second wash be recirculated to a higher crystallization stage. The first C-sugar is discharged into a mixer where it is converted in magma by mingling the sugar with higher purity molasses, clarified juice or water. Diluted B molasses (70 °Brix) at 70 °C (158 °F) is suggested for the preparation of the first magma. The resulting first magma is again centrifuged washing with water and steam. The second C sugar is mingled with syrup (30% syrup for 70% C sugar) and pumped to the pan floor. First magma purity ranges from 80 to 85 and Second magma purity ranges from 88 to 92 are recommended for the production of high polarization raw sugar (Hugot 1986). Milner and Houstonk (1997) recommends for the first purge a standard centrifuge (30° basket) equipped with magma mixing and with open bottom, operating at no more than 2000 G (G-factor) to reduce crystal breakage and that for the second purge, centrifuges (25 ° basket) can operate at 1200 to 1600 G. Approximately one to three is the capacity ratio between second purge to first purge centrifuges because of the higher capacity of the second centrifugation stage (easier task). There exist centrifuges built with two stages (double centrifuges) but, two individual centrifuges offer more flexibility to change capacity to optimize results, and the maintenance of each separation stage is easier (Milner and Houstonk 1997). In addition, mixing and scrubbing of crystals before the second centrifugation is recommended to release the molasses layer attached to the crystal (Meade and Chen 1977; Milner and Houstonk 1997). Changes in the double purge system such as first magma purity, magma preparation, wash molasses purity and second magma purity have effects on non-sugars recirculation, raw sugar quality and required capacity of low grade pans (Hugot 1986; Perez, Moreno et al. 1997). It was found in Hawaii that if the purity of the first magma drops from 75 to 65, the required capacity of C pans increases by 30% (Hugot 1986). Using second purge wash molasses instead of water to prepare the first magma resulted in more required capacity for the whole double purge system (magma preparation and double purge centrifuge), and produced a second magma with higher color and higher turbidity (Perez, Moreno et al. 1997). Milner and Houstonk (1997) state that the use of cold water reduced the color on affined raw sugar. Makina (2000) states that in the case of sugar beet production, a single purge can reduce color by 94 – 96% while with double purge the color reduction is about 96 – 98 % for low grade massecuites. Perez, Moreno et al. (1997) compared the use of double purge molasses and water only on magma preparation, for the production of plantation white sugar with sulphitation and a VHP boiling scheme (B magma as footing for A strike and melting of C sugar). It was found that the quality of white sugar in terms of color and turbidity were significantly different when comparing first magma preparation with 2nd molasses and with water (Table 2.6). Producing ~19 m^3 / hour (667 ft^3 / hour) of C-massecuite, the used equipment and capacities were: 18 m^3 (634 ft^3) mixer; three continuous centrifuges (one standby) with capacity of 6.8 m^3 / hour-centrifuge (240 ft^3 / hour-centrifuge); two magma pumps (one standby) with capacity of 16.8 m^3 / hour-pump (594 ft^3 / hour-pump, 74 gpm) and; two molasses pumps (one standby) with capacity of 34.2 m^3 / hour-pump (594 ft^3 / hour-pump, 150 gpm).

Table 2.6 Double purge parameters and sugar quality preparing magma with double purge (DP) molasses and with water (Perez, Moreno et al. 1997)

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

A double purge technology can be implemented in factories operating with three-boiling system to improve raw, white and refined sugar quality. It seems that the double centrifugation system to improve raw sugar quality lost popularity around 1960. New boiling systems alternatives like double magma and VHP busted by local or world sugar prices and the introduction of continuous centrifuges discouraged the propagation of double purge for raw sugar production. For instance, the costs for capacity expansion and required new equipment to implement a double magma system may be as high as 18 cents per pound of raw sugar (Arias 1993) while the actual sugar price is about 25 cents per pound (Haley 2015). Double purge of C-magma is important for those raw sugar factories in Louisiana that must improve raw sugar quality, but cannot afford high capital investments while the price of raw sugar is near to the profit margin.

2.8. Boiling House Optimization

At the sugar cane industry most optimization strategies have focused on the agricultural (varieties, harvesting, transportation, etc.) and on the energy efficiency points of production (Singh, Riley et al. 1997; Ensinas and Nebra 2006; Paiva and Morabito 2009; Morandin, Toffolo et al. 2011). In the boiling house the optimization research has been centered on pan scheduling, crystallization and centrifuge operation (Jullienne 1982; Steindl, Broadfoot et al. 2001; van Wissen, Smeets et al. 2003; Galvanauskas, Georgieva et al. 2006). Spreadsheets and software have been applied to create sugar process models, simulating and evaluating process responses to changes in plant configuration, taking also into consideration the changes in the quality of the input or the quality of the sugar (Weiss 1989; Weiss 1994; Stolz and Weiss 1997; Thompson 1997; Broadfoot 1999; Weiss 1999; Broadfoot 2001; Broadfoot and Pennisi 2001; Steindl, Broadfoot et al. 2001; Broadfoot and Pennisi 2002; Peacock 2002; Schorn, Peacock et al. 2005; Broadfoot 2006; Saska 2008; Schellen 2014).

Clarke (1999) evaluated the concepts that need to be considered for the implementation and integration of new technologies in the sugar manufacturing process. He stated that on the process integration of a new technology, productivity improvement has to be paired with quality improvement and, this has to be approached as a multi-factor problem considering the efficient use of resources like equipment and energy, costs and adverse conditions. Optimization techniques have been applied to isolated operation units (e.g. centrifuges and pans) or to scheduled batch pans. It is recognized that evaluation and integration of new technologies needs to combine multiple factors with respect to productivity and quality to guarantee its success and sustainability in the industry. Galvanauskas, Georgieva et al. (2006) used a dynamic model with multiple response optimization to determine optimal controlled inputs for batch crystallization. Multiple response or criteria optimization strategies have also been used to evaluate energy crops for biogas production (Vindiš, Stajnko et al. 2012), selection of materials for pipes used in the sugar industry (Darji and Rao 2014). The multiple response optimization strategy proposed on this research applying computer simulations, metamodeling and desirability functions to optimize the integration of the double purge system is novel and will offer a new alternative strategy on the implementation of new technology at the sugar industry.

CHAPTER 3 DOUBLE PURGE OF C-MAGMA – FACTORY SCALE IMPLEMENTATION

3.1. Introduction

In Louisiana and other countries, raw sugar factories operating with a three-boiling crystallization scheme must reduce color and, in general, improve raw sugar quality to increase profits and satisfy quality requirements from refineries (Rein, Bento et al. 2006; Madho and Davis 2008). Raw sugar color reductions to ~1400 color units – CU are required both in order to meet the specifications of local Louisiana refineries and to be competitive with respect to the world raw sugar market. Any new technology must take into account energy efficiency, factory performance and production yield prior to application.

Double purge of C-magma is a technology that can be implemented by introducing a small number of changes to the basic three-boiling scheme. It requires adapting one of the continuous centrifuges that is used to separate the C sugar crystals to a different purpose. Figure 3.1 illustrates the sequence and some equipment that can be used to implement double purge of C-magma.

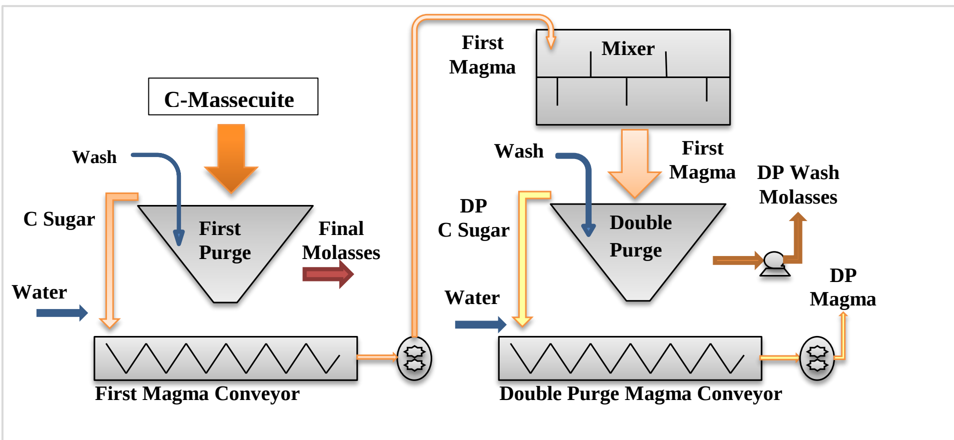


Figure 3.1 Double purge system equipment: first magma mixer, double purge centrifuge, double purge magma conveyor and pumps for double purge materials molasses and magma. Product parameters: First magma purity, second magma purity and point of recycle of molasses

Installing this system only requires the addition of piping, pumps, a magma preparation system (mixer) and adapting a C-centrifuge to double purge the C-magma. Capacities and sizes of equipment and pipes were estimated by mass balance using a software created specifically for the sugar industry (Sugars^{TM} 2014). The double purge of C-magma was implemented and applied by 3 Louisiana raw sugar factories in 2012, 2013 and 2014 sugarcane harvesting seasons.

3.2. Materials and Methods

The introduction of the double purge system at factory scale was conducted in stages: preliminary modelling, factory trials and full implementation, including automation. Routine sampling, chemical analysis, statistical analysis and daily graphical evaluation of trends (product parameters and factory performance indices) were used to supply continuous feedback to factory engineers and managers and to guarantee the successful integration of the second centrifugation of C-magma. Initial trials were conducted in 2011 at Sterling Sugars Inc. – ST, and then the system was fully implemented by Lula Sugar Factory – LU (2012), Westfield Sugar Factory – WF (2013) and Cora-Texas Mfg. Co., Inc. – CT (2013).

The quality of the cane and the grinding rate fluctuate during the harvest season. These changes affect the quality of the input (syrup purity, color and solids rate) to the boiling house requiring the adjustment of the double purge product parameters such that the raw sugar produced will meet refinery whole color standards, and maintain or improve sucrose recovery and throughput. To achieve these goals the specific objectives for this stage – implementation were:

1. Create a three boiling system model (using Sugars™), attaching a second centrifugation step for C-magma. Using the preliminary model, evaluate boiling house responses to an integration of double purge of C-magma and estimate flow-rates and required equipment capacity for implementation.
2. Quantify, from factory sampling analysis and data collection, the improvement on raw sugar color relative to the input and outputs of the second centrifugation stage and; evaluate purity and color profiles at the boiling house and the effects on quality and boiling house performance of the recycle point of the double purge molasses
3. Adjust the preliminary model based on results (sampling and factory data).
4. Establish variability ranges on boiling house parameters and correlations for future simulations and optimization.

3.2.1. Boiling House Sampling

Sampling was conducted during the 2013 Louisiana sugarcane harvesting season at Lula Sugar Factory, Westfield Sugar Factory and Cora-Texas Mfg. CO., Inc. Sampling points and sampling frequency, specifically for the double purge system, were determined with the objectives of process control and research. The factory laboratories added samples of double purge magma and double purge molasses to their routine sampling (each 3 hours) and also collected weekly composite samples of syrup, molasses and raw sugar. Two sets (2013 sugarcane harvesting season) of boiling house grab samples for purity and color (from syrup to final molasses) analysis were taken at each factory. Table 3.1 and Figure 3.2 detail the routine sampling location and frequency.

Table 3.1 List of factory routine sampling and composite sampling for industrial control purpose and evaluation of boiling house performance

Point	Sample Description	Frequency (hours)	Composite (hours)	Composite (days)
1	Evaporators syrup	1	4	7
2	A-massecuite	batch	NA	NA
3	Raw Sugar	1	3 and 24	7
4	A-molasses tank	4	NA	NA
5	B-massecuite	batch	NA	NA
6	B-molasses tank	4	NA	NA
7	Grain (C-seed)	batch	NA	NA
8	C-massecuite - pan	batch	NA	NA
9	Final molasses	3	NA	7
10	First magma	3	NA	NA
11	Double purge magma	3	NA	NA
12	Double purge molasses	3	NA	NA

3.2.2. Analytical Procedures

Where possible, analytical methods were according to the ICUMSA methodology (ICUMSA 2007). The composite raw sugar samples were analyzed for color according to a refinery protocol defined by the Sugar No. 16 contract (ICE 2008). The most relevant raw sugar analyses were: polarization (ICUMSA GS1/2/3-2), moisture (ICUMSA GS2/1/3/9-15), conductivity ash (ICUMSA GS1/1/3/4/7/8-13), whole and affined raw color (modified ICUMSA 4 – 1978), dextran (MAU), and starch (ICUMSA GS1-17 - 2005). The instruments used in the analytical procedures are the following:

- Precision scale: Ohaus Adventurer™ AV2102, 2100 x 0.01 (g)
- Analytical scale: Ohaus Adventurer™ AX324, 320 x 0.0001 (g)
- Refractometer: Bellingham and Stanley RFM340, accuracy 0.03 °Brix
- Polarimeter: Rudolph Research Autopol 880 saccharimeter, accuracy 0.01 °Z, wavelengths: 589 and 880 nm
- Conductivity meter: Oakton CON 510, range: 0 µS to 200 mS, accuracy: ±1% full-scale
- Spectrophotometer: GENESYS™ 10S Vis, wavelength range: 325 to 1100nm

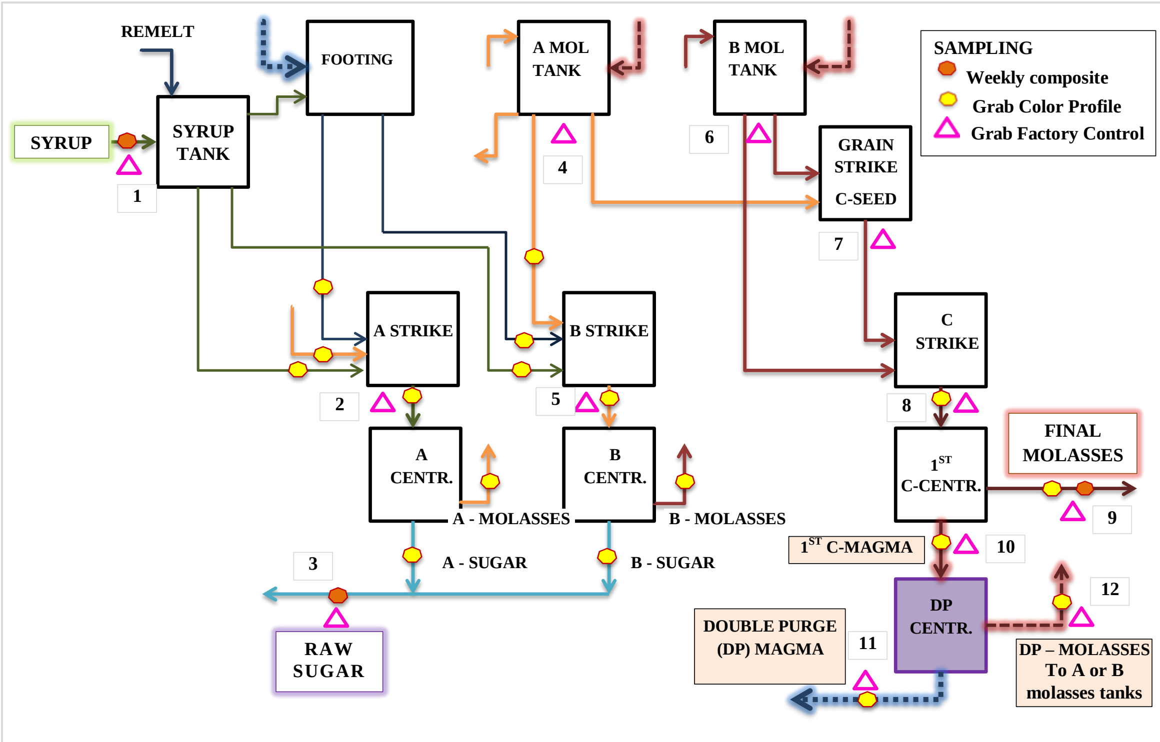


Figure 3.2 Boiling house block diagram including sampling points and sampling type (weekly composite, grab for color profile or grab for factory control by routine analysis)

3.2.2.1. Sample Preparation

Table 3.2 shows the dilutions, using deionized – DI water, of boiling house process streams for analysis by polarization, °Brix, conductivity ash and color of the sugar solution. Samples were analyzed immediately or they were preserved (in a refrigerator or a freezer) to avoid sucrose destruction and additional color formation.

Table 3.2 Sample weights in grams for analysis of refractometric dry substance – DS (°Brix), polarization, conductivity ash and color in solution

	Refractometric DS and Polarization		Color (sample or dilution)	
Sample	**Sample, w_s**	**Water, w_w**	**Sample, $w_{dil\text{-}s}$**	**Water, w_w**
Syrup (2:1)	80.0	40.0	5.0	45.0
Massecuites (1:1)	50.0	50.0	2.5	50.0
Magmas (1:1)	50.0	50.0	2.5	50.0
Molasses (1:1)	50.0	50.0	1.0	50.0
Sugars A, B and Raw	N.A.	N.A.	25.0	75.0
Affined Raw Sugar	N.A.	N.A.	50.0	50.0

Dilution factor calculated using equation 3.1

$$\text{Dilution Factor}=\text{DF}=\frac{w_s+w_w}{w_s} \qquad \text{(Equation 3.1)}$$

3.2.2.2. Refractometric Dry Substance (RDS % or Brix)

By definition, dry substance is the mass of solids remaining after the water is evaporated using an oven drying method. The refractive index is related to the sucrose concentration but also is influence by non-sucrose components in a solution. The measurement of the refractive index gives an approximation of the total dissolved solids, graduated in °Brix, which is also named refractometric dry substance (RDS). The analytical procedure is ICUMSA 4/3-13 (2007) (ICUMSA 2007).

°Brix reading: The diluted sample is piped into labeled plastic centrifugation vials (1.5 mL) and after centrifugation (5 minutes, 10,000 rpm); few drops of the centrifuged diluted sample are dispensed over the refractometer prism. The °Brix (± 0.03°) are determined from the display and calculated for the sample °Brix using equation 3.2

Brix=Brix reading·DF (Equation 3.2)

DF= Dilution Factor

3.2.2.3. Raw Sugar Moisture

Moisture and non-sucrose impurities can be located on different parts of a raw sugar crystal, such as on the surface (free moisture), between crystal layers (bound moisture) and within the crystal structure (inherent moisture). The drying method only determines free moisture. Sample weight, temperature and time of drying and, cooling conditions have to be followed exactly and the analysis is done in duplicate. The analytical procedure is ICUMSA GS2/1/3/9-15 (2007) (ICUMSA 2007).

- Dry aluminum drying dishes and lids in a forced draught atmospheric pressure oven at 105 °C for ~30 min. Let the dishes and lids cool in a desiccator until the surface temperature is 2 °C above ambient temperature and rapidly weigh them, ±0.1 mg (m_1=weight of dish and lid, g).
- Approximately, 15 g of raw sugar are rapidly weighted on the drying dishes, register the weight of sugar, ±0.1 mg (m_2=weight of dish, lid and wet sample, g).
- Place the uncovered drying dishes with the lids into a forced draught atmospheric pressure oven at 105 °C and dry the raw sugar samples for exactly 3 hours.
- Let the covered dishes with samples cool in a desiccator until the surface temperature is 2 °C above ambient temperature and rapidly weigh them, ±0.1 mg (m_3= weight of dish, lid and dry sample, g).

Loss on drying –moisture is calculated using equation 3.3 and Brix of Raw Sugar with equation 3.4

$$\text{Raw Sugar Moisture}=\left[\frac{m_2-m_3}{m_2-m_1}\right]*100$$ (Equation 3.3)

(m_1, m_2, m_3 are defined as above)

Raw Sugar °Brix = 100 - Raw Sugar Moisture (Equation 3.4)

3.2.2.4. Polarization or Apparent Sucrose Content (Pol)

The concentration of sucrose in a solution can be determined from the angle of optical rotation when a plane-polarized light passes through it. The optical rotation is measured with a polarimeter and the reading is called polarization or "pol". The polarization according to the International Sugar Scale is expressed in °Z where, 100 °Z is the optical rotation for a normal pure sucrose solution (26.000 g of pure sucrose in 100.000 mL of pure water at 20 °C) (ICUMSA, 2007).

At the end of the crystallization process, massecuites and molasses have more non-sucrose than sucrose components. The optical rotation for a sugar solution containing substances with (+) dextrorotatory and (–) levorotatory optical activity is the result of the summation of the individual rotations, which affects the determination of sucrose concentration. The "pol" values normally give an approximation for sucrose concentration which is lower than true values. This procedure is widely used in the sugar industry for a quick assessment at each stage of the manufacturing sugar process.

The procedure for determination of polarization (pol) for diluted boiling house samples of syrup, massecuites, and magmas (see sample preparation 3.2.2.1) is as follows:

- Weigh 26.00 g (w_{sln}) of the diluted sample into a 200 ml volumetric flask and bring to volume with DI water
- Transfer the solution to a 200 ml jar and add a lead-free clarification agent Octapol™ (e.g. for high purity materials ~5 g and for very-low purity materials ~15 g) covering the jar with a lid and shake

- Filter through a filter paper Whatman 91 (or equivalent) discarding the first ~10 ml of filtrate
- Collect approximately 100 – 150 mL of the filtrated solution to rinse and fill the polarimeter cell (200 mm glass cell or stainless steel continuous cell)
- Place the cell in the polarimeter and read the polarization at 589 nm (the instrument should be zeroed for the same cell containing DI water

The polarimeter reading has to be corrected for the temperature of the solution, which is determined by placing a thermometer in the mouth of the cell (a continuous cell is programed to give the solution's temperature). Equation 3.5 was used to calculate the pol of the sample.

$$\text{Pol (°Z)}=\text{Pol read}\cdot\text{DF}\cdot\frac{52}{w_{sln}}\cdot[1-c\cdot(t-20)] \qquad \text{(Equation 3.5)}$$

- w_{sln} = weight the of diluted sample, g
- DF = dilution factor
- t = temperature of the tested sample solution, °C
- c = 0.000467 tube and flask: borosilicate (589 nm) ICUMSA 1/2/3/9-1 (2007)
- c = 0.000490 tube and flask: borosilicate (880 nm) ICUMSA 1/2/3-2 (2005)
- c = 0.000455 tube: steel; flask: borosilicate (589 nm) ICUMSA 1/2/3/9-1 (2007)
- c = 0.000478 tube: steel; flask: borosilicate (880 nm) ICUMSA 1/2/3-2 (2005)

The procedure to determine polarization (pol) of raw sugar is a modification of the ICUMSA GS1/2/3-2 (2005) (ICUMSA 2007). The basic steps of this procedure are:

- Weight 26.000 ± 0.002 g (w_{sug}) of the raw sugar sample and quantitatively transfer to a 100 ml volumetric flask and bring to volume with DI water at 20°C
- Filter through a filter paper Whatman 91 (or equivalent) containing ~ 3.5g of Celite discarding the first ~15 ml of filtrate. Minimize evaporation covering the top of the filter funnel with a glass watch
- Collect approximately 50 – 60 mL of the filtered solution to rinse and fill the polarimeter cell (200 mm glass cell)
- Place the filled cell in the polarimeter and read the polarization at 589 nm (the instrument should be zeroed before for the same cell with DI water).

The polarimeter reading has to be corrected by the temperature of the solution which is determined placing a thermometer in the mouth of the cell. Equation 3.6 was used to calculate the pol of the raw sugar sample.

$$\text{Pol(°Z)}=\text{Pol read}\cdot\frac{26.000}{w_{sug}}\cdot[1-c\cdot(t-20)] \qquad \text{(Equation 3.6)}$$

- w_{sln} = weight the of raw sugar, g
- t= temperature of the tested sample solution, °C
- c = 0.000467 tube and flask: borosilicate (589 nm) ICUMSA 1/2/3/9-1 (2007)

The apparent purity of boiling house process streams and raw sugar is determined by equation 3.7.

$$\text{Purity}=\frac{\text{Pol}}{\text{Brix}}\cdot 100 \qquad \text{(Equation 3.7)}$$

3.2.2.5. Conductivity Ash

In the sugar industry, routine measurement of ash, which, by definition would require incineration of the organic compounds, is determined by electrical conductivity, which correlates well with the gravimetric method – sulfated ash. Conductivity ash determines largely inorganic compounds (introduced with the cane and/or the processing aids) which are soluble and dissociated in water. The dissociated inorganic compounds include cations such as potassium, calcium, sodium and iron and the anions chloride, sulfate, sulfite, silicate, carbonate, oxalate, citrate and phosphate. Some of these compounds are associated with colloids of high molecular weight which are responsible for turbidity and color in solution (van der Poel, Schiweck et al. 1998; Rogé, Bensouissi et al. 2007). Conductivity ash and color in sugar solutions are correlated.

Conductivity ash in raw sugar and in other process streams was analyzed using ICUMSA GS1/3/4/7/8-13 (ICUMSA 2007). The sample dilution is such that the conductivity measurement does not exceed 500 µS/cm. For raw sugar, 5.000 g of sample are weighed, dissolved in 40 mL of deionized-DI water, quantitatively transferred to a 100 mL and brought to volume with DI water. For boiling house streams, 0.6 g to 1.0 g (w_S) of the diluted samples (see sample preparation 3.2.2.1) were weighed into a 100mL volumetric flask and then brought to volume with DI water

The calibration of the conductivity meter is adjusted using a 0.0025 mol/L Potassium Chloride (KCl) solution which has a conductivity of 328 µS/cm at 20 °C (362.4 µS/cm at 25 °C). The conductivity of the DI water, used to prepare the solution, is read and registered (water conductivity must be less than 2 µS/cm) and then the same is done to the sample solution, rinsing the probe 3 times with the same solution before reading. The solution level must be above the upper steel bar of the probe.

Temperature correction for a temperature range of 20 ± 5 °C.

$$C_{20°C}=\frac{C_T}{[1+0.023\cdot(t-20)]} \qquad \text{(Equation 3.8)}$$

C_t= Conductivity in µS/cm at t in °C

Correction for water conductivity

$$C=C_{s@20°C}-C_{w@20°C} \qquad \text{(Equation 3.9)}$$

- $C_{s@20°C}$ = Sample conductivity in µS/cm at 20 °C
- $C_{w@20°C}$ = specific conductivity of the water in µS/cm at 20 °C

The conductivity ash of the actual sample is calculated by equation 3.10

$$\text{Conductivity Ash (\%sample)}= (16.2+0.36\cdot DS)\cdot 10^{-4}\cdot C\cdot f \qquad \text{(Equation 3.10)}$$

- DS = Dry substance concentration of the g/100mL solution ($DS = w_S \cdot B_s/100$)
- B_s= Brix reading of diluted sample (sample preparation 3.2.2.1)
- w_s= Weight of diluted sample
- S = Mass of sample (g) in 100 mL ($S = w_S \cdot DF$)
- DF= Dilution Factor (sample preparation 3.2.2.1)
- f = Correction factor to 5 g/100 mL (f = 5/S)

3.2.2.6. Color in Solution

Color of sugar solutions is both pH and wavelength dependent. It is an apparent measure of the effects of a number of different colored compounds present in sugar juices. The analytical procedures to determine 'color in solution' of the diluted sample (see sample preparation 3.2.2.1) require: filtration through 0.45 µm cellulose nitrate membrane filters (modified ICUMSA GS1/3-7) or filtration through 1.2 µm glass microfiber membrane filters Whatman GF/C or equivalent (modified ICUMSA 4 – 1978). The second protocol is used to determine the color value used to trade raw sugar between the factories and refineries. The pH of the filtered solution is adjusted to pH 7.0±0.1 (modified ICUMSA GS1/3-7) and/or to pH 8.5±0.1 (modified ICUMSA 4 – 1978)

Affined sugar samples are prepared by washing 500 g of raw sugar, by steadily adding 140 mL of 64 Brix saturated sugar solution over 4.5 minutes, with continuous slow mixing (Cuisinart stand mixer, speed 1) and then, the mixing is continued for an additional minute. The mixture is immediately centrifuged for 2.25 minutes at 3000 rpm. The affined sugar then is spread (< ¼ inch layer) over a clean surface to dry at ambient temperature while periodically mixing by hand.

Color analysis: vacuum filter the diluted sample through the 0.45µm or 1.2 µm filter. Carefully adjust the pH, using a solution 0.05N hydrochloric acid (HCl) or 0.05N sodium hydroxide (NaOH), to pH 7.0±0.1 or to pH 8.5±0.1. Pour the solution into a 10 mm spectrophotometer square cuvette and read the absorbance at 420 nm (and at

720 nm for the solution filtered at 1.2 µm to correct for turbidity). Use DI water as a blank. Read the refractometric dry substance (RDS) of the solution. Calculate color with equation 3.11 or 3.12.

$$\text{Color }(0.45\mu m)=\frac{10^8 \cdot A_s}{b \cdot RDS \cdot \rho} \qquad \text{(Equation 3.11)}$$

$$\text{Color }(1.2\mu m)=\frac{10^8 \cdot (A_{s@420}-2 \cdot A_{s@720})}{b \cdot RDS \cdot \rho} \qquad \text{(Equation 3.12)}$$

- Color = Color units, CU (cm^2/kg dry substance)
- A_s= sample absorbance at 420 or 720 nm (absorbance units)
- b= path length spectrophotometer square cuvette, cm
- RDS=refractometric dry substance, °Brix
- ρ= sugar solution density from ICUMSA Table SPS-4, kg/cm^3

3.2.3. Modelling a Three-Boiling Crystallization Scheme with Double Purge of C-Magma

The boiling house in a raw sugar factory traditionally contains a sequence of batch or continuous crystallization stages, denominated strikes, which are identified by letters or numbers (e.g. A, B, C or first, second and third) (Chen and Chou 1993). At each stage, evaporation changes the concentration and the solubility of the sucrose present in the feed. Increasing the sucrose concentration beyond supersaturation drives the crystallization. Supersaturation is the core concept in sucrose crystallization. Each crystallization stage, which only removes a specific amount of the sucrose present in the feed, is constrained or limited by impurities which modify sucrose solubility and by viscosities of the final materials (strike massecuite). The crystals produced are separated from the liquid (molasses) by centrifugation, and small amounts of water are added to remove some of the surface molasses. The mother liquor (molasses) separated from the crystals may go either to the next crystallization stage or may be sent back to an upstream stage. In a three-boiling scheme, the crystals separated from the last strike are normally used as crystal seed to produce commercial raw sugar in the first and second strikes.

The methodology used for a material balance from available data from boiling house operations was that of Birkett and Schaffer (1978). Solids (soluble solids or dry substance) and sugar balances (equations) were combined to solve the system. With some assumptions, it was possible to estimate mass and volumetric rates for each material stream and determinate the required amount of vapor. The solids and sugar balances were created from laboratory analytical results. The concentration of non-sucrose compounds on the soluble solids is calculated as 100 – purity. These calculations gave estimations of soluble solids, sucrose and non-sucrose (impurities) concentrations but the estimation error increases as the concentration of impurities increases, thus the term 'apparent' purity is more appropriate. At steady state, assuming no formation or destruction of sucrose in the boiling house, the overall solids, sugar and color balances were estimated using equations 3.13 to 3.17. The same balance was conducted for each station in the boiling house, as the output of one station becomes the input of one or more stations upstream or downstream in the process. Figure 3.3 shows the inputs and outputs for an overall balance. Balances are conducted assuming steady state, but the input to the boiling house changes slowly with time. Considering the retention time on equipment and tanks, the process from syrup to final molasses can take more than 3 days. This is important for statistical analysis since blocking data on weekly periods can be more representative of the variation than using shorter time periods.

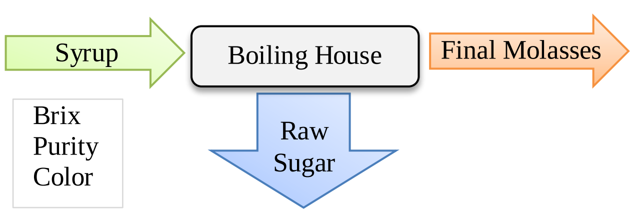


Figure 3.3 Overall boiling house input and outputs for solids, sugar and color balance

Overall Soluble Solids (or Dry Substance – DS) balance: (Equation 3.13)

$$w_{syrup} * B_{syrup} = w_{sugar} * B_{sugar} + w_{molasses} * B_{molasses}$$

Overall 'Apparent' sugar balance: (Equation 3.14)

$$DS_{syrup}*P_{syrup}=DS_{sugar}*P_{sugar}+DS_{molasses}*P_{molasses}$$

- w = weight or mass-rate (kg/hour)
- B = Brix/100 (solids fraction)
- DS = w·B = Soluble solids – dry substance rate (kg solids/hour)
- P = apparent purity= pol % Brix /100 (kg sugar per kg soluble solids)

Overall Color Balance

"Colorants" compiles "all non-sugars compounds" that compose the measurement of "color in solution" and the results from the analytical procedures are given by the equation 3.15 (Henpelmann 2006).

$$q_{col/sol}=\frac{n_{col}}{DS_{sol}} \quad \text{(Equation 3.15)}$$

- $q_{col/sol}$ = colorants content per weight of soluble solids
- n_{col} = quantity of colorants
- DS_{sol} = weight of soluble solids

Thompson and Fry (2003) from van der Poel, Schiweck et al. (1998) state that since 'colorants' form part of the non-sucrose fraction, (assuming no formation or destruction of non-sucrose components) it is more convenient for mass balance on centrifuges (separation of sucrose and non-sucrose components) and for color formation quantification to use the color non-sucrose ratio, equation 3.16 (Schick 1994; Thompson and Fry 2003), instead of a color soluble solids ratio (equation 3.15).

$$\frac{\text{Color}}{\text{NS}}\ \text{ratio}=\frac{\text{Color ICUMSA}}{\text{100-Purity}}/100 \quad \text{(Equation 3.16)}$$

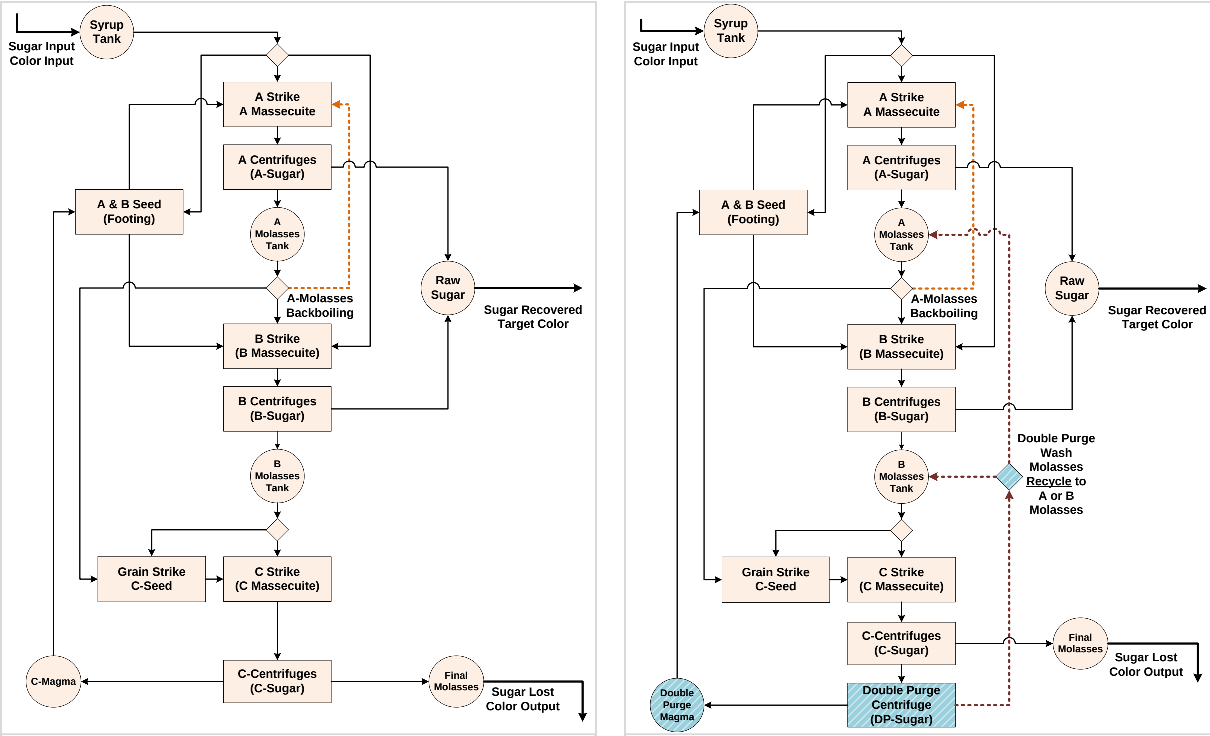
- NS = 100-purity = kg of non-sucrose per 100 kg of soluble solids
- (Color/NS)ratio = cm^2 per 10 mg of non-sucrose compounds

Overall color balance (steady state): (Equation 3.17)

$$DS_{syr}*NS_{syr}*\frac{\text{Color}}{NS_{syr}}=DS_{sug}*NS_{sug}*\frac{\text{Color}}{NS_{sug}}+DS_{mol}*NS_{mol}\frac{\text{Color}}{NS_{mol}}-\Delta\text{Color}$$

- DS = Soluble solids – dry substance rate (kg solids/hour)
- NS = Non-sucrose compounds fraction = (100-Purity)/100
- ΔColor = color formation in the whole boiling house

Figure 3.4 shows a flow pattern for a traditional three-boiling scheme with both single and double purge of C-magma. The solution for a total mass, components and energy balances requires an iterative methodology, and a more rigorous model, to compare different boiling schemes, simulate changes on product parameters, simulate the addition of more stages or the use of a different type of pan crystallizer and, to model color profiles (Weiss 1989; Wright 1989; Broadfoot, Miller et al. 1994; Radford 1996; Wright 1996; Thompson 1997; Broadfoot and Pennisi 2001; Broadfoot and Pennisi 2002; Peacock 2002; Broadfoot 2006; Saska 2008).



a) Three-boiling scheme (single purge of C-magma)

b) Three-boiling scheme with double purge of C-magma

Figure 3.4 Process Flow diagrams for traditional three-boiling crystallization scheme a) with single purge and b) with double purge of C-magma. The new blocks for the double purge system are on blue color (VISIO$^{(R)}$ 2007)

Sugars^{TM} (2014) is a software specifically created for the sugar industry which allows construction of a flow sheet that combines different stations, each with control parameters, that can be customized to reflect the actual performance of each station (Stolz and Weiss 1997; Weiss 1999). Figure 3.5 shows the template for a three-boiling crystallization scheme including basic boiling house equipment (shapes). Other shapes can be added for more equipment or for a specific objective such as to apply a blending rule or to modify a separation.

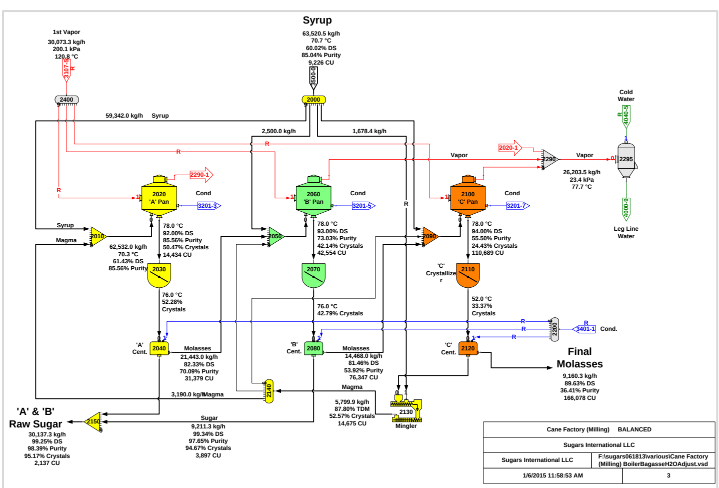


Figure 3.5 Three boiling crystallization template including basic equipment such as batch pans, crystallizers, centrifuges, magma mixer, etc. (Sugars^{TM} 2014)

Year-to-date data from factory reports and mass and energy balance reports (Birkett 2011) were used to create boiling house specific models for each factory that was implementing a double purge of C-magma system. Figure 3.6 illustrates some of the data that was required to define the inputs and to set the pans and centrifuges on a model created on Sugars™.

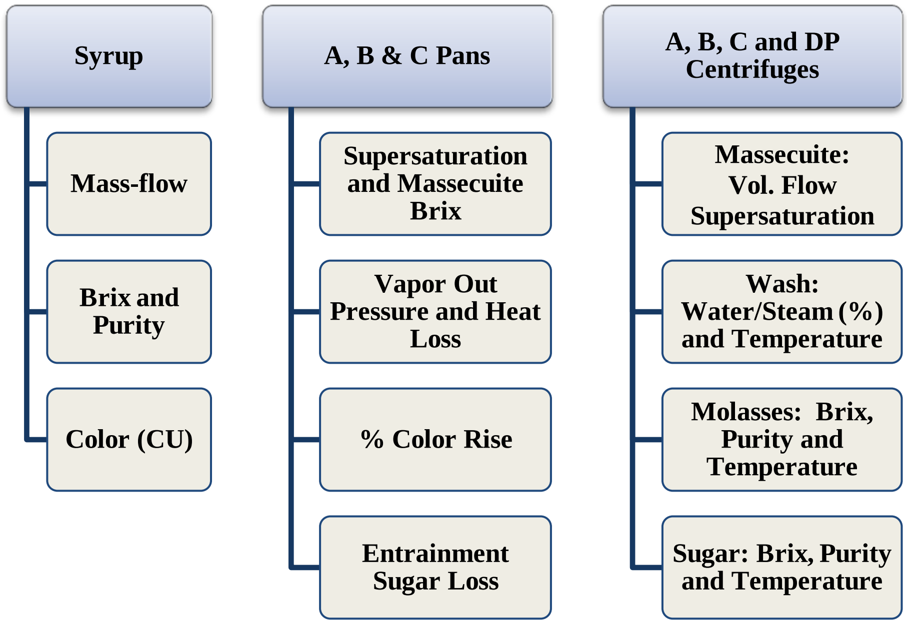


Figure 3.6 Process parameters and flow streams parameters (product parameters) inputs to model or simulate the operation of the boiling house using (Sugars™ 2014)

3.3. Milestones in Factory Implementation

This overall project was initiated by Dr. Vadim Kochergin and proposed to the Louisiana sugar mills in 2010/2011. Sterling Sugars Inc. accepted responsibility to test a double purge system in 2011. Models were created to evaluate capacities of equipment and pumps at Sterling. A partial system was installed and operated during the 2011. This attracted the interest of Lula Sugar Factory. Models were created and Lula Sugar Factory did a complete installation of the system in 2012. The results obtained during the 2012 crop season attracted two more Louisiana factories. In 2013, Double purge of C magma was operated by Lula (LU), Westfield (WF) and Cora-Texas (CT) factories during the complete crop season (October-January). In all, four factories have installed equipment in relevance to this project. Results collected from the start of the double purge of C-magma project from these factories are given below. They demonstrate that double purge produces significant color improvement in raw sugar. Enough information was collected to validate and improve boiling house models, with emphasis on profiles of whole color and product parameter correlations that could be used with the Sugars™ model for optimization of the system.

3.3.1. Sterling Sugars Trial

During 2011 sugar processing season, Sterling Sugars Inc. (Franklin, Louisiana), ran a trial to test a double purge system to reduce raw sugar color. Information on their boiling house equipment and 2010 performance reports were assembled to create a model using Sugars™. Figure 3.7 is a sketch of the boiling house equipment at Sterling Sugars Inc. and the proposed integration of double purge of C-magma.

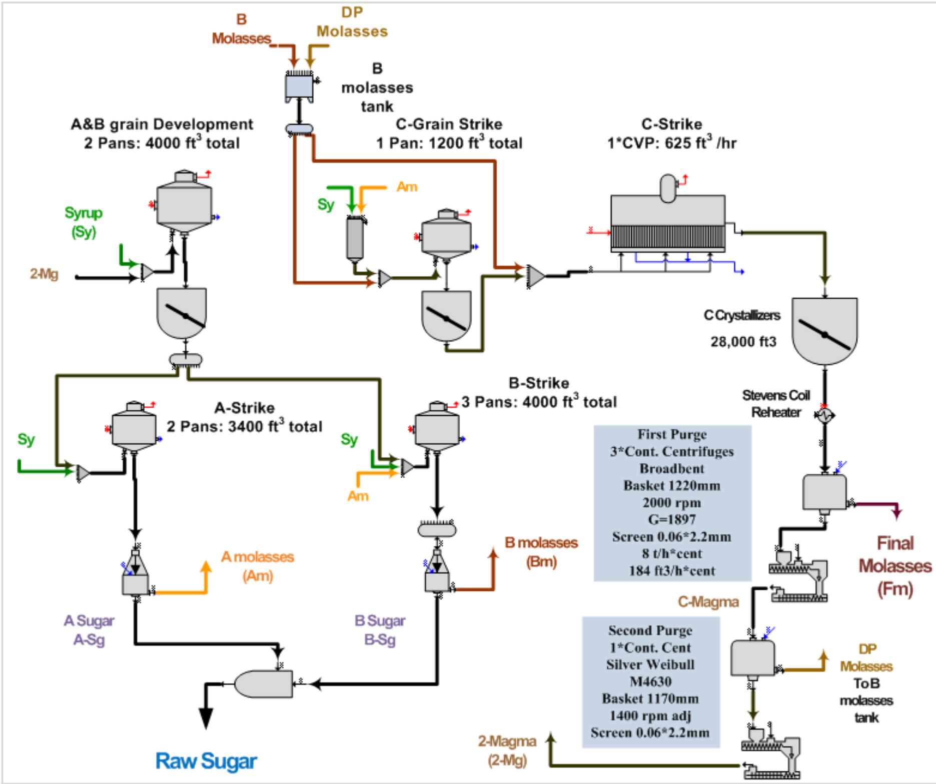


Figure 3.7 Boiling house at Sterling Sugars Inc. integrating double purge of C-magma

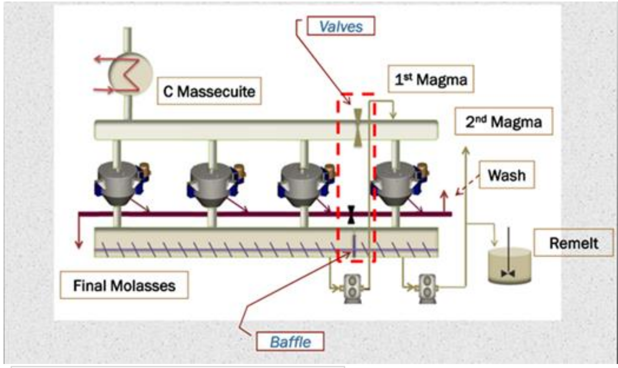
Based on purity profiles, sugar yields and raw sugar whole color from the 2010 sugarcane crop season at Sterling Sugars Inc., a double purge C-magma system was modeled and simulated using Sugars™ software. The simulation results were used to estimate equipment and pump capacities. Table 3.3 shows a summary of expected mass and volumetric rates (11,500 tons of cane per day, ~479 tons cane /hour) obtained from the simulation, with first and double purge magma purities compared to model results for the traditional three boiling scheme and the average results obtained by the factory during the 2010 season. Color input on syrup and color formation of each strike was assumed and adjusted according to the reported raw sugar whole color.

Table 3.3 Preliminary models and simulations (S) results comparison for a 3-boiling scheme and different combination of first C-magma (M) and double purge C-magma (DPM) purities. Model assumptions: color input in syrup of 28,720 CU and approximated color formations of 26%, 37% and 65% for A, B and C strikes for a total color formation of ~ 247% (Sterling Sugars Inc.)

DESCRIPTION	STRIKES		RAW SUGAR			MOLASSSES		M	DPM
	A + B	C	PDN	POL	COLOR	PDN	TRUE	APP.	APP.
	ft^3	ft^3	lb	°Z	CU	gal	PURITY	PURITY	PURITY
2010-ACTUAL	--	--	215	98.6	3,840	5.1	43.8	85.4	NA
2010-MODEL	4.8	1.0	230	98.6	3,799	6.1	42.9	85.5	NA
S1 (M=81/DPM=93)	4.6	1.1	234	99.2	1,406	5.9	38.7	81.4	93.0
S2 (M=81/DPM=95)	4.6	1.1	233	99.2	1,342	6.0	39.8	81.5	95.1
S3 (M=81/DPM=97)	4.6	1.1	231	99.2	1,292	6.0	40.2	81.5	97.0
S4 (M=85/DPM=95)	4.6	1.0	230	99.2	1,325	6.3	42.3	85.0	95.1
Rates of strikes volumes, molasses and sugar production normalized respect cane grinding rate									
Traditionally, US sugar industry uses the English system of units									
1 lb = 0.454 kg, 1 short ton = 2000 kg, 1 ft3 = 0.028 m^3, 1 gal = 0.0038 m^3									

This simulation showed that using of magmas of different purities (1st magma: ~81 purity, and DP magma: ~93 purity) at Sterling Sugars, Inc. it will be achieved a significant sugar color improvement (from 3800 to 1400

CU), a reduction in high grade massecuite (A&B) volumes of about 3% and an incremental volume of low grade massecuite (C) of approximately 8%. Figure 3.8 shows the modifications made to the C centrifugation station. A valve was installed in the C-massecuite feeding pipe to separate the feed to the continuous centrifuge for the double purge and a baffle was placed in the magma screw conveyor to separate the first and the second magma.

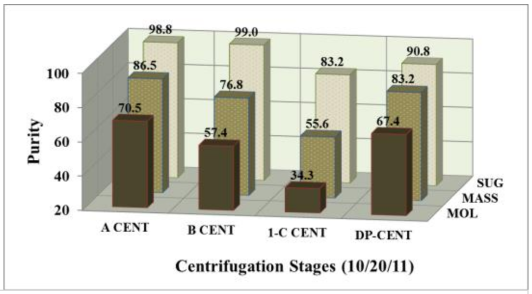


a) Double purge diagram

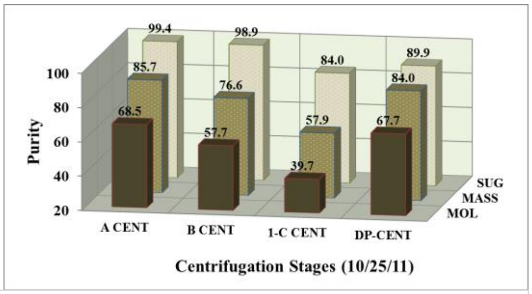
b) Double purge centrifuge

Figure 3.8 C-magma double purge trial implementation a) diagram and b) centrifuge used for double purge (Sterling Sugars Inc.)

During the 2011 season implementation of double purge was promising, but the system required changes to correct cross- contamination of the 2^{nd} magma due to the by-passing of the first magma at the baffle. The double purge worked within the parameters estimated by the simulation at the beginning of the season when sugarcane milling rates were low. However, as the mill increased its grinding rate, the first magma pump capacity was exceeded. This material overflowed the baffle in the magma conveyor contaminating the second magma, and it overflowed first magma to the C-massecuite crystallizer. Figure 3.9 shows purity profiles of each centrifugation stage, obtained on two sampling days, showing the purity drops between massecuites and molasses and the descending purities from the start to the end of the crystallization (e.g. from ~86 for A massecuite to ~ 56 for C massecuite). This purity drop on massecuites is important in order to achieve a low purity in final molasses (less sugar loss). Estimation of sugar losses in final molasses for 10/20/11 was ~ 35% and for 10/25/11 was ~ 41% (final molasses purities of 35 and 41 respectively).



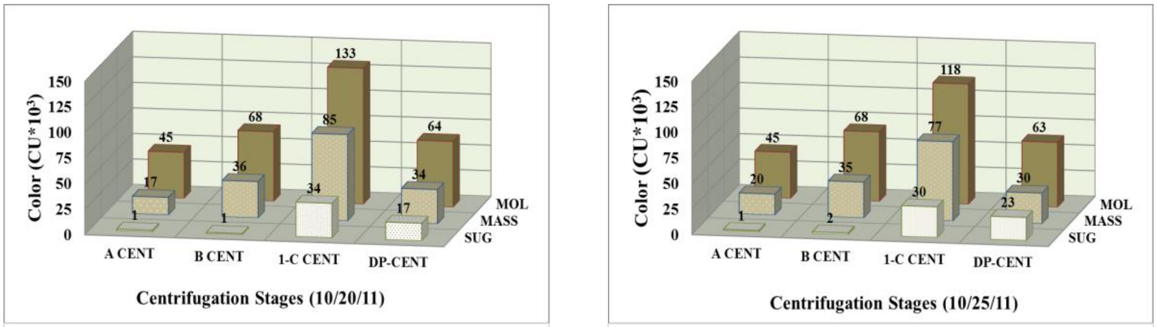
a) Centrifuges input/output purities – 10/20/11



b) Centrifuges input/output purities – 10/25/11

Figure 3.9 Input/Output purities of massecuites (MASS), sugars (SUG) and molasses (MOL) at the A, B, C and double purge (DP) centrifugation stages on a) 10/20/11 and b) 10/25/11

Figure 3.10 shows color profiles at each centrifugation stage, obtained over the same two sampling days, showing increasing color from the A massecuite (top) to C-massecuite, after cooling crystallization (from ~20,000 CU for A massecuite to ~ 80,000 CU for C massecuite). The figures show that C massecuite has almost double the color of the B massecuite. This is, because of an increasing concentration of non-sugars and color, as the sucrose is removed from the first strike to the third strike.

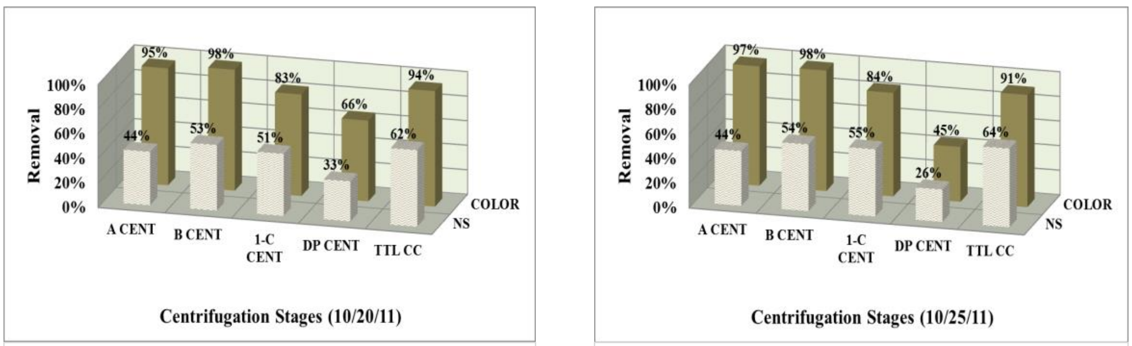


a) Centrifuges input/output color – 10/20/11

b) Centrifuges input/output color – 10/25/11

Figure 3.10 Color (at 8.5pH and 0.45μm, CU) of massecuites (MASS), sugars (SUG) and molasses (MOL) at the A, B, C and double purge (DP) centrifugation stages on a) 10/20/11 and b) 10/25/11

Figure 3.11 illustrates the removal of non-sugars and color at each centrifugation, as well as the combined removal by the first and the second centrifugations of C-magma. In total the removal of color from C-massecuite was 94% and the non-sugar removal was 62%.



a) Non-sucrose and color removal – 10/20/11

b) Non-sucrose and color removal – 10/25/11

Figure 3.11 Non-sucrose (NS) and color removal at the A, B, C and double purge (DP) centrifugation stages on a) 10/20/11 and b) 10/25/11

Figure 3.12 shows a correlation between color (CU), and purity (%) for the first and second C-magma. It can be seen from the graph that the color of the magma ~28,000 CU at purity of ~85 in the simple three boiling scheme can be improved to ~ 15,000 CU by having a purity of ~92 in the double purge of C magma, without increasing sugar losses to final molasses at the first centrifugation. The sugar losses with final molasses are because it is required to add more hot water on C-centrifuges to increase the purity of the magma from 85 to 93.

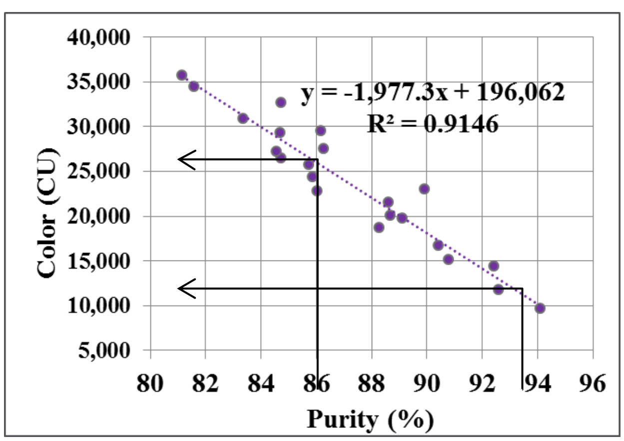
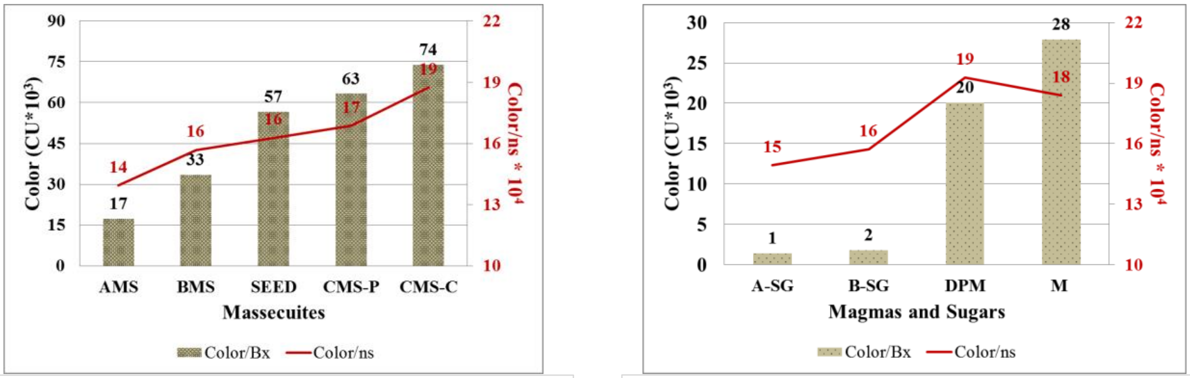


Figure 3.12 Color (at 8.5pH and 0.45μm, CU) versus purity of first and double purge magma samples taken on 2011 crop season at Sterling Sugars Inc.

Figure 3.13 shows how color (CU) increases or concentrates in massecuites and molasses as the sucrose is removed from the mother liquor, while the color on sugar decreases with crystal size, from the small crystals (first magma) to the large crystals of the highest purity (A sugar). Assuming perfect crystals with no color absorption or color formation during crystallization, the color/non-sugars ratio (line trend) should remain the same through the crystallization stages. The ascending trend of the color/ns ratio line indicates color formation on each of the crystallization stages

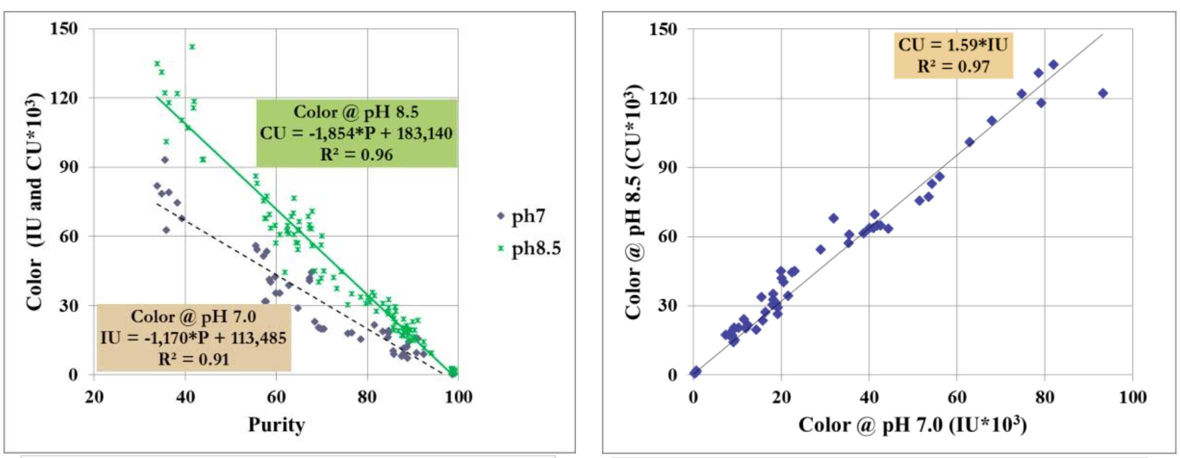


a) Color and color/non-sucrose for massecuites

b) Color and color/non-sucrose for magmas and sugars

Figure 3.13 Boiling house color (bars) and color/non-sucrose ratio (line) profiles (Sterling Sugars, 2011). From high to low purity massecuites: AMS, BMS, SEED and CMS (P-pan and C-after cooling crystallization). From low purity magmas (M-first magma and DP-double purge magma) to raw sugar (A-SG plus B-SG)

Approximately 130 composite (3 hours) samples were taken on one day each week, during 9 weeks of the processing season. The samples were analyzed for Brix, apparent purity and color, from a filtered (0.45 µm) solution, at pH 7 (official ICUMSA method, IU units) and at pH 8.5 (color units, CU units). Color measurement at 7 or 8.5 pH inversely correlates with the purity of the material; the higher the purity, the lower the color. Color measurement at pH 8.5 is higher and positive correlated with the color measurement at pH 7, Figure 3.14.



a) Color (pH 7 and 8.5) versus purity

b) Color pH 8.5 (CU) versus color pH 7 (IU)

Figure 3.14 Correlations plots for color of diluted boiling house process streams filtered (0.45 µm) a) color at pH 7 (ICUMSA Units – IU) and color at pH 8.5 (Color Units – CU) versus material purity b) color at pH 8.5 (CU) versus color at pH 7 (IU)

The results from a double purge trial in 2011 proved that the implementation will not cause an increase on massecuite processing volumes which reduces crystal yield and increases the demand of energy and, the capital investments were not as high as with a different boiling scheme. These results were presented in 2012 at the annual meeting of the American Society of Sugarcane Technologists (ASSCT) – Louisiana division (Polanco, Kochergin et al. 2012) and, at the Audubon Sugar Institute Factory Operation Seminar (Polanco, Kochergin et al. 2012) promoting the full implementation of the system by another factory.

3.3.2. Full Implementation at Lula Sugar Factory

Lula Sugar Factory (Belle Rose, Louisiana) installed a double purge system for the 2012 sugarcane crop season. Figure 3.15 shows boiling house equipment and their integration of a double purge system.

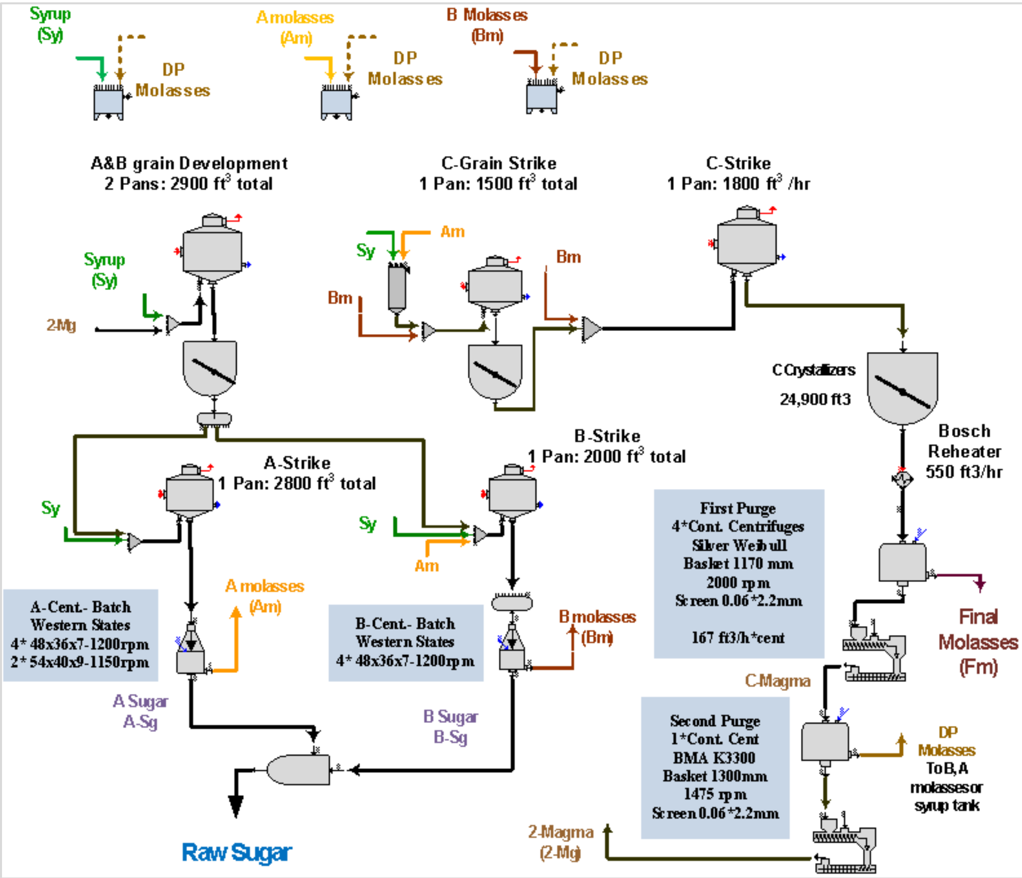


Figure 3.15 Boiling house at Lula Sugar Factory integrating double purge of C-magma

Lula system includes a magma receiver which feeds a BMA k3300 continuous centrifuge (Bartsch and Geyer 2010); the receiver is equipped with a mixer and level control. The double purge molasses separated from the C sugar crystals is routed to a dilution tank equipped with instrumentation to control Brix and levels in the tank. The diluted molasses is conducted by a line with valves that can be manually or remotely manipulated to send this material to the A molasses, B molasses or syrup tanks. The affined or double purge magma is received by a screw conveyor and then it is pumped to magma receivers at the pan floor. Any excess of first or second magma is automatically sent to a remelt tank. The system was automated and can be remotely operated. Details of the double purge are presented in Figure 3.16.

Because of limited capacity of the double purge molasses pump, large amounts of B molasses (leaks of final molasses to C magma) and accumulations of crystals in the magma compartment, the double purge centrifuge required frequent stops to clean at the beginning of the season. When these problems were resolved the system was continuously operated for the entire crop, stopping only for liquidation at the end of the season. Because of the high purity of the double purge molasses, this stream was routinely sent to the A molasses tank. Analyses of the two new streams – double purge magma and double purge wash, were added to the laboratory work load. The purity range for the double purge molasses were defined and then closely monitored by Lula personnel. The results of the analysis of raw sugar from the double purge system at Lula factory were compared to those from Westfield factory, which operated a traditional 3-boiling scheme and had equivalent quality of sugarcane (common sources). Raw sugar daily analysis for Lula and Westfield were performed by the factory lab at Westfield. The method used for color analysis was the one used for sales purposes – modified ICUMSA method 4 (1978); specifying the use of a glass membrane (1.2 μm) for filtration, solution at 25% solids, pH adjustment of filtered solution at 8.5, and absorbance reading at

two wavelength 420 and 720 nm. The same specifications are applied to analyze the 50% solids solution of affined raw sugar. Besides color tests, the daily composite samples were analyzed for the other parameters that are measured for payment purpose; conductivity ash, invert sugars, starch and dextran. These parameters define raw sugar quality for its process ability at the refinery and storage stability.

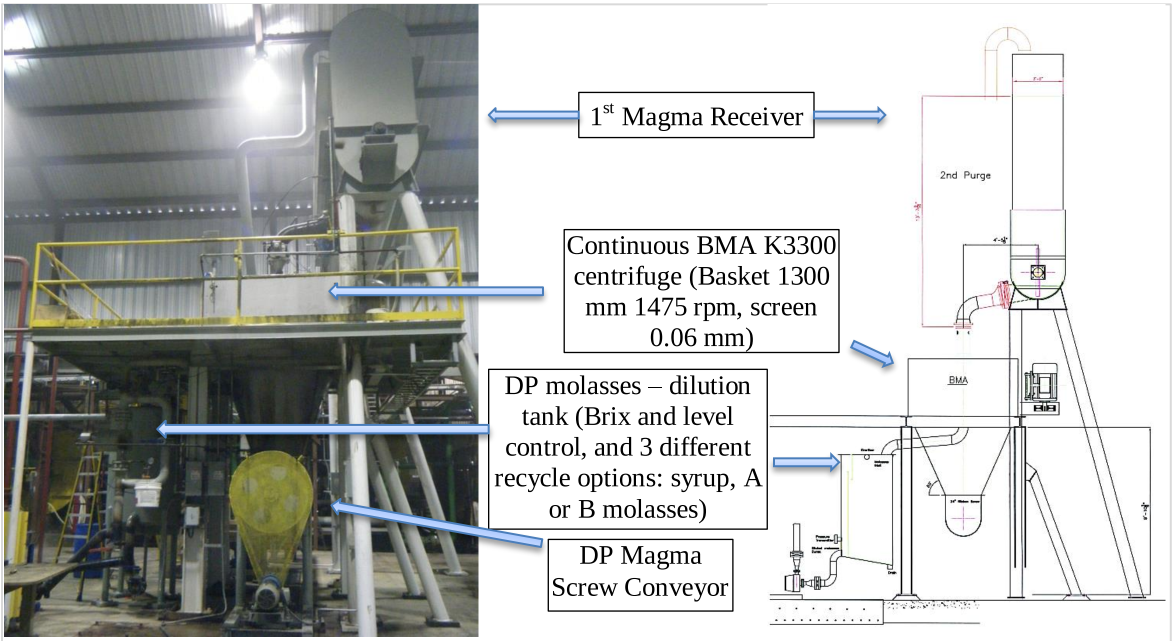


Figure 3.16 Double purge of C-magma at Lula Sugar Factory – 2012. Drawing courtesy of G. Carline (Lula)

Figure 3.17 shows the average daily purities of the magmas and double purge (DP) molasses throughout the 2012 season at Lula. Spikes of very low purity wash (DP) molasses probably were due to the leaks of final molasses to the C - magma at the first set of centrifuges.

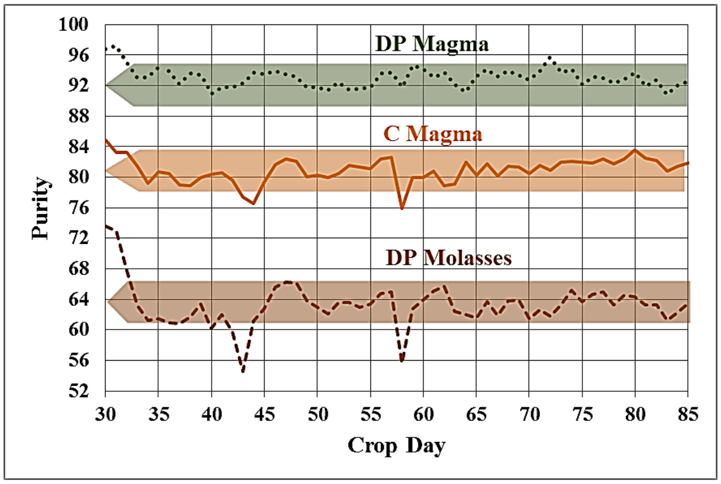
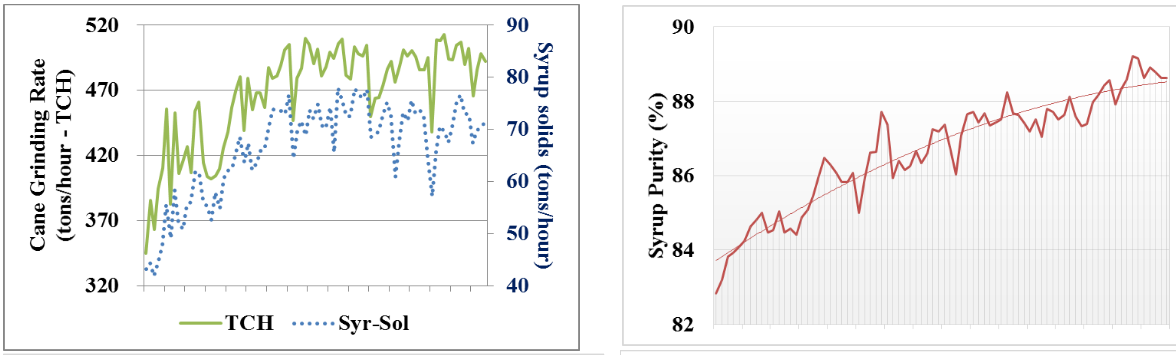


Figure 3.17 Double purge purities daily variation at Lula 2012: double purge (DP) magma, first (C) magma and double purge (DP) molasses purities

Figure 3.18 illustrates the rising trends in grinding rate, syrup solids rate and syrup purity during the 2012 sugarcane crop season. With low syrup purity the capacity required for the low grade station (pans, crystallizers and centrifuges for C massecuites) is larger than for high syrup purity; the increased capacity demand on equipment for low grade massecuites is compensated by a slower grinding rate. On the other hand, when the syrup purity is high, if the capacity of pans and centrifuges for the high grade crystallization stages (A and B) is low, the crystallization

time will be not enough to render a suitable crystal yield. Thus, the low grade station will be compromised because of the higher volume rate of C-massecuites.



a) TCH and syrup solids rate per grinding date (2012)

b) Syrup purity trend per grinding date (2012)

Figure 3.18 Line plots for daily variation at Lula – 2012 of a) cane grinding rate (short tons cane per hour – TCH) and estimated syrup solids rate (short tons per hour) and b) syrup purity

Figure 3.19 shows the crystals yields (crystal content) with respect to the purity of the massecuites for A, B and C strikes. These crystal contents were estimated from solids and sugar balances on centrifuges. Crystal content and molasses purity are directly related to the purity of the massecuite. The higher the purity of the massecuite, higher the crystal content and the purity of the molasses separated from the crystals.

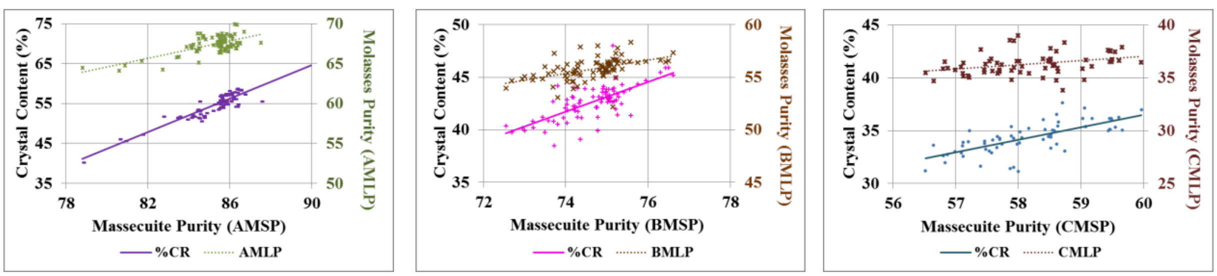


Figure 3.19 Crystal content and molasses purities for A, B and C strikes versus massecuite purity (Lula – 2012)

Figure 3.20 compares four quality parameters versus sugar polarization that were used to define the raw sugar quality, between Lula (double purge) and Westfield (single purge) from 10/23/2012 to 11/26/2012. Comparing these raw sugar quality parameters at the same polarization (99.2 °Z), the level of improvement of whole color achieved by Lula was approximately 43%, affined color (which is related to the color included in the core of the crystal) was lowered by ~14%; conductivity ash was reduced by ~ 56% and inverts (reducing sugars) were reduced ~ 2%. Double purge of C-magma primarily helps to reduce the amount of colorants and inorganic solids (ash) in raw sugar.

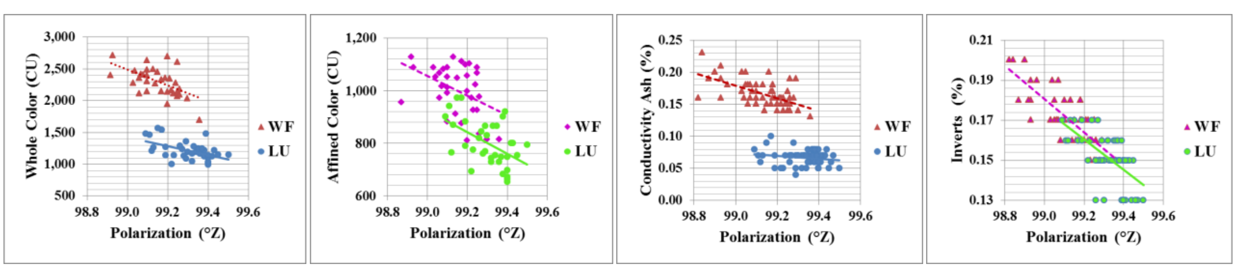


Figure 3.20 Raw sugar analysis results comparison between Lula (LU) and Westfield (WF): whole color (8.5 pH, CU), affined color (8.5 pH, CU), conductivity ash (%) and inverts (%). Note: actual values of whole and affined color for both factories were increased by a factor of 1.4 after comparing with the values obtained by the refinery due to spectrophotometer calibration failure

Table 3.4 shows weighted averages for sugar quality parameters, evaluated over a month of continuous operation of double purge system (10/23/12-11/26/12). Approximated average reductions were 47% for whole color, 20% for affined color, 57% for conductivity ash and 8% for reducing sugars for Lula versus Westfield. Starch and dextran did not change with double purge of C-magma.

Table 3.4 Comparison of raw sugar parameters (weighted-average) between Lula and Westfield from 10/23/12 to 11/26/12

Parameter	Unit	Lula	Westfield	%Reduction
Polarization	°Z	99.3	99.1	-0.2
Whole Color	CU	1,552	2,923	47
Affined Color	CU	1,026	1,289	20
Ash (Conduct.)	%	0.07	0.15	57
Reducing Sugars	%	0.15	0.17	8
Starch	ppm	188	187	
Dextran	MAU	31	26	

Table 3.5 summarizes color profiles by strike. Double purging of C-magma produced a 75% color reduction (in color units, CU) equivalent to 87% color removal. The total color removal for first and second purge was about 96%. Color removal on A centrifuges (A sugar) and on B centrifuges was approximately 98%. Color (CU) ratio of the massecuite to whole raw sugar color was about 0.04 for A massecuites and 0.03 for B massecuites.

Table 3.5 Boiling house color profile at pH 8.5 with 0.45 μm Nylon membrane filter (analysis at Audubon Sugar Institute – ASI)

Material	A Strike	B Strike	C Strike
Feed	22,590	48,590	76,220
Seed	20,640	21,020	70,770
Massecuite	19,680	38,940	73,190
Sugar	740	1,270	42,060
Double Purge			10,620

Table 3.6 compares 2012 factory performance parameters with two previous years': ground cane per day, syrup purity, overall factory sugar recovery, molasses true purity (sucrose by high performance liquid chromatography – HPLC) and target purity difference – TPD. The TPD is the difference between molasses true purity and a target purity empirically estimated from the amount of total reducing sugars (glucose and fructose by HPLC) and conductivity ash. The double purge system had no impact on the overall recovery at Lula factory in 2012, even though grinding rates had increased ~20% from 2010. Lula reduced the true purity of final molasses by 8% over this period (ASI – Molasses Survey), however, the target purity difference (TPD) did not improve (TPD=9.7) from 2011 to 2012, although it dropped by 10% from 2010. Figure 3.21 shows 12 years variation on True purity, target purity and TPD for Lula. Lula and Westfield had similar overall recoveries (85.8% and 85.5% respectively) during 2012 and the same drop in molasses true purity (8%). Any assessment of the performance of a boiling house must also consider other factors like: increased process materials flow rate for higher grinding rate; water input on syrup (evaporator performance), molasses dilution and magma melting; centrifuges performance and; steam (energy) availability, because all reduce the equipment capacity of the boiling house.

Table 3.6 Comparison of 3 years of some performance parameters from Lula's factory reports and from Audubon Sugar Institute "molasses survey report"

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

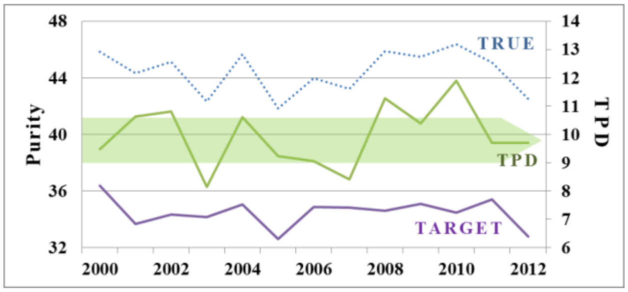


Figure 3.21 True purity, target purity and target purity difference on 12 years for Lula Sugar Factory (Audubon Sugar Institute – ASI, molasses survey)

The results attained by the full implementation of the double purge of C-magma at Lula compared to the traditional single purge system at Westfield (2012 crop system) were presented on three meetings in 2013 to the Louisiana and Florida sugar industry (Polanco, Kochergin et al. 2013). The implementation and ad hoc simulations results were published in 2014 by the peer review International Sugar Journal (Polanco, Kochergin et al. 2014). The simulations were directed to evaluate the effects of changes on syrup purity on the boiling house with single purge and double purge of C-magma, Figure 3.22.

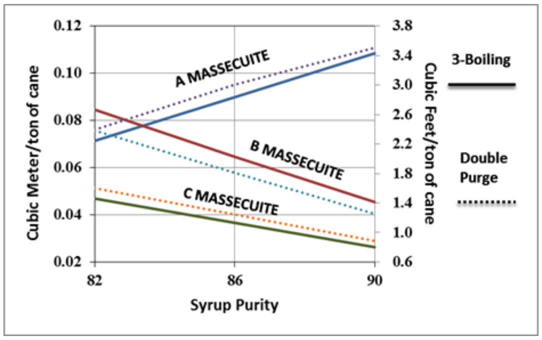


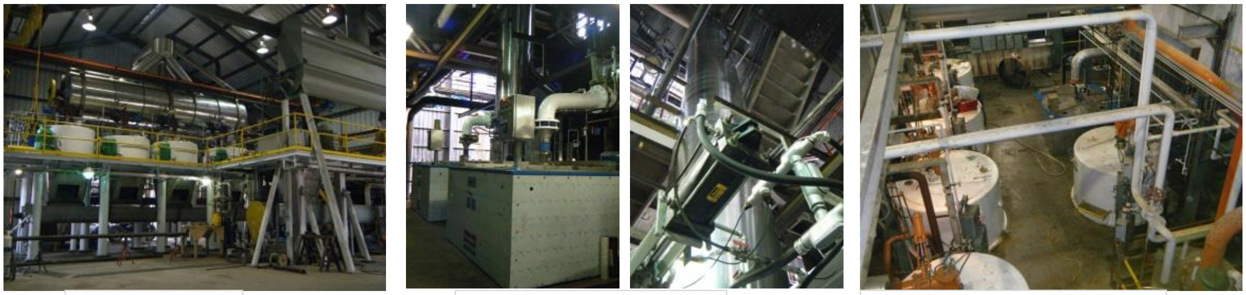
Figure 3.22 Massecuite flow-rates as a function of syrup purity respect to cane for the traditional three-boiling scheme and for three-boiling with double purge of C magma (Metric and British units) (Polanco, Day et al. 2014)

3.3.3. Performance Comparison (Lula, Westfield and Cora-Texas)

In 2013, besides Lula Sugar Factory (Belle Rose, Louisiana), Westfield Sugar Factory (Paincourtville, Louisiana) and Cora-Texas Mfg. CO., Inc. (White Castle, Louisiana) installed a double purge system, which allowed another opportunity to collect information since the management and equipment of the boiling house, are different from one factory to another. Table 3.7 shows some of the changes in double purge operation by the three factories. Lula increased the rpm for the second purge from 1475 to 1600 rpm. Figure 3.23 shows pictures of double purge for Lula (First purge moved to the same building that double purge on 2013), for Westfield two new BMA k3300 centrifuges were installed, one for first purge and another for double purge and, for Cora-Texas it shows the first purge station (4 centrifuges) and the second purge station (2 centrifuges), only the BMA k3300 was used in this season.

Table 3.7 Main components of the double purge system on Cora-Texas, Lula and Westfield 2013

	Cora-Texas	**Lula**	**Westfield**
Magma Preparation	Water Heat & Mix 3 rpm	Water Mix 7.8 rpm	Water No Mix
Centrifuge	1,800 rpm	1,680 rpm	1,680 rpm
Molasses Preparation	Water Level & Brix	Water Level & Brix	N.A.
Molasses Recycle	B molasses	A molasses	A molasses



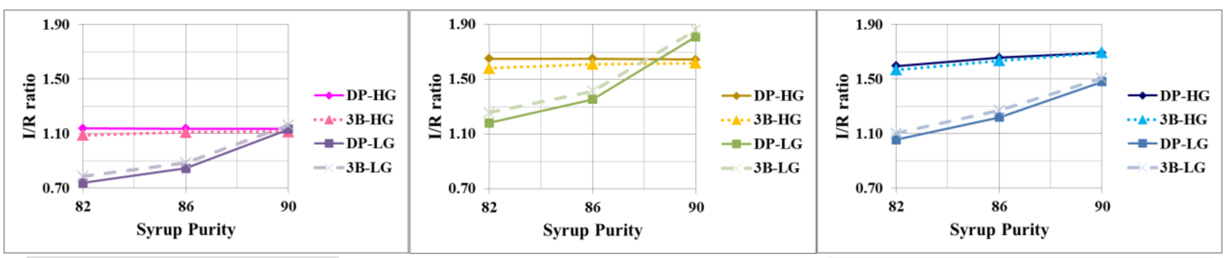
a) LULA - LU

b) WESTFIELD - WF

c) CORA-TEXAS - CT

Figure 3.23 Double purge of C-magma equipment at a) Lula Sugar Factory (Belle Rose, LA), b) Westfield Sugar Factory (Paincourtville, LA) and c) Cora-Texas Mfg. CO., Inc. (White Castle, LA)

Simulation of capacity requirements at different syrup purities for each factory were produced by applying the estimated best double purge parameters found in 2012. Figure 3.24 illustrates the results obtained from boiling house models on Sugars™ and from calculations of pan capacity using the empirical cycle times on pans for each factory (Birkett 2011); comparing single (3B) and double purge, recycling double purge molasses to A molasses (DPRC). Maximum grinding rate (tons cane per day, TCD) for each factory used in the simulation (critical condition) was 12,000 TCD for Lula, 13,500 TCD for Westfield and 18,000 TCD for Cora-Texas. These factories, had sufficient installed capacity, but the low grade pan capacity was critical for Lula at low syrup purities. During the 2013 crop season Lula ground cane above 12,000 TCD at least for 10 days, Westfield and Cora-Texas ground cane above these critical values in some days. At grinding rates above the critical values, the capacity of the boiling house is compromised, affecting sugar yields per strike and increasing sugar losses.



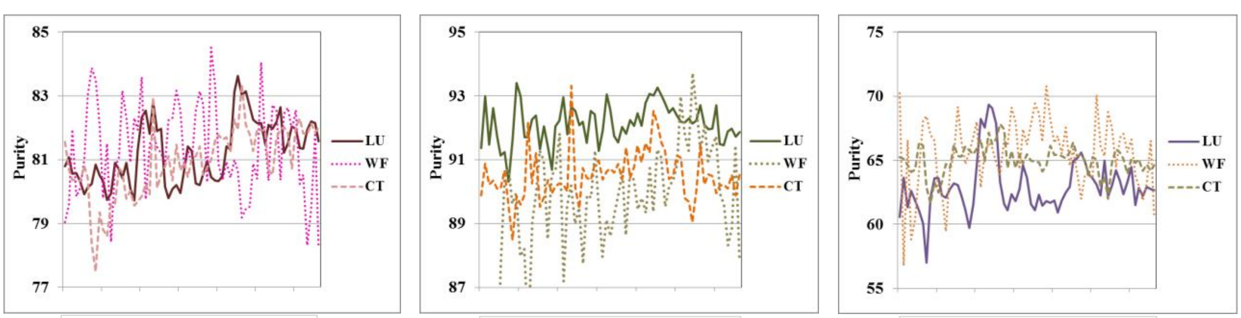
a) LULA – 12,000 TCD

b) WESTFIELD – 13,500 TCD

c) CORA-TEXAS – 18,000 TCD

Figure 3.24 Boiling house single (3B) and double purge (DP) of C-magma simulation results for installed/required (I/R) ratio of high grade (HG) and low grade (LG) pans versus syrup purity for a) Lula, b) Westfield and c) Cora-Texas

Figure 3.25 show daily variations of purity for the double purge system at each factory. Lula operated the system controlling the second magma purity around 92; the purity of the first magma was controlled around 82 to avoid the problem of excess B molasses and; the purity of the double purge molasses was around 65. Lula and Westfield controlled the second magma purity around 90 and first magma around 82.



a) C-Magma

b) DP-magma

c) DP-molasses

Figure 3.25 Daily average variation (daily factory report, 2013) for purities of double purge magma (DPM), first magma (CM) and double purge molasses (DPML) on Lula (LU), Westfield (WF) and Cora-Texas (CT)

Figure 3.26 shows the daily variation and composite weekly variation on whole color (1.2 µm, pH 8.5) for those factories with double purge of C-magma. At Cora-Texas, adjustments on performance of the double purge system in the middle of the season helped in reducing color of the raw sugar. Whole color was on average 1,200 CU at Lula, 1,500 CU at Westfield and 1,600 CU at Cora-Texas after parameter adjustments (First magma Brix and purity).

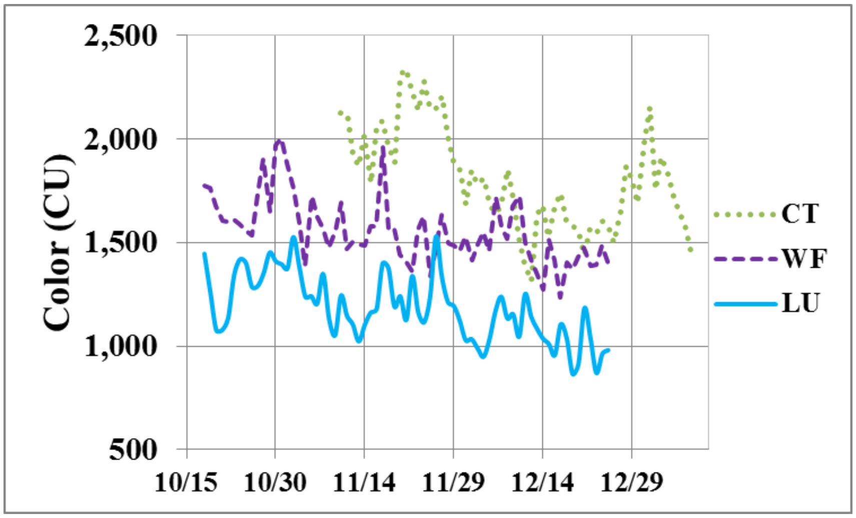
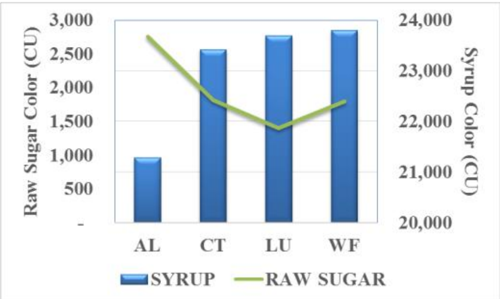
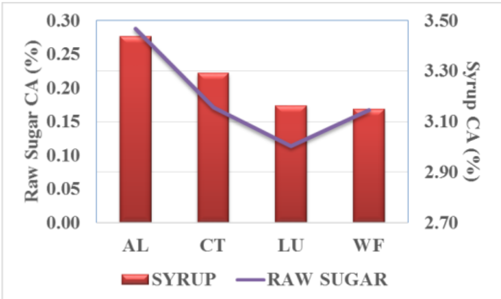


Figure 3.26 Daily raw sugar whole color (8.5pH and 1.2 µm) variation at Lula (LU), Westfield (WF) and Cora-Texas (CT) during 2013 sugarcane crop season with double purge of C-magma

The improvement in whole color of raw sugar was also evaluated by comparing syrup and raw sugar weekly composite samples for the three factories with double purge system with an additional factory (Alma Plantation at Lakeland, Louisiana) operating with single purge of C magma. Figure 3.27 shows the average results obtained from the analysis of the weekly composite samples. Compared to Alma, it can be seen that Lula improved raw sugar color and conductivity ash from 50% to 62% while the improvement for Cora-Texas and Westfield for both parameters were 36% and 41%.



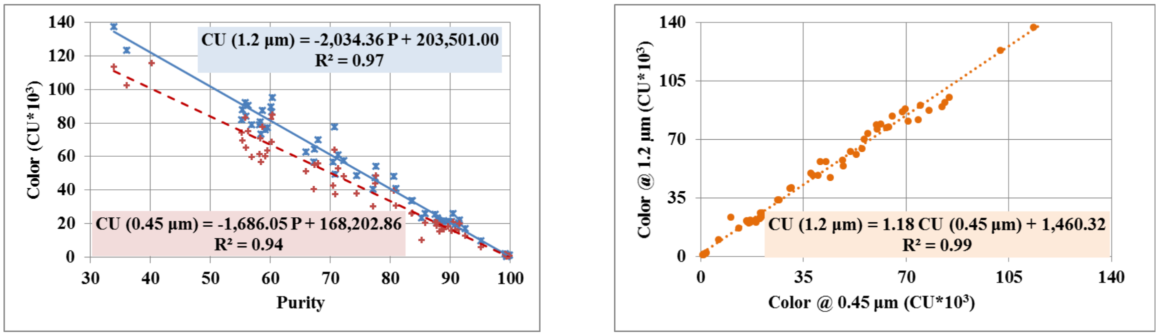
a) Raw sugar and syrup color



b) Raw Sugar and syrup conductivity ash

Figure 3.27 a) Raw sugar and syrup color (pH 8.5, 1.2 µm, CU) and b) raw sugar and syrup conductivity ash – CA (%) for factories with double purge of C-magma (Lula-LU, Westfield-WF and Cora-Texas-CT) compared to a factory with single purge (Alma-AL). Means from 9 weekly composite sample analysis

In general, the correlation of color in solution with the purity of the material is very high. Measurement of color in solution can be used to estimate the amount of colorants in each stream and also can be used for mass balances. Figure 3.28 shows correlations between purity and color of filtered solutions (pH 8.5), at 0.45 µm and at 1.2 µm, and the correlation between color measurements filtered through 0.45 µm or 1.2 µm membrane filters.



a) Color (1.2 and 0.45 µm) versus purity

b) Color at 1.2 µm versus color at 0.45 µm

Figure 3.28 Correlations for Color (8.5 pH) in solution: a) color at 0.45 µm and at 1.2 µm versus purity b) color at 0.45 µm versus color at 1.2 µm for boiling house process streams (58 samples)

Figure 3.29 shows the changes in color/non-sucrose ratio from the beginning to the end of the crystallization process and the differences between factories (one grab sample of each product per factory). Crystallization temperature, pan heating surface area and material circulation at the pan can cause color formation and account for different ratios between factories (assuming no sucrose destruction).

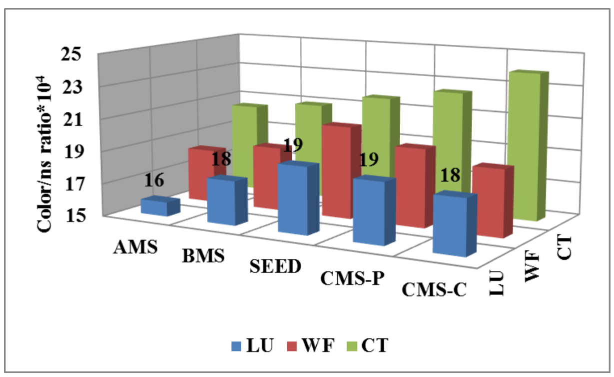


Figure 3.29 Color/non-sucrose ratio profiles for massecuites (MS): A, B and C (P-pan and C-crystallizer) at Lula (LU), Westfield (WF) and Cora-Texas (CT)

Figure 3.30 shows also a profile of conductivity ash % non-sucrose for Lula and Westfield. Comparing to the color/non-sucrose ratio, there was no significant variation in the conductivity ash % non-sucrose. Considering that if the conductivity ash clustered all the inorganic impurities and color also groups the colored impurities, using the conductivity ash profile as a reference, it is confirmed that there is color formation during the crystallization process.

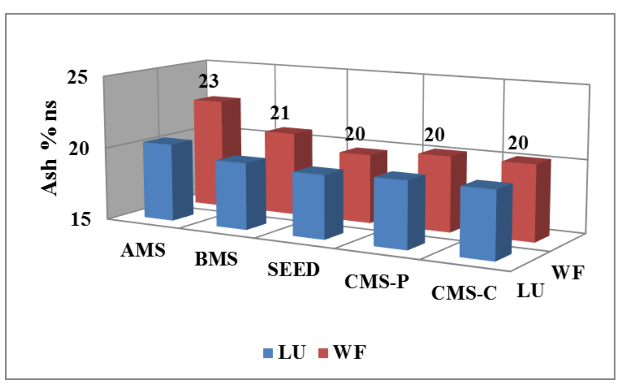


Figure 3.30 Conductivity ash % non-sucrose profiles for massecuites (MS): A, B and C (P-pan and C-crystallizer) at Lula (LU), Westfield (WF) and Cora-Texas (CT)

Figure 3.31 shows the correlation between syrup and high grade massecuites (A and B) color/non-sucrose ratio. The correlation highlights the importance on the quality of the cane as a major factor for raw sugar quality.

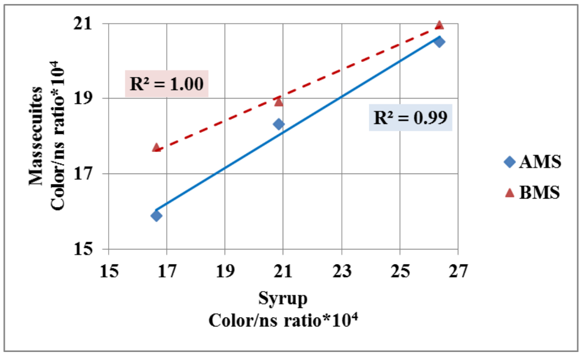


Figure 3.31 Color/non-sucrose ratio on syrup and high grade massecuites (A and B) correlations

3.3.4. Implementation Remarks

The evaluation of the data collected on implementation of double purge at Louisiana raw sugar factories corroborates that double purge of C-magma improves (reduces) the concentration of colorants and inorganic components on raw sugar and it also indicates which operational purity ranges for the first magma (80 – 82) and for double purge magma (91 – 93) should be set on the respective centrifuges. Models and gained experience from real factory implementation showed that the double purge system settings can affect flow rates of important process streams and also sugar recovery.

Implementation results obtained on 2013 were already presented on 2014 at the annual meeting of the American Society of Sugarcane Technologists (ASSCT) – Louisiana division (Polanco, Day et al. 2014) and also presented at the Audubon Sugar Institute factory operation seminar at Saint Gabriel (April 22, 2014) and at the 44th Annual Joint Meeting of the American Society of Sugarcane Technologists (ASSCT) at Bonita Springs, Florida (June 19, 2014).

3.4. Factory Data and Sugars™ Model

The boiling house model (Sugars™) simulates the effects of the purity on the magma input (first magma), the magma output (double purge magma) and the recirculation of double purge molasses. It is based predominantly on statistical analysis of factory data from Lula Sugar Factory. However, some required correlations combined data from the other two factories (Westfield and Cora-Texas). The information presented is converted from the English to the International System of unit (SI units).

3.4.1. Syrup Input

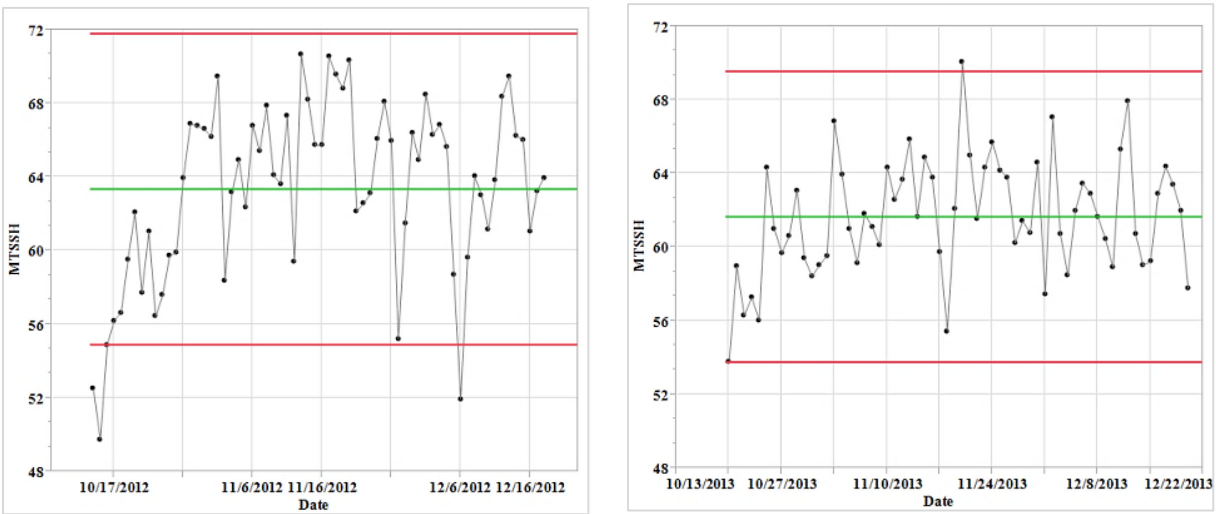
The flow rate of syrup coming from the evaporators to the boiling house is estimated with the data from the daily factory report (Lula Factory) using (equation 3.18),

$$MTSH = MTCH \cdot MJ\%C \cdot \frac{B_j}{B_s} \qquad \text{(Equation 3.18)}$$

- MTSH: syrup mass rate, metric tons of syrup per hour
- MTCH: grinding rate, metric tons of cane per hour
- MJ%C: mixed juice % cane, %
- B_j: soluble solids % juice – juice °Brix
- B_s: soluble solids % syrup – syrup °Brix

Environmental, cultivar and processing conditions vary from one sugarcane crop season to another, therefore, the characteristics of the syrup input to the boiling house change between and within seasons. According to the 2012 and 2013 control charts for Lula (Figure 3.32), the syrup solids input ranged between 56 to 72 metric tons of syrup solids per hour. The soluble solids input are important factors that influence the processing capacity of

the boiling house, affecting its performance (e.g. retention time on pans and crystallizers and non-sugars separation on centrifuges). It is important to determine the purity goals for the first and second centrifugation and the recirculation point as they will change with the change of solids throughput.

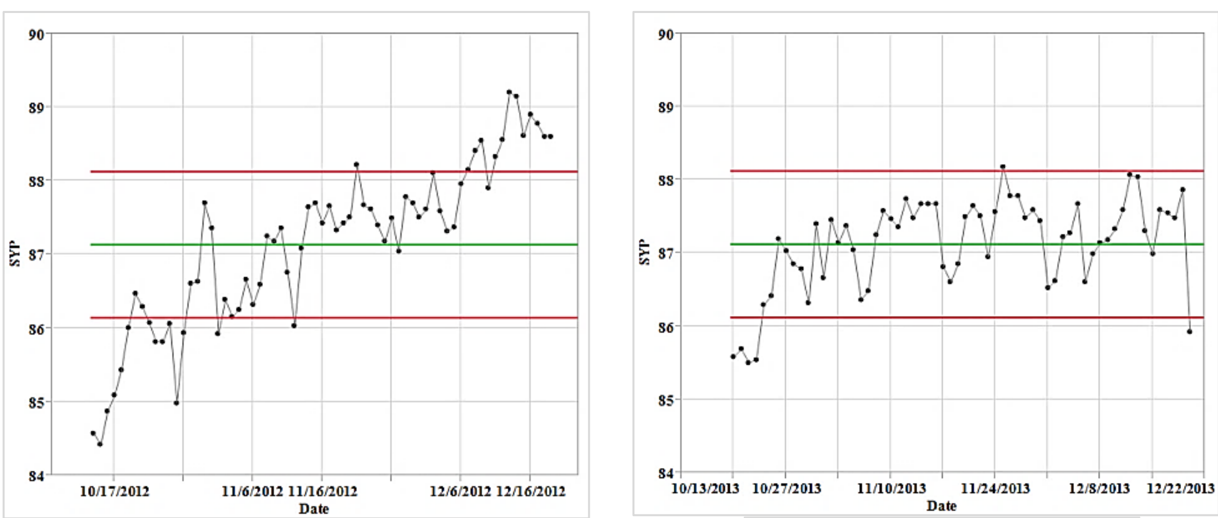


a) Syrup soluble solids rate (Lula-2012)

b) Syrup soluble solids rate (Lula-2013)

Figure 3.32 Control charts for the variability of the daily estimated metric tons of syrup solids per hour (MTSSH) delivered to the boiling house in a) 2012 and b) 2013 sugarcane crop season at Lula Sugar Factory

The concentration of soluble solids and the sucrose concentration define the purity profile of the boiling house, sugar production and the demands on capacity for each stage. High syrup purity demands increased capacity of pans and numbers of centrifuges for the A and B strikes (high grade strikes), low purity syrup demands more capacity in C-pans, cooling crystallizers and numbers of C centrifuges. Commonly, when the season starts the level of maturity of the cane is low, increasing capacity demand for the low grade station (pans, crystallizers and centrifuges). Figure 3.33 illustrates the variability on syrup purity during the 2012 and the 2013 season. The purity of the syrup can go from below 80 to above 90 depending on the quality of the cane; accordingly the settings of the double purge system will change.

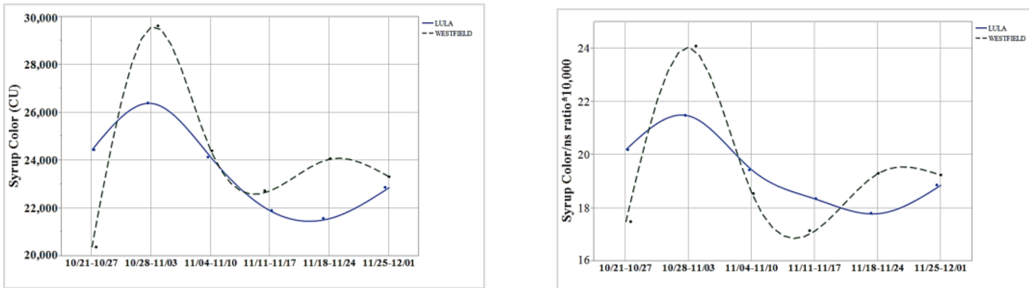


a) Syrup purity (Lula-2012)

b) Syrup purity (Lula-2013)

Figure 3.33 Control charts for the variability of the daily average purity (sucrose % soluble solids) of the syrup delivered to the boiling house in a) 2012 and b) 2013 sugarcane crop season at Lula Sugar Factory

The first or A strike contains the soluble solids coming from the syrup, thus, the colorant fraction of these soluble solids or soluble non-sugars will have an strong effect on the final color of the raw sugar. Intensive research has been done on the partitioning coefficient for non-sugars (impurity in the crystal/impurity in the feed), and especially colored non-sugars (Lionnet 1986; Wright 1996; Lionnet 1998; Bento 2003; Martins, Ferreira et al. 2009). Figure 3.34 a) and b) show changes in the weekly composite syrup color, on solids and non-sucrose basis during the sampling period. Syrup color tends to be high at the start of the season when the cane is more immature. Variations in color from 18,000 CU to 30,000 CU can be expected.



a) Syrup color (CU)

b) Syrup color/ns ratio*10^4

Figure 3.34 Weekly variation a) syrup color (8.5 pH and 1.2 µm) and b) color / non-sugars ratio at Lula and Westfield (composite weekly samples analysis – 2013)

The changes in the solids, sucrose and color input of the syrup sent to the boiling house will affect the factory performance, requiring adjustment of settings for the crystallization stages to preserve quality and achieve good performance. Double Purge system settings also need to be adjusted to achieve good results.

3.4.2. Correlations for Molasses Purities after Centrifugation

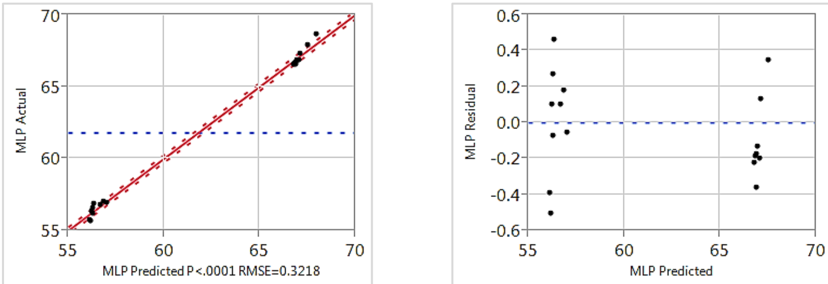
Evaluation of operational variables of C-centrifuges, showed a significant correlation between the purity of final molasses and the purity of the C-sugar, with no effect of the operational variables (e.g. temperatures, rpm, basket diameter, wash). One point rise of C-sugar purity will increase the purity of final molasses on 0.1, causing sugar loss (Jullienne 1987). Carter (1970) suggests that purity of final molasses is related to the purity of the C-massecuite. Though, the crystallization a low purity massecuite produces small crystal size and a wide size distribution, affecting the separation on C-centrifuges (Broadfoot, Miller et al. 1983). The boiling house product parameters such as purities of massecuites and magmas (or sugars) – have a strong effect on crystallization and centrifugal separation operations hence, they become operational parameters that must be optimized.

The boiling house model on Sugars requires input of massecuite supersaturation and product parameters for some streams. The massecuite supersaturation is the ratio of sucrose concentration in molasses (mother liquor around and between sucrose crystals) with respect to the sucrose concentration on a saturated solution at the same massecuite temperature. The supersaturation is affected by the presence of impurities. Supersaturation is estimated by filtration of the massecuite through a Nutsch filter then measuring sucrose concentration on the filtered molasses. Lacking this measurement, it is assumed that when the molasses are exhausted and at the saturation point (supersaturation=1), then the difference between actual sucrose concentration in molasses and sucrose concentration at saturation was a function of the dilution of sucrose crystals (causing purity rise). Assuming a supersaturation of one, the model at the centrifugal stations only requires the purity of sugars and purity of molasses that are separated. The purity of the molasses separated was statistically analyzed (JMP(R) 2014) with respect to product parameters, Brix and purity of the massecuite input and sugar output from the centrifuge. Defining the specification of the sugar output (controlled variable) and having the Brix and purity of the massecuite input to the centrifuge, the purity of the molasses can be estimated from a regression model.

- A and B molasses purity correlation

A correlation was made from statistical analysis of the weekly averages from Lula factory reports between the purities of A and B molasses. Figure 3.35 a) and b) show the actual values and the residuals versus predicted

values for A and B molasses purities. Although the residuals plot shows a concentration of points on both sides of purities of A and B molasses the predicted values are closed to the actual values and are normally distributed. Validating with average 2003 daily values the standard error is ~ 0.05 for A and B molasses purity.



a) Predicted versus actual A + B molasses purity

b) Residuals versus predicted A + B molasses purity

Figure 3.35 a) A and B molasses purity (MLP) actual versus predicted values and b) residuals versus predicted

Table 3.8 shows a summary of the mean and variation range of the data (simple statistics) as well as estimates of the coefficients of correlation for A and B molasses purities. The estimates and the t-ratio values indicate that increasing massecuite purities have a significant effect on increasing the purities of the molasses and that an increase of Brix of the massecuites has a significant effect on reducing the purity of the molasses. The adjusted coefficient of correlation of the model is r^2=0.937 with F-value=334,269.

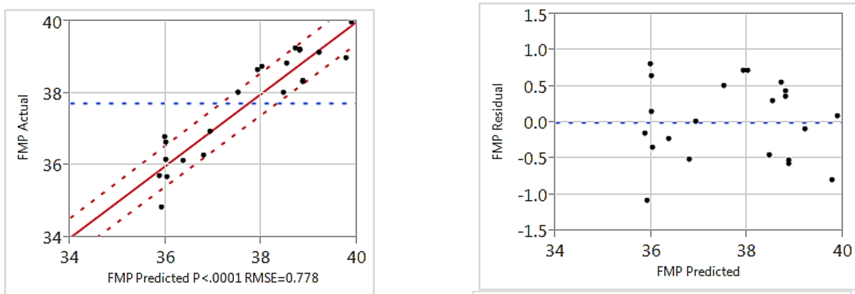
Table 3.8 Statistics summary for the correlation of molasses purity (MLP) with the Brix and purity of massecuites (MSBX and MSP) from A and B strikes

Strike	Statistics	MSBX	MSP	MLP
A	N	9	9	9
A	Mean	91.66	84.91	67.11
A	Minimum	91.49	84.57	66.54
A	Maximum	91.79	85.8	68.66
B	N	9	9	9
B	Mean	93.54	74.36	56.47
B	Minimum	93.44	74.03	55.69
B	Maximum	93.67	74.97	57.02
Model	Estimates	-0.1748	0.9792	
	Standard Error	0.033	0.039	0.344
	t-Ratio	-16.30	78.68	
	p-value	<0.001	<0.001	

- Final molasses purity correlation

Figure 3.36 a) and b) show the fit for the actual values and the residuals versus predicted values for final molasses purities.

Table 3.9 shows the simple statistics and the estimates of the coefficients for the correlation for final molasses purity. The estimates indicate that increases in C massecuite purities and C-magma purities have a significant effect on the final molasses purities, and that by increasing Brix of C-massecuite, the purities of final molasses is reduced. The adjusted coefficient of correlation of the model is r^2=0.952 with F-value=18,827.



a) Predicted versus actual final molasses purity

b) Residuals versus predicted final molasses purity

Figure 3.36 a) Final molasses purity (FMP) actual versus predicted values and b) residuals versus predicted

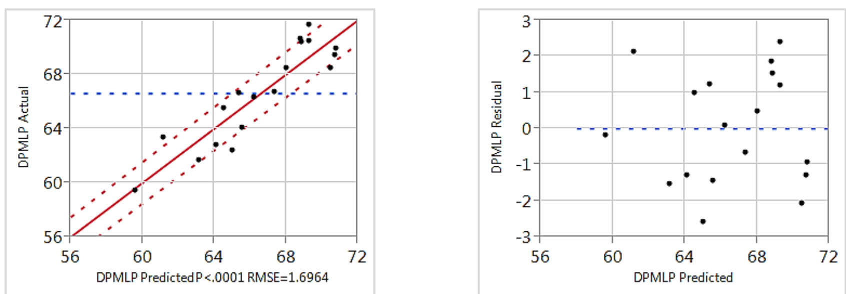
Table 3.9 Statistics summary for the correlation of final molasses purity (FMP) with the Brix and purity of C massecuite (CMSB and CMSP) and purity of the first C magma (MP) for C strikes

Statistics	CMSB	CMSP	MP	FMP
N	24	24	24	24
Mean	93.26	58.44	82.24	37.72
Min	92.10	55.80	75.93	34.83
Max	94.78	60.66	87.68	39.97
Model Estimates	-0.1893	0.8030	0.1027	
Standard Error	0.068	0.087	0.049	0.805
t-Ratio	-2.80	9.24	2.10	
p-value	0.011	<0.001	0.048	

- Double purge molasses purity correlation

Figure 3.37 a) and b) show the fit for the actual values and the residuals versus predicted values for double purge molasses purities.

Table 3.10 shows the simple statistics and the estimates of the coefficients for the correlation for double purge molasses purities. They indicate that increases in the first C-magma purities and double purge magma purities have a significant effect on increasing double purge molasses purity. The adjusted coefficient of correlation of the model is r^2=0.928 with F-value= 6,949.



a) Predicted versus actual double purge molasses purity

b) Residuals versus predicted double purge molasses purity

Figure 3.37 a) Double purge molasses purity (DPMLP) actual versus predicted values and b) residuals versus predicted

Table 3.10 Statistics summary for the correlation of double purge molasses purity (DPMLP) with the Brix and purity of the first C-magma (MB and MP) and the purity of the double purge C magma (DPMP)

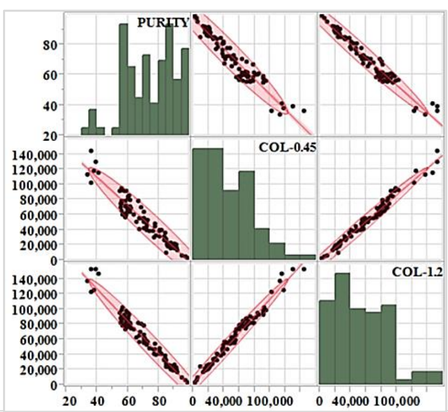
Statistics	MB	MP	DPMP	DPMLP
Mean	91.49	81.18	92.43	66.59
Min	88.11	76.28	88.04	59.41
Max	93.6	85.26	97.46	71.71
Estimate	-1.5746	0.4675	1.1134	
Standard Error	0.395	0.193	0.281	1.953
t-ratio	-3.99	2.42	3.96	
p-value	0.001	0.030	0.001	

3.4.3. Boiling House Purity and Color Profiles

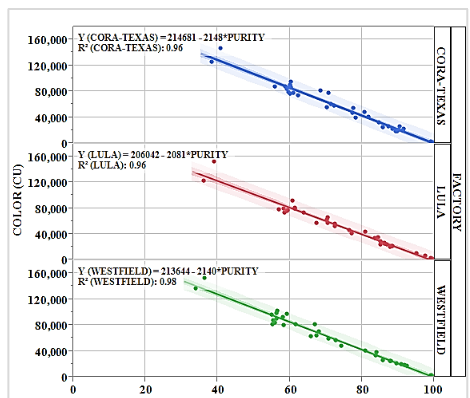
During the 2013 sugarcane season, six sets of boiling house samples were analyzed for purity and color. Table 3.11summarizes the statistical analysis. Figure 3.38 a) and b) show the correlations between color and purity. Sucrose crystallization is an efficient purification process; consequently, as the sucrose is removed from solution non-sucrose components become more concentrated. The correlation between color measurement at 0.45 μm and 1.20 μm is 0.995. Since a refinery protocol for raw sugar whole color refers to filtered solutions through 1.20 μm membrane filter and pH adjusted to 8.5, this was the measurement applied for modelling, simulation and optimization.

Table 3.11 Statistics for the tests perform on all boiling house process streams at 3 Louisiana Sugarcane Factories

Analysis	N	DF	Mean	Minimum	Maximum
Purity	113	112	75.08	33.91	99.61
Color @ 0.45 μm, 8.5 pH	113	112	44,718	560	144,408
Color @ 1.20 μm, 8.5 pH	113	112	52,101	710	152,789



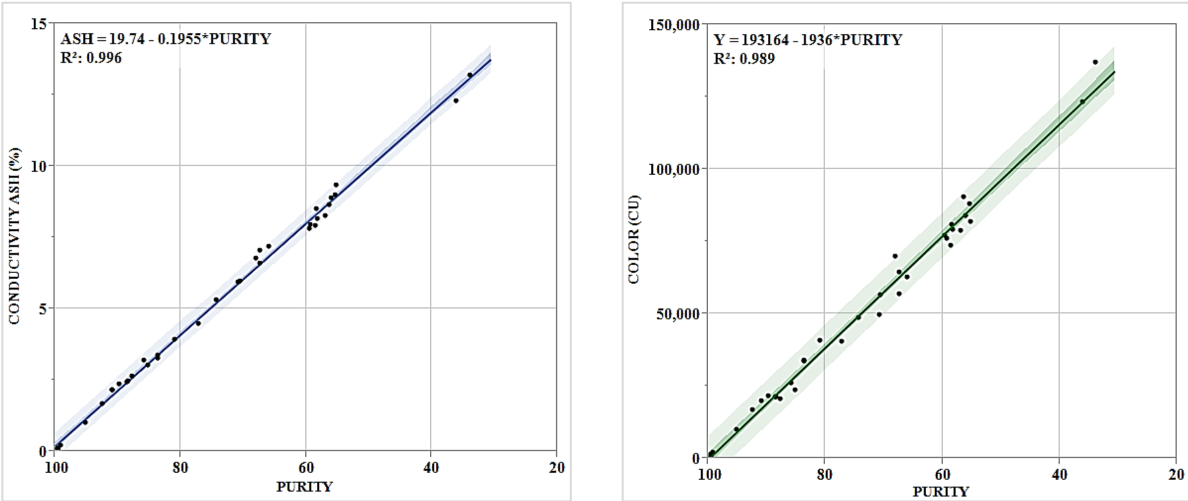
a) Scatter plot boiling house color and purity profile



b) Correlation plot for color (CU) versus purity

Figure 3.38 a) Scatter plots and histograms for the boiling house purity and color profiles @ 0.45 μm (COL-0.45) and @ 1.20 μm (COL-1.2) and b) color @ 1.20 μm (Y) correlated to the purity of the process stream for each factory (Cora-Texas, Lula and Westfield)

Figure 3.39 a) and b) shows a correlation between the conductivity ash and color versus purity of the material. This shows that the color measurement represents the concentration of colored compounds and behaves in the same manner as the conductivity ash, which includes inorganic compounds present in the non-sucrose fraction.

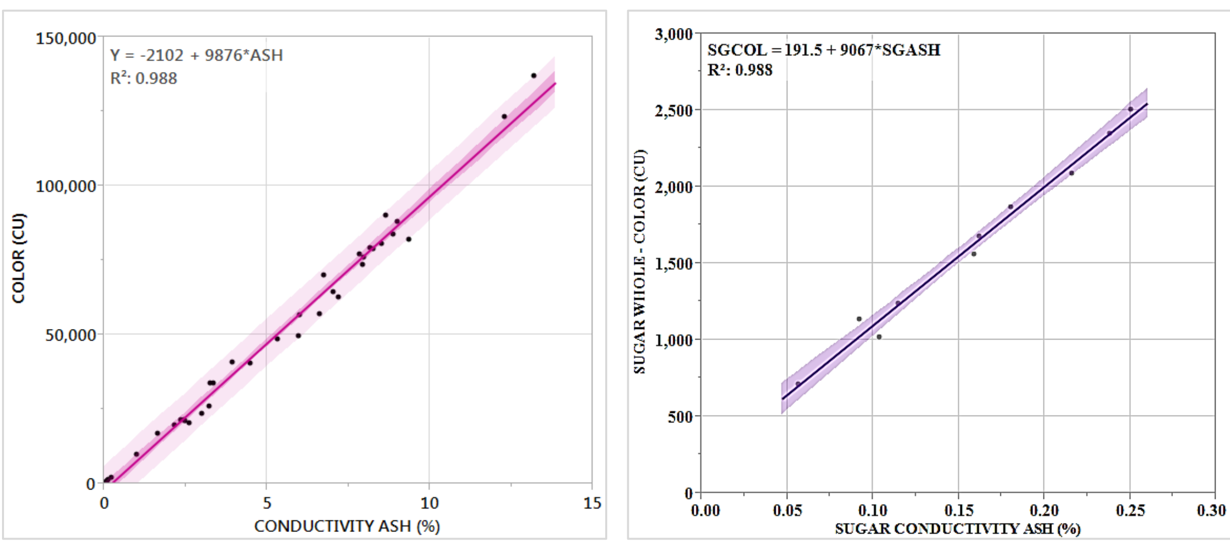


a) Correlation plot for conductivity ash versus purity (inverse purity axis)

b) Correlation plot for color (CU) versus purity (inverse purity axis)

Figure 3.39 a) conductivity ash and b) color in solution versus purity (reverse scale) of boiling house process streams from Lula and Westfield (37 samples)

Figure 3.40 a) and b) illustrate a correlation between the ash represented by the conductivity ash measurement and color represented by the 'color in solution' measurement (Beer-Lambert Law). Color in solution is a good estimation of the concentration of colored compounds on each process stream.



a) Correlation plot for color (CU) versus conductivity ash (%) – boiling house materials

b) Correlation plot for color (CU) versus conductivity ash (%) – A + B sugars

Figure 3.40 Conductivity ash and color regression models for a) all boiling house process streams (37 samples) from Lula and Westfield and b) A and B sugars (10 samples) from Lula, Westfield and Cora-Texas

The color profiles determined at Lula and Westfield (two sampling for each factory) were modeled using Sugars™ matched with the average values of the sampling date (sugar quality and production reports), to find the percentage of color increase at each stage and the color separation on centrifugation of A and B sugars. Statistically (t-test), comparing the measured and modeled color profiles there was no significant differences (Table 3.12). Figure 3.41shows the linear fit between the model and the actual values.

Table 3.12 Statistic matched pair's difference analysis comparing color measured for each stream and color for each stream given by the Sugars' models (4 models)

Color Profile Measured – Mean	62,762	**t-Ratio**	0.14
Color Profile Modeled – Mean	62,708	**DF**	43
Mean Difference	53	**Prob > |t|**	0.89
Standard Error	388	**Prob > t**	0.45
N	44	**Prob < t**	0.55
Correlation	0.997		

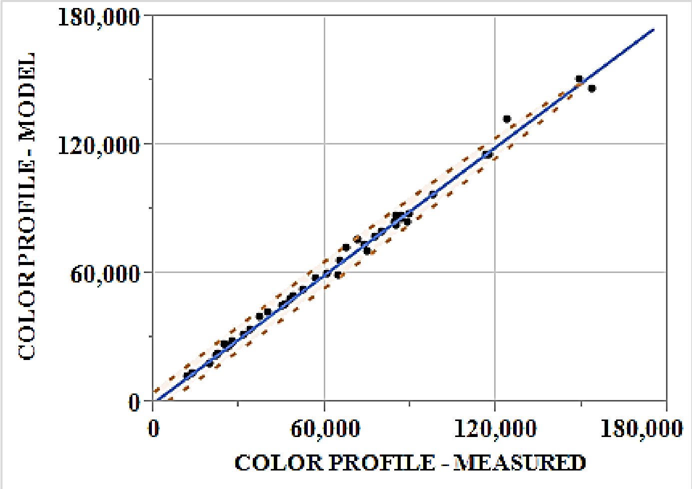


Figure 3.41Bivariate fit of color profile obtained with the Sugars^{TM} models (4) and the color profile observed

The most important information obtained from the models created, using the actual color measurements, is an estimation of the minimum and maximum percentage of color increase for each strike (Table 3.13). The color rise includes all the stages, from each strike in the pans, to the discharge from the centrifuges. Studies performed in South Africa reveal a large variability in color formation and color transfer from factory to factory, depending on crystallization parameters and syrup quality (Smith 1990; Radford 1996). The average color rise (ICUMSA, pH 7 and 0.45 µm filtered solution) per strike reported in South Africa is 11% on A-Strikes (range: -5% to 24%), 18% on B-strikes (range: 1% to 35%) and 20% on C-strikes (range: 3% to 41%) (Getaz and Bachan 1989).

Table 3.13 Minimum and maximum percentage of color rise determined matching color measured (pH 8.5 and 1.20 µm filtered solution) for each stream with the models created on Sugars^{TM} for Lula, Westfield and Cora-Texas

Strike	Minimum	Maximum
A	0	1.5
B	0.5	3.5
C	15	23

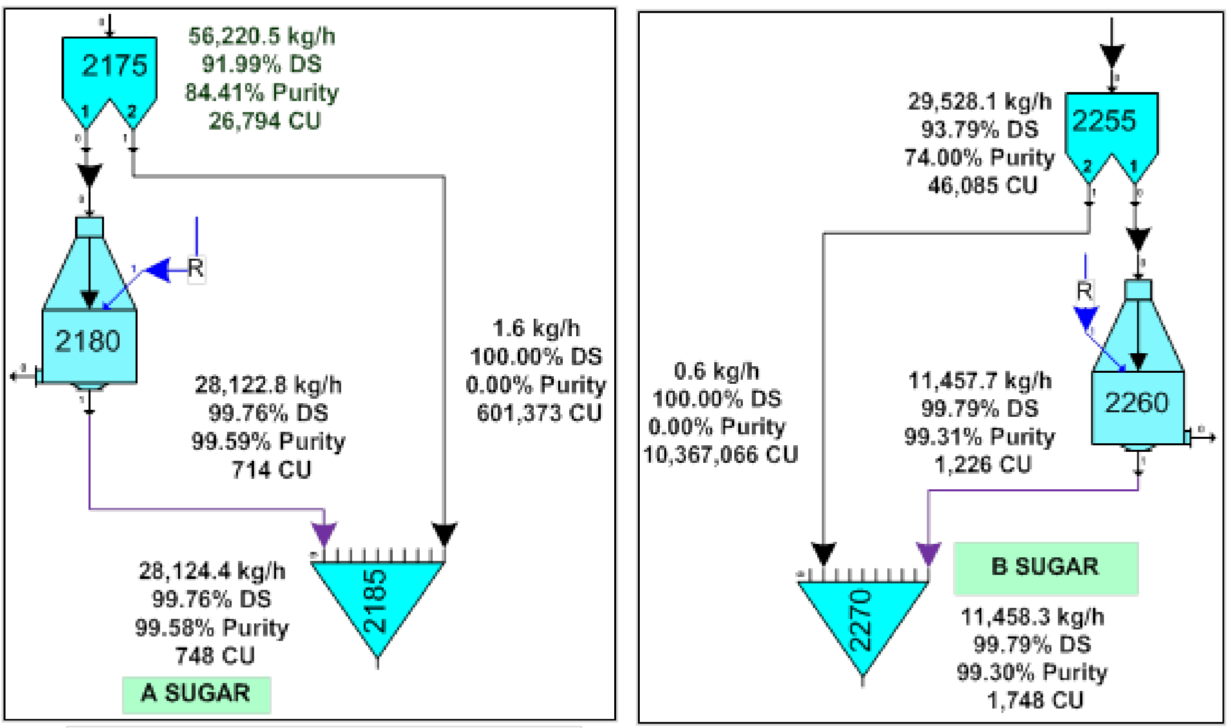
The addition of a color separator before the centrifuge station for A and B strikes, avoids introducing false high color formation to match raw sugar whole color. For instance, the total color rise assumed on the initial models for Sterling (3.3.1) was ~ 247% while, from measured color profiles models the total color rise is between 23 – 30% respect to the color input with the syrup.

3.4.4. Correlation for Color Separation in A and B Centrifuges

The use of an attenuation index or absorptivity (extinction coefficient) to represent the mass of colorants for mass balance purpose and to evaluate color formation and factors affecting color at the boiling house has been reported in several publications (Chiu and Sloane 1980; Jullienne 1984; Lionnet 1987; Getaz and Bachan 1989; Smith 1990; Schick 1994; Radford 1996; Wright 1996; Doherty and Wright 2001; Wright 2002).

Chiu and Sloane (1980) stated that besides the quality of the syrup, the crystal yield or exhaustion (based on purities) and the operational variables on each crystallization stage have a significant effect on sugar quality. The purity of C-magma which determines the recirculation of non-sugars, because of the final molasses layer surrounding the crystal, has been reported to have effects on the quality of sugar (Staunton 1938; Stevenson 1964; van Hengel 1983; Jullienne 1989). Stevenson (1964) compared color index of sugar crystals made using low magma purity (78-80) and high magma purity (90-93) as a crystallization seed for three Australian factories. On average the color index was ~35% lower for the sugar crystals made with high purity magma, with a correlation coefficient between -0.893 and 0.979 at a significance level of 0.1%.

A template model was created according to the color profiles determined from sampling and analysis. To adjust the boiling house model for color separation on A and B centrifuges, a 'color separator' was added before the centrifuge station. This adjustment avoids forcing a high color rise at each stage to match the whole color of the raw sugar. Settings for the color separator were determined from the actual color measured for each stream. The color separator sends a small amount (~0.01%) of non-sugars soluble solids, that are mainly color (~99%), to the sugar, by-passing the centrifuge station. Figure 3.42 illustrates the application of a color separator station before the a) A centrifuge station and b) B centrifuge station to model the whole color of raw sugar. Appendix A shows a complete boiling house model for Lula Sugar Factory.



a) Color separation unit (2175) for A-sugar

b) Color separation unit (2255) for B-sugar

Figure 3.42 Sugars™ model sections for a) color separation station 2175 inserted before centrifuge station 2180 and blending station 2185 to model whole color of A sugar and, b) color separation station 2255 inserted before centrifuge station 2260 and blending station 2270 to model whole color of B sugar. Raw sugar is the blend of A and B sugars on a three-boiling crystallization scheme

Table 3.14 summarizes the mass and color balance for A sugar and B sugar calculated by the Sugars™ model for Lula on December 2, 2013. Color rise on A strike is 1% and on B strike is 1.5% of the total color feed to each strike. The ratios for whole color on sugar per color on massecuite are around 0.015 and 0.016 for A and B sugars.

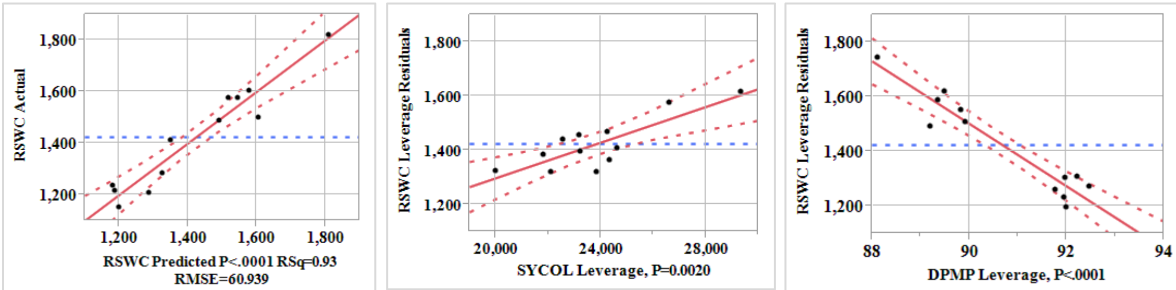
Table 3.14 Mass and color balance for A strike and B strike at Lula Sugar Factory according to color profile measured 12/02/13. Color units (CU) = cm^2/kg dry substance (Sugars™)

	Stream	Mass	°Brix	Purity	Color	Color*10^{-2}	Color*10^{-6}
	Source	**kg/hour**	**% solids**	**%**	**(CU)**	**/NS**	**/hour**
	Syrup	86,674.0	57.1	86.6	22,231	1,664	110,023
A-STRIKE	**A-feed**	81,222.7	59.1	83.7	27,521	1,690	132,041
	DP-magma seed	4,061.2	92.1	93.3	13,799	2,053	5,161
	A-massecuite	56,220.5	92.0	84.4	26,794	1,719	138,571
	A-sugar - 2180	28,122.8	99.8	99.6	714	1,623	2,003
	A Separator - 2175	1.6	100.0	0.0	601,373		96
	A-sugar - 2185	28,124.4	99.8	99.6	748	1,781	2,099
	Color A-Sugar % Color A-Massecuite:						**1.5%**
B-STRIKE	**B-feed**	38,473.1	67.8	72.8	47,303	1,740	123,407
	DP-magma seed	1,744.1	92.1	93.3	13,799	2,053	2,217
	B-massecuite	29,528.1	93.8	74.0	46,085	1,773	127,630
	B-sugar - 2260	11,457.7	99.8	99.3	1,226	1,777	1,402
	B Separator - 2255	0.6	100.0	0.0	10,367,066		622
	B-sugar - 2270	11,458.3	99.8	99.3	1,748	2,497	1,999
	Color B-Sugar % Color B-Massecuite:						**1.6%**
	Raw Sugar	39,582.7	99.8	99.5	1,038	2,076	4,099
	Color Raw Sugar % Color Syrup:						**3.7%**

Weekly composite analysis of syrup, raw sugar and final molasses (molasses survey – Audubon Sugar Institute) combined with the averages from routine analysis and boiling house performance from Lula and Westfield reports were used to balance mass and color using a boiling house model created using Sugars™. Boiling house models assume a constant % color rise on each strike determined from boiling house profiles for each factory. The % color rise used on each model for Lula and Westfield is: 1% and 0% for A strikes, 2% and 0% for B strikes and, 20% and 5% for C strikes respectively for each factory. It was necessary to use data from both factories to increase the range of variability of the studied product parameters. This is justified since Lula and Westfield processed sugar cane from the same sources and raw sugar samples are analyzed at the Westfield laboratory.

Figure 3.43 (a, b and c) shows that raw sugar whole color is predicted by the color of the syrup and the purity of the double purge magma. The leverage plots are basically residuals scatter plots adding the mean of the Y and X variables and, they are used to evaluate the individual effects – X on the response – Y (JMP(R) 2014). The leverage plots indicate that the whole color of raw sugar is significant and linear related to the color input with the syrup (99.8% probability) and the purity of the double purge magma (~ 100% probability) used as a seed to grow raw sugar crystals.

Figure 3.44 (a, b) shows inverse predictions with a confidence interval of 95% of the required purities of double purge magma depending on the specified raw sugar whole color by the color input with the syrup. Notice if the syrup color increases from 20,000 CU to 28,000 CU, the purity of the double purge magma has to be raised from ~92 to ~94 in order to produce raw sugar with 1,200 CU whole color. The estimated average color reduction is ~7% for each point increase on purity of the double purge magma.

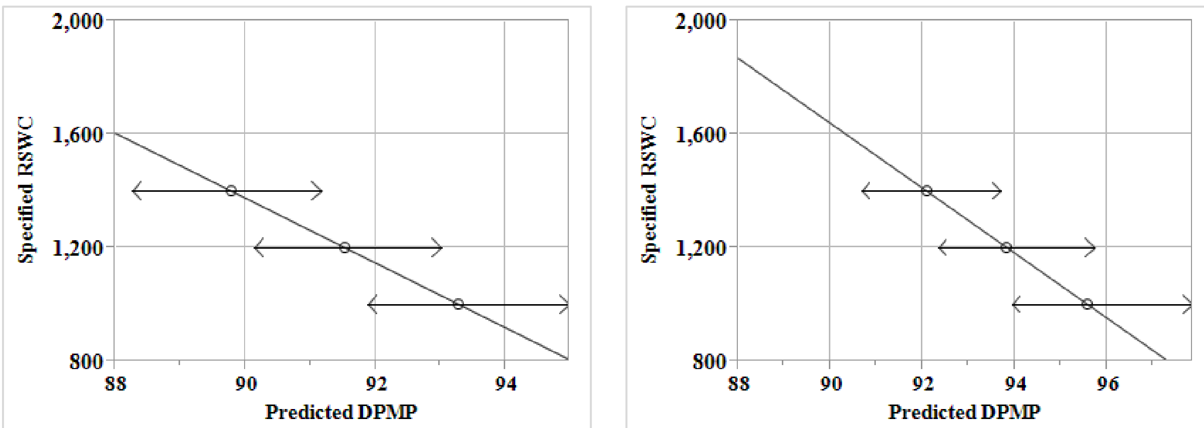


a) Predicted versus actual raw sugar whole color

b) Leverage plot: whole color versus syrup color

c) Leverage plot: whole color versus DP magma purity

Figure 3.43 a) Predicted plot for raw sugar whole color (RSWC) with a coefficient of correlation r^2=0.93, b) leverage plot of whole color versus syrup color (SYCOL) and c) leverage plot of whole color versus purity of double purge of C-magma. Weekly boiling house models created using Sugars™



a) Predicted DP magma purity versus whole color for SYCOL=20,000 CU

b) Predicted DP magma purity versus whole color for SYCOL=28,000 CU

Figure 3.44 Specified raw sugar whole color – RSWC versus predicted double purge magma purity – DPMP by color on syrup a) 20,000 CU and b) 28,000 CU (95% confidence interval). Adjusted R^2=0.912, RSM (root square mean error) = 61 CU

From the point of view of sugar refiner's, affined color of raw sugar has more importance than whole color. Depending on the contract, the price of raw sugar is penalized when whole color is above the upper limit and a fixed premium (% of contract price) is given when the whole color is below the low limit. In the same way, the contract penalizes if the affined color is above the upper limit but, when the whole color and affined color are below the lower limits the raw factory receives premiums for whole color and a % premium for affined color that increases in steps according to how low is the affined color of the raw sugar (ICE 2008). These premiums favor and motivate the raw factory to improve or lower the color of raw sugar. For instance, in the case of Lula, if the low limit for affined color is 750 CU, the whole color target has to be ~ 1,200 CU in order to receive both color price premiums (Figure 3.45)

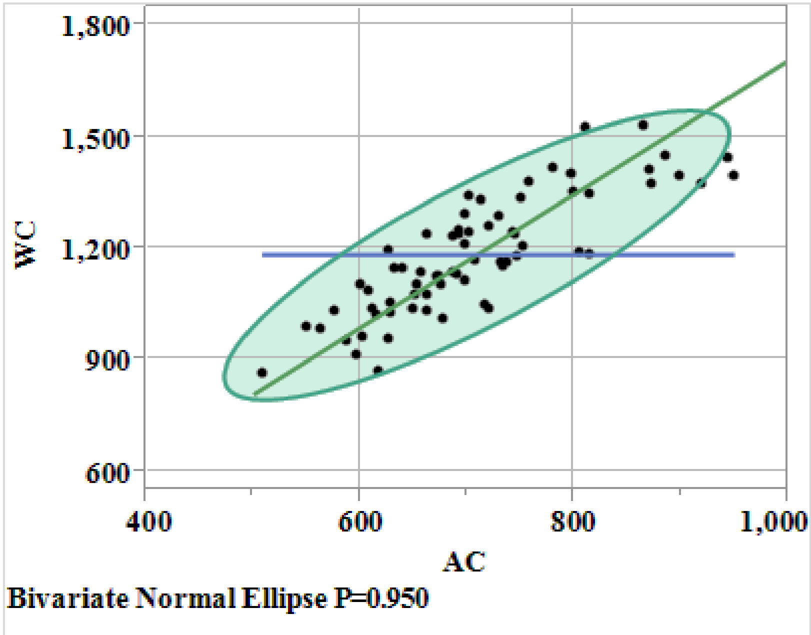
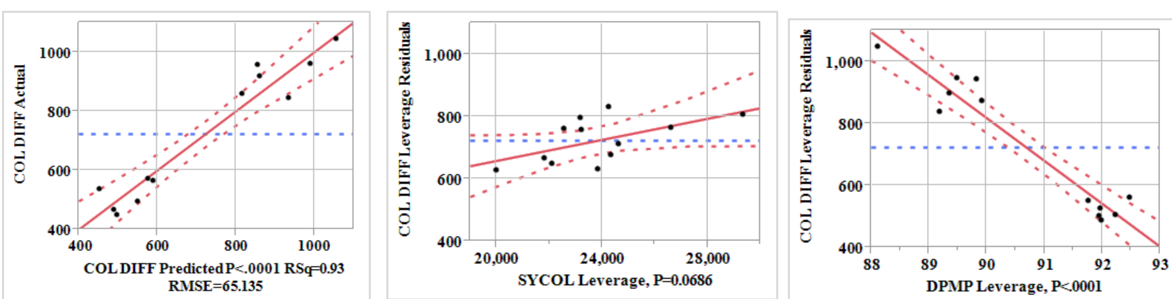


Figure 3.45 Raw sugar whole colors (RSWC – CU) versus affined colors (RSAC – CU) from daily quality report Lula factory 2013 season. Correlation: 0.84 and standard error of 19 CU for 69 raw sugar samples

Figure 3.46 (a, b and c) show the prediction and the importance of double purge magma purity in the color difference (whole color minus affined color), which approximately represents the color on the external layer of the raw sugar crystal. It is expected that most of the color of the raw sugar is located on the external layer and is attributed more to syrup (white sugar crystallization), however, for the three- boiling scheme using C-magma as a seed, the purity of the magma has a more significant effect on the color of the outer shell crystal layer than the color of the syrup, indicated by the slopes of the fit lines on the leverage plots. The probability of syrup color affecting the color difference is now 97% compared to 99.8% for the whole color correlation.



a) Predicted versus actual color difference	b) Leverage plot: color difference versus syrup color	c) Leverage plot: color difference versus DP magma purity

Figure 3.46 a) Predicted plot for the color difference between raw sugar whole color and affined color (COL DIFF), b) leverage plot for the effect of syrup color (SYCOL) and c) leverage plot for the effect of the purity of double purge magma (DPMP). Weekly boiling house models created using Sugars^{TM}

Table 3.15 summarizes the mean, minimum and maximum values of double purge magma purity and percentage of color separation use to model weekly data from Lula and Westfield sugar factory. Figure 3.47 shows a scatter plot with a fit line for the correlation between double purge magma purity and the average color separation. This correlation was used for simulations.

Table 3.15 Simple statistics values for purity of double purge magma (DPMP) and the percentage of color separation for A and B sugars and the average color separation used to adjust color of raw sugar on Sugars™ model

Statistics	DPMP	A	B	AVG
Mean	90.68	99.378	99.366	99.372
Min	88.33	98.780	98.780	98.780
Max	92.53	99.810	99.700	99.700

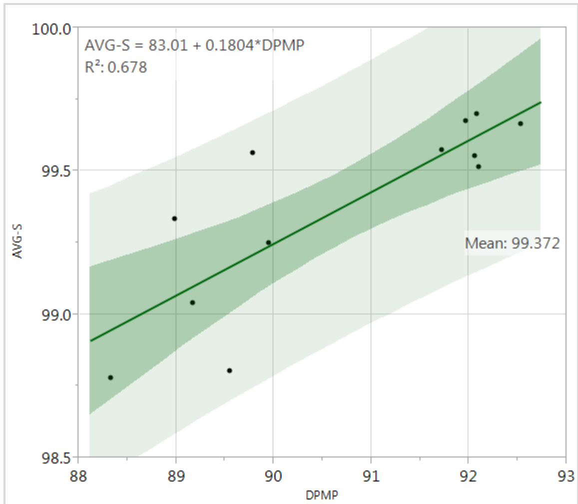


Figure 3.47 Line fit for the average percentage of color separation (AVG-S) versus purity of double purge magma (DPMP)

3.5. Conclusions

Color improvements up to 47% were achieved with the implementation of double purge of C-magma integrated to a three-boiling crystallization scheme in three factories. Models were used to estimate volume rates used for system design and to check the installed capacity of the boiling house. Initial models were improved adding a separation color station before centrifuges to account for the color located at the outer layer on the crystal surface that is correlated to the purity of the double purge magma. Through color profiles from three factories, it was found that the highest color rise is at the low grade station (from C pan through C centrifuges) of approximately 20% and, that the total color rise is about 30% compared to the 250% assumed on the earliest models. Statistical analysis showed that raw sugar whole color is correlated to both syrup color and purity of the double purge magma. Fixing raw sugar color to a target value, the purity required for the double purge magma will change from 90 to 92 depending on the color of the syrup. In addition, a significant correlation was found between whole and affined color and that a whole color target of ~1,200 CU will help raw factories obtain a better price for the sugar according to the premiums (additional premium for lower affined color) established on the sugar contract with the refinery.

From initial models, it was expected that the implementation of double purge, recirculating the wash molasses to B molasses, would reduce by ~3% the capacity requirements at the high grade station (volumes of A and B strikes) and will increase by ~8 % the required capacity of the low grade station. This lower volume requirement for high grade strikes and at lower magma purity on the first magma purge, will allow achievement of a higher sugar yield from A and B strikes, improving sugar recovery. This statement was not confirmed with the actual data but it is recognized that on real operation, multiple factors change randomly and hide the effects of the double purge on factory performance and on energy requirements. Also from implementation experience, it was found that the recirculation of wash molasses to A molasses resulted on an easier boiling house management compared to recirculation to B molasses. The success of the implementation generated questions about the effects of purity combinations for the first and the double purge magma with respect to processing volumes, changes on grinding rate through the season and changes on the quality of the syrup during the harvesting season or on other seasons. Simulations, experimental design, surrogate models for the responses and multiple criteria or response optimization is the strategy applied to visualize the effects and a better approach to the possible changes that may occur from one season to another.

The goals for the next stage on this research (see Chapter 4) were to find the optimal values of the purities of first and double purge magma when the operational conditions change and the effect of the recirculation of the wash double purge molasses to A molasses and to B molasses. Expected changes in processing conditions are quality of cane (purity and color) and solids throughput at the boiling house caused by variation on grinding rate or on solids concentration on the sugar cane juice.

CHAPTER 4 MULTIPLE RESPONSE OPTIMIZATION TO THE INTEGRATION OF DOUBLE PURGE OF C-MAGMA TO A THREE-BOILING CRYSTALLIZATION SCHEME

4.1. Introduction

For a complete integration of a second centrifugation of C-magma (or double purge) it is required to optimize product parameter combinations at different possible processing scenarios. The boiling house objectives selected for this optimization are: to match a target raw sugar whole color, maximize sugar recovery, and minimize required capacity and energy in response to changes during a harvest season. To this end, a multiple objective optimization strategy is applied to the multistage crystallization process. It is a complex process environment where multiple factors are changing simultaneously; additionally the delay between the input and output, from syrup to final molasses (3-5 days), makes it practically impossible to perform controlled experiments at a factory scale to determine optimal double purge system settings (first and double purge magma purities and recycle point). Hence, a boiling house model, built using a specialized sugar industry software (Sugars^{TM} 2014), and correlations derived from sampling and analysis of the actual double purge of C-magma implementation were applied to simulate 'virtual experiments'. This software solves the boiling house model iteratively with an accuracy of 0.01%. The model is basically deterministic – the outputs of two replicates are the same. Experimental design of simulation experiments and analysis and construction of the models is the hub of the applied optimization strategy (Kleijnen, van den Burg et al. 1979; Goethals and Cho 2011). The followed optimization strategy is based on stepwise modelling and response surface methodology – RSM. Each stage of the optimization strategy pursues specific objectives:

1. Data collection to relate the effects of controlled parameters on quality and performance: making assumptions, defining correlations, fixing parameters values, defining variation range of the controlled and random parameters and, selecting responses
2. Simulation and experimental design to screen and effectively understand the system behavior (sensitivity) by prediction of the responses (surrogate models)
3. Metamodeling to make a valid representation of individual responses (surrogate models) with respect to a set of inputs, using data from simulations and statistical tools
4. Multiple objective optimization with desirability functions to combine several responses into a single objective function assigning a selected importance or weight to each response.

The goodness and validity of the multiple response optimization results are linked to the applied strategy: model settings and boundaries, experimental design for the inputs, statistical analysis and fit of simulation outputs (surrogate models) and the optimization approach. The goal is to obtain practical results that can help to understand parameters effects and interactions that can be used, either to establish settings for a double purge system, or as a troubleshooting guide to the boiling house operation, in order to improve sugar color and boiling house performance.

4.1.1. Response Surface Methodology (RSM) and Surrogate Models

Response surface methodology (RSM) employs a group of techniques for determining the empirical relationships between inputs factors and system responses and identifying factors which maximize or minimize the response (Tekin and Sabuncouglu 2004; Baş, Arslan et al. 2010). RSM techniques were initially used for optimization of real-world chemical processes, applying experimental designs for screening which require fewer experiments than full factorial designs (Box and Wilson 1951; Kleijnen 2007). Full factorial 2^k and fractional factorial $2^{(k-p)}$ (p indicating the fraction) like the Plackett-Burman are the most common experimental designs used to explore or screen surface response (Simpson, Peplinski et al. 2001). Experimental designs were developed to run and statistically analyze agricultural experiments about 1930, and have been used for engineering and psychological experiments since ~1950 (Kleijnen 2005).

A computer simulation can be considered a controlled experiment that is performed to understand a complex system (Whitt 2006). Simulations are applied in many research fields for different purposes, such as validation and verification of a model, sensitivity or "what if" analysis, risk analysis and optimization (Kleijnen 2007). Still, computer simulations can be expensive with regard to computer resources and time. Hence, experimental designs are required to make the application of computer simulations more efficient and effective

(Simpson, Peplinski et al. 2001; Cavazzuti 2013). The lack of random error in computer simulations, which means that the responses from several replicates are the same (deterministic), implies a different approach for experimental design and on the model fitting (Birnbaum 1959; Kleijnen and Sargent 2000; Brenneman and Nair 2001; Bingham and Chipman 2007; Kleijnen 2007). Figure 4.1 shows the representation of an input/output process represented by a complex simulation model (black-box). The surrogates are models of the simulation model relating the objective responses to the controlled parameters of interest.

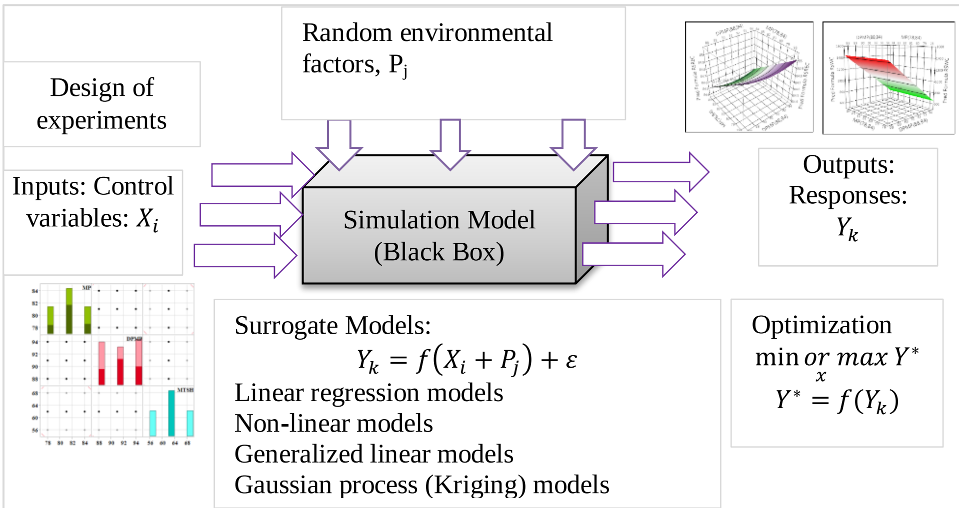


Figure 4.1 Response surface methodology analogy to metamodeling on computer simulation for multiple-response optimization. Surrogate models mimic the responses pattern respect to the inputs with a more simple input/output empirical relation compared to the simulation model – black-box (Kleijnen 2007)

The solution of a deterministic computer simulation model does not depend on random variation but rather on the input factors and variation ranges (Johnson, Montgomery et al. 2012; Cavazzuti 2013). Randomization, replication and blocking that are considered the foundations for traditional experimental designs are not important for computer simulations. Instead, space-filling designs like fractional factorials, Latin hypercube and orthogonal arrays which have more exploration of the design space (domain), are more appropriate for selecting the inputs for simulations (Crombecq 2011). Computer aided experimental designs, like optimal designs, are based on previous knowledge of the shape of the response, guiding the search for an optimal to an identified region of interest – exploitation (Crombecq 2011). The algorithm initially searches for candidate samples using the full factorial experimental design and a model goal (e.g. finding estimators for main factors and second order interactions – RSM linear-polynomial model), searching for those sets of samples which minimize the objective function – optimality criteria (Cavazzuti 2013). These designs are efficient for minimizing either the variance of the parameter estimates (D-Optimal) or the variance of the predicted response (I-Optimal) in the design space (Steinberg and Hunter 1984; Kuhfeld, Tobias et al. 1994; Cavazzuti 2013). I-optimality criterion is the appropriate choice for finding the best operating conditions or the region in the design space where the range of variation of the response is acceptable – optimization (SAS 2014). The goodness of an experimental design is evaluated in terms of the model goals which are to reduce the variance of estimators and predicted responses and the bias due to confounding and model misspecification (Montgomery and Jones 2011).

Surrogate models are used to mathematically represent the relationship between the input and the output in order to a) understand, b) predict, c) optimize or d) verify and validate the behavior of the system or process simulated. Linear regression models (polynomials) and Gaussian process models are the most common model approximations used for computer simulations (Johnson, Montgomery et al. 2012). Surrogate or approximation models are important for industrial applications of computer simulations for factors exploration and optimization. The strategy applied for experimental design and analysis is an important key for computer simulations used for sensitivity analysis and optimization (Rossouw, Coetzer et al. 2010).

Depending of the curvature of the response surface, the preferred surrogate model is a low-order linear polynomial (Kleijnen 2007). A first order polynomial for low curvature and a second order polynomial for greater curvature are the most popular models for response surface methodology – RSM (Simpson, Peplinski et al. 2001).

Equation 4.1 shows the model of a quadratic polynomial response surface for p factors. Equation 4.2 shows the basic linear model in matrix notation (Simpson, Peplinski et al. 2001; Cavazzuti 2013).

$$y(x)=\beta_0+\sum_{i=1}^{p}\beta_i x_i+\sum_{i=1}^{p}\beta_{i,i}x_i^2+\sum_{i=1}^{p-1}\sum_{j=i+1}^{p}\beta_{i,j}x_i\,x_j+\varepsilon \qquad \text{(Equation 4.1)}$$

$$Y_{n\times 1}=X_{n\times p}\beta_{(p-1)\times 1}+\varepsilon_{n\times 1} \qquad \text{(Equation 4.2)}$$

$$\begin{bmatrix} Y_1 \\ Y_2 \\ \vdots \\ Y_n \end{bmatrix}=\begin{bmatrix} 1 & X_{1,1} & X_{1,2} & \dots & X_{1,p-1} \\ 1 & X_{2,1} & X_{2,2} & \dots & X_{2,p-1} \\ \vdots & \vdots & \vdots & \ddots & \vdots \\ 1 & X_{n,1} & X_{n,2} & \cdots & X_{n,p-1} \end{bmatrix}\begin{bmatrix} \beta_0 \\ \beta_1 \\ \vdots \\ \beta_{p-1} \end{bmatrix}+\begin{bmatrix} \epsilon_1 \\ \epsilon_2 \\ \vdots \\ \epsilon_n \end{bmatrix}$$

- $Y_{n\times 1}=$ responses column vector
- $X_{n\times p}=$ design matrix
- $\beta_{(p-1)\times 1}=\hat{\beta}=$ regression coefficients or least-squares estimators column vector
- $\varepsilon_{n\times 1}=$ column vector of errors ~ $N(0,\sigma^2)$

The regression coefficients are estimated by the least squares method. Equation 4.3 shows the calculation in matrix notation where the “prime” ()' indicates the transpose of the matrix and the subscript (-1) indicates the inverse of the matrix (X'X), which is called the information matrix.

$$\hat{\beta}=(X'X)^{-1}X'Y \qquad \text{(Equation 4.3)}$$

The predicted response is represented in matrix notation by equation 4.4 where H is called the “hat matrix”. The hat matrix relates the predicted values $\hat{Y}$ with the observed values Y. Equation 4.5 shows also in matrix notation the calculation of residuals ($\hat{r}$).

$$\hat{Y}=X\hat{\beta}=X(X'X)^{-1}X'Y=HY \qquad \text{(Equation 4.4)}$$

$$\hat{r}=Y-X\beta=Y-\hat{Y}=(1-H)Y \qquad \text{(Equation 4.5)}$$

Deterministic data does not have a random component and the residuals from the model fit do not measure the noise, rather the residuals indicate what the bias is at a determined point. On surrogate models from computer simulations, the pattern of the residuals may also indicate the necessity to add new terms to reduce bias. The meaning of the low p-values and very high F-statics in metamodeling is that the variation is explained by a model term (SAS 2014). Hamada and Balakrishnan (1998) reviewed the literature about the analysis of unreplicated factorial experiments which can be applied for deterministic computer simulations. The half-normal probability plot (Daniel 1959) and the “pseudo error estimate” – PSE (Lenth 2006) rely on the “effect sparsity” concept (few effects are active) which is applied to determine the effects or factors on unreplicated experiments. Model adequacy can be tested by the R-square, adjusted R-square, residuals pattern, validation of the model with different data points and leave-one-out cross validation (Simpson, Peplinski et al. 2001). There is controversy about the use of statistical methods for fitting models from computer simulations because of the lack of random error; nonetheless, “if actual data is in agreement with predicted data why not to use it” (Simpson, Peplinski et al. 2001). Design and analysis of computer experiments or simulation experiments (DACE or DASE) publications give the strategies and methods that can be applied to deterministic and random (pseudo random input variables) computer simulations (Simpson, Peplinski et al. 2001; Chen, Tsyum et al. 2003; Kleijnen 2007; Montgomery and Jones 2011; NIST/SEMATECH 2013; Ramu and Prabhu 2013).

In this research, the surrogate models for optimization are built using JMP® response screening analysis for modelling which allows the inclusion of categorical parameters (SAS 2014). The R-square compares the variation of the response with the variation given by the model; a value close to 1 indicates that the responses are closely predicted by the model. The adjusted R-square is used to avoid over fitting the model by the comparison of two models with different numbers of effects. When adding an additional effect (main or interaction), if the adjusted R-square takes a lower value than before the addition, then this indicates over fitting the model. The root mean square (RMSE) estimates the standard deviation of the random error. The Lenth’s pseudo-standard error (PSE) as well as

the half-normal probability plots (un-replicated experiments) helps identify the active effects. Pareto plots evaluate the importance of the regression coefficients (estimators) by relating the absolute value of the orthogonalized and standardized estimators to the sum of all estimators' absolute values. The assessment of variable importance uses methods that are independent of the type of model or the fitting methodology and indicates how much a factor contributes (alone or combined with other factors) to the variation of the response. This assessment allows organizing the factors according to importance relative to the total effects for multiple responses. In general, the default significance level (α) used to evaluate effects and model fit is 0.05 (SAS 2014). Statistical analysis and graphical evaluation to create and test the adequacy of the surrogate models are performed using the JMP software tools (JMP$^{(R)}$ 2014).

4.1.2. Multiple Response Optimization Problem

Optimization techniques can be applied to multiple fields from science, engineering, finances and social fields where the information is numerical or can be transformed to numerical values and can be represented by a mathematical expression. By definition, optimization is a methodology applied to find the values of a set of variables ($x_1, x_2..., x_n$) that makes a mathematical function – $f(x_1,x_2\ldots,x_n)$ the objective function, to reach a minimum or maximum value subject to some constraints – $g(x_1,x_2\ldots,x_n)$ (Deb 2010). A single optimization problem solution can be expressed by equation 4.6 where *f* is called the objective function and *g* is the constraint (bounds, equality or inequality functions).

$$\min/\max_{x_i} f(x_i) \qquad \text{(Equation 4.6)}$$
$$\text{Subject to } g(x_i) \geq 0$$

For a linear differentiable function, the variables values making the first derivative of the objective function zero ($f'(x_i) = 0$) locate the stationary points of the function on the research space – valleys or hills (minimum or maximum). Then, if the same values at the stationary point making the second derivative of the objective function positive (or negative), it means that there is a minimum (or maximum) value of the function at that location ($f''(x_i) > 0 \rightarrow$ minimum or $f''(x_i) < 0 \rightarrow$ maximum) (Dennis and Schnabel 1996).

The basic steps for finding the optimal set of values are first, to define the starting point and the direction of the search (minimum – descent or maximum – ascent), then define the step size and compare new with old values and the procedure is repeated until the optimization criteria is reached (Babu 2004). There are diverse optimization techniques to find local and global optima. Numerical gradient methods have been traditionally used to find local optima, which are the minimum or maximum values found in a small neighborhood. However, most real systems are characterized as being high dimensional with possible combinations of continuous and categorical variables. The objective function of a real system can be discontinuous and not differentiable and, it can have multiple local optima. The search for the global optimal requires multiple trials starting on different points inside the research space. Heuristics and metaheuristic methods are applied for global optimization (Babu 2004; Tekin and Sabuncouglu 2004). Tekin and Sabuncouglu (2004) give a list (Table 4.1) of techniques used for local and global optimization, the new techniques for global optimization combine traditional and metaheuristics methods – hybrids.

Table 4.1 Local and global Optimization techniques for computer simulations (Tekin and Sabuncouglu 2004)

Local Optimization		**Global Optimization**
Discrete	**Continuous**	
Ranking and selection	Response surface methodology	Tabu search
Multiple comparison	Finite difference estimates	Simulated annealing
Ordinal optimization	Perturbation analysis	Bayesian / sampling algorithms
Random search	Frequency domain analysis	Gradient surface method
Simplex/complex search	Likelihood ratio estimates	Evolutionary algorithms
Single factor method	Stochastic approximation	
Hooke-Jeeves pattern search		

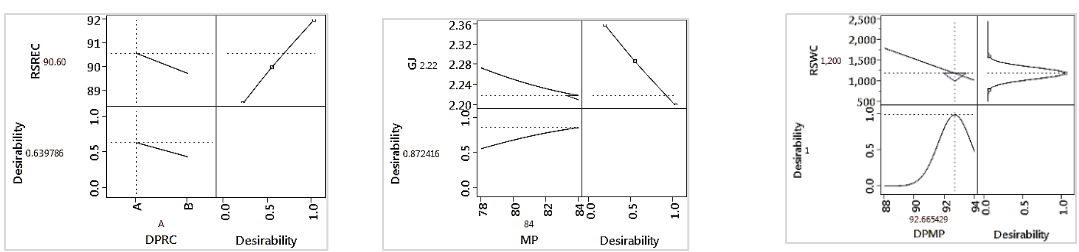
The approach of response surface methodologies (RSM) to multiple response optimization problems is to combine the models for selected responses into a single scalar value or cost function and solve the problem as a single objective optimization (Park and Kwang-Jae 2005; Baş, Arslan et al. 2010). For multiple response optimization, the objective function of one response may oppose with other objective responses and may also

involve several constraints, generating multiple optimal solutions (Deb 2010). The desirability function is a transformation of each predicted response model $Y_i(x)$ to particular functions $d_i(Y_i(x))$, whose solution values range between 0 and 1 (0 ≡ undesirable and 1≡ ideal response) depending on the response value of $Y_i(x)$ and the 'desired' objective of the response (Harrington 1965). For example, one-side transformations (equation 4.7) are applied for maximization and minimization response objectives (higher-the-better and smaller-the-better) and two-side transformations (equation 4.8) are applied when the objective is to match a target value (Derringer and Suich 1980). Y_{max} and Y_{min} are the maximum and minimum values of the response and T is the target value. The parameters r, s, and t are chosen to adjust the shape of the function.

$$d_i\big(Y_i(x)\big)=\begin{cases} 0 & \text{if } Y_i(x)\leq Y_{min-i} \\ \left(\frac{Y_i(x)-Y_{min-i}}{Y_{max-i}-Y_i}\right)^r & \text{if } Y_{min-i}\leq Y_i(x)\leq Y_{max-i} \\ 1 & \text{if } Y_i(x)\geq Y_{max-i} \end{cases} \qquad \text{(Equation 4.7)}$$

$$d_i\big(Y_i(x)\big)=\begin{cases} \left(\frac{Y_i(x)-Y_{min-i}}{T_i-Y_{min-i}}\right)^s & \text{if } Y_{min-i}\leq Y_i(x)\leq T_i \\ \left(\frac{Y_i(x)-Y_{min-i}}{T_i-Y_{max-i}}\right)^t & \text{if } T_i\leq Y_i(x)\leq Y_{max-i} \\ 0 & Y_i(x)<Y_{min-i} \text{ or } Y_i(x)>Y_{max-i} \end{cases} \qquad \text{(Equation 4.8)}$$

Giving a weight or importance (w_i) to each response, a geometric mean of the individual desirability functions is used to determine the total desirability – "overall objective function" (Del Castillo, Montgomery et al. 1996). The desirability function was initially developed by Harrington (1965) and then the functions were modified by Derringer and Suich (1980). The desirability function approach has been widely used on research for multiple response optimization problems (Obermiller 1997; Kim and Dennis 2000; Kros and Mastrangelo 2001; Ribardo and Allen 2003; Ful-Chiang 2005; Park and Kwang-Jae 2005; Bera and Mukherjee 2010; Bera and Mukherjee 2012; Salmasnia, Baradaran Kazemzadeh et al. 2012; Midi, Mustafa et al. 2013). SAS (2014) uses differentiable, smooth, piecewise desirability functions shaped to fit user defined control points to maximize, minimize or achieve a target value. Figure 4.2 shows the individual desirability plots that represent the optimization goals at the design space.



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

Figure 4.2 Desirability plots a) maximize sugar recovery, b) minimize heat requirements and c) match a target value on raw sugar whole color (JMP$^{(R)}$ 2014)

Equation 4.9 shows the expressions to calculate the geometric mean in order to estimate the "overall desirability" (D*) when all k numbers of individual desirability $d_i(Y_i(x))$ have the same importance. Equation 4.10 applies a logarithmic transformation to both sides of the desirability equation 4.9. Equation 4.11 is applied when each response has a defined importance or weight w_i. The optimization ultimate goal, is to maximize the total desirability (D*) equation 4.12 (Ramsey, Stephens et al. 2005).

$$D^*=\sqrt[k]{d_1\cdot d_2\cdot\cdots\cdot d_k} \qquad \text{(Equation 4.9)}$$

$$D=\ln\big(D^*\big)=\frac{1}{k}\left[\ln(d_1)+\ln(d_2)+\cdots+\ln(d_k)\right] \qquad \text{(Equation 4.10)}$$

$$D=\left[w_1\ln(d_1)+w_2\ln(d_2)+\cdots+w_k\ln(d_k)\right] \qquad \text{(Equation 4.11)}$$

$$\max_x D^*=\max_x e^D \qquad \text{(Equation 4.12)}$$

- $0\leq w_i\leq 1.0$ and $\sum_{i=1}^{k} w_i=1.0$

JMP algorithm uses a constrained Newton's method approach with step shortening to find the maximum desirability for continuous factors while, for categorical factors the approach is a greedy optimization algorithm taking one variable at a time. When the optimization problem contains both continuous and categorical factors, the JMP algorithm combines the two approaches together with multiple random starts to escape from local optimal points. The JMP algorithm adjusts itself the steps of the search according to the problem and the pattern of the optimization output (Vorburger 2014).

4.2. Methodology

The purpose of this research is to employ a methodology that has greater potential applications in order to understand the effects of product parameters and how they interact with the random inputs (environment) for any sucrose crystallization scheme. The core of the methodology is the definition of the optimization problem and the limitations. Figure 4.3 shows a sketch of the optimization strategy. The strategy is adaptive going back and forward to improve the model responses.

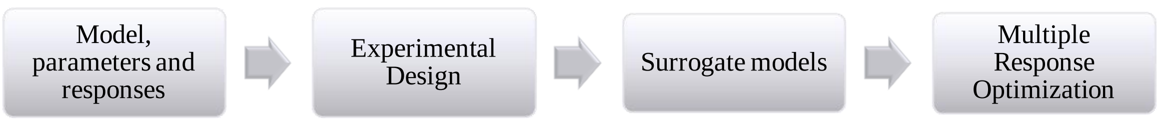


Figure 4.3 Strategy approach to optimize the integration of a double purge system to improve whole raw sugar color on a traditional three boiling scheme

4.2.1. Model Parameters and Responses

The management of the boiling house is well defined for each factory modeled. For the integration and optimization of the double purge system, the model is built utilizing the data from one particular sugar factory – Lula Sugar Factory. Variation ranges and fixed values for the majority of the parameters are defined using historical factory data (Table 4.2).

Table 4.2 Boiling house simulation (Sugars^{TM}) input parameters: type, fixed values and levels, and the parameter abbreviation label

Parameters Summary			**Simulation**	**Parameter**
Product	**Parameter**	**Type**	**Values**	**Abbreviation**
Syrup	Metric Tons Sol/hr	Random	56 - 62 - 68	MTS
	Brix	Fixed	62.5	SB
	Purity	Random	82 – 85 – 88	SP
	Color*10^3	Random	18 – 23 – 28	SYCOL
A Massecuite	Brix	Fixed	92.0	AMSB
	Purity	Covariate	83 - 86	AMSP
	% Color Rise	Random	1 – 3	CRA
	DP-Magma%AMS	Covariate	10 – 20	DPM/AMS
B Massecuite	Brix	Fixed	94.0	BMSB
	Purity	Fixed	74.5	BMSP
	% Color Rise	Random	1 – 3	CRB
	DP-Magma%BMS	Covariate	5 – 10	DPM/BMS
C Massecuite	Brix	Fixed	96.0	CMSB
	Purity	Covariate	56 - 58	CMSP
	%Color Rise	Random	20 - 25	CRC
	G % CMS	Covariate	18 – 22	G/CMS
Grain	Brix	Fixed	89.0	GB
	Purity	Fixed	60.0	GP
Magma	Brix	Random	92 – 93 - 94	MB
	Purity	Controlled	78 – 81 - 84	MP
DP-Magma	Brix	Fixed	92.5	DPMB
	Purity	Controlled	88 – 91 - 94	DPMP

Table 4.2 continued

Parameters Summary			Simulation	Parameter
Product	**Parameter**	**Type**	**Values**	**Abbreviation**
A & B Sugar	Brix	Fixed	99.7	RSB
	Purity	Fixed	99.5	RSP
	°Z	Fixed	99.2	RSPOL
A Molasses	Brix	Fixed	72.0	AMLB
	Purity	Covariate	64 - 70	AMLP
B Molasses	Brix	Fixed	72.0	BMLB
	Purity	Covariate	54 - 58	BMLP
DP-Molasses	Brix	Fixed	72.0	DPMLB
	Purity	Covariate	58 - 70	DPMLP
	Recycle (A – B)	Controlled	0 , +1	DPRC
Final Molasses	Brix	Fixed	82.0	FMLB
	App. Purity	Covariate	34 - 38	FMLAP
P/S= ~0.86	True Purity	Covariate	40 - 44	FMLTP

The boiling house responses with respect to raw sugar quality and system performance are:

Y_1: Raw Sugar Whole Color (RSWC) = Color Units (CU) on final Raw Sugar. It is desired for this optimization objective to meet a Target ~ 1,200 CU

Y_2: Sugar Recovery (RSREC) = Sucrose out with raw sugar/sucrose in with syrup (equation 4.13). It is desired for this optimization objective to be the higher the better (maximize)

$$RSREC=\frac{w_{sugar}*B_{sugar}*P_{sugar}}{w_{syrup}*B_{syrup}*P_{syrup}}*100 \quad \text{(Equation 4.13)}$$

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

Y_3: Giga Joules per metric ton of syrup solids (GJ) = latent heat input with total vapor per syrup solids input used at the boiling house (equation 4.14). It is desired for this objective function to be the smaller the better (minimize)

$$GJ=\left(\frac{w_{vapor}\cdot L_P}{w_{syrup}*B_{syrup}}\right)*10^{-3} \quad \text{(Equation 4.14)}$$

- w : weight or mass-rate (kg/hour)
- B : Brix/100 (solids fraction)

- L_P: latent heat of vapor (P=177.2 kPa – evaporators vapor I) = 2,212.27 kJ/kg

Y_4: High Grade Volume Ratio (HGV) = Installed/Required capacity ratio [I/R] for A, B and magma development strikes (equation 4.15 and Table 4.3). It is desirable for this optimization objective to be the higher the better (maximize)

Y_5: Low Grade Volume Ratio (LGV) = Installed/Required capacity ratio [I/R] for C, grain development and grain strikes (equation 4.15 and Table 4.3). It is desirable for this optimization objective to be the higher the better (maximize)

$$HGV\ OR\ LGV=\frac{I}{R}=\frac{I}{Q_A\cdot t_A+Q_B\cdot t_B+Q_C\cdot t_C+Q_{Seed}\cdot t_{Seed}} \quad \text{(Equation 4.15)}$$

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

Table 4.3 Strike cycle times and operational installed capacity of high and low grade batch pans – Lula Sugar Factory (Birkett 2011)

	High Grade Pans			**Low Grade Pans**		
	A Strike	**B Strike**	**Magma Development (Seed)**	**C Strike**	**Grain Development (Seed)**	**Grain Strike (Seed)**
Cycle Time, t (hours)	2.5	2.8	2.5	5.0	5.0	4.0
Installed Cap., I (m^3)	218			93		

4.2.2. Design of Experiments, Surrogate Models Fit and Optimization

The first stage toward defining the inputs and the number of simulations for optimization (design of experiments – DOE) is to establish which practical questions that need to be answered about the system.

The hypothesis for designing and performing the simulations (experiments) for optimal integration of a double purge of C-magma system is as follows: It was confirmed from factory implementation analysis, that purity of double purge magma has a significant effect on the raw sugar color. Simultaneously, the combination of purity of double purge magma and purity of the magma fed to the second centrifugation, dictate the amount of solids and sucrose in solution that is recycled with the wash molasses. The amount, composition and returning point for the wash molasses affects processing volumes, total sugar recovery and heating requirements in the boiling house. The importance of each variable (purities in and out of the magmas and the recirculation point of wash molasses) is different depending on solids throughput and the quality of the syrup fed to the boiling house. Hence, there is an optimal combination for double purge system recirculation and product parameters which satisfy the combined requirements of the most important responses with respect to raw sugar color and boiling house performance indices.

The first stage to design the experimentation is to define which set of product parameters that need to be controlled and which responses are critical in terms of raw sugar color and boiling house performance for the integration of a double purge of C-magma system. It is known that random or uncontrolled parameters may affect or interact with the control parameters for each response. This requires definition of the scope and boundaries of the process space, including assumptions, fixed parameters, range of variation of controlled and random variables. Initially, in order to understand the behavior of the raw sugar whole color response with respect to different inputs, two level factorial designs on SAS and JMP, were used to define the required simulations and to analyze the results. Figure 4.4 shows a block diagram of the final two-level full factorial design.

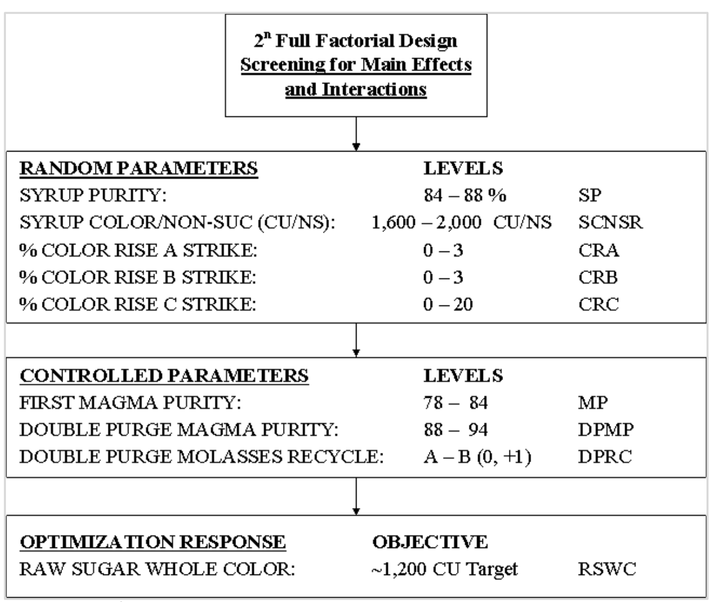


Figure 4.4 Block diagram for the 2^8 full factorial (256 simulations) experimental designs to screen for main factors and interactions for the optimization of double purge integration using SAS

An analysis of the results of two level full factorial designs (256 simulations) indicate the importance of these factors and also give the approximate pattern of the raw sugar whole color response and the patterns for other relevant responses. The knowledge gained from the first simulation experiment was used to refine subsequent simulations. The last sets of simulations were determined using (JMP(R) 2014) custom experimental design, because it has features that allow using and generating random parameters, choosing the type of optimal design for model prediction (I-Optimal) and a possibility for augmenting these designs (SAS 2014). The design space was separated into three scenarios, for low, medium and high syrup purity, and an I-Optimal design was selected for model prediction, detecting main factors and second order interactions (RSM), adding center points for a total of 257 simulations. Figure 4.5 illustrates the random and controlled parameters used for each I-Optimal design. The weight or importance of each response was assigned, with higher importance assigned to raw sugar whole color and sugar recovery, and lower importance for heat and for high and low grade capacity ratio's, for a total sum of weights of 1.0.

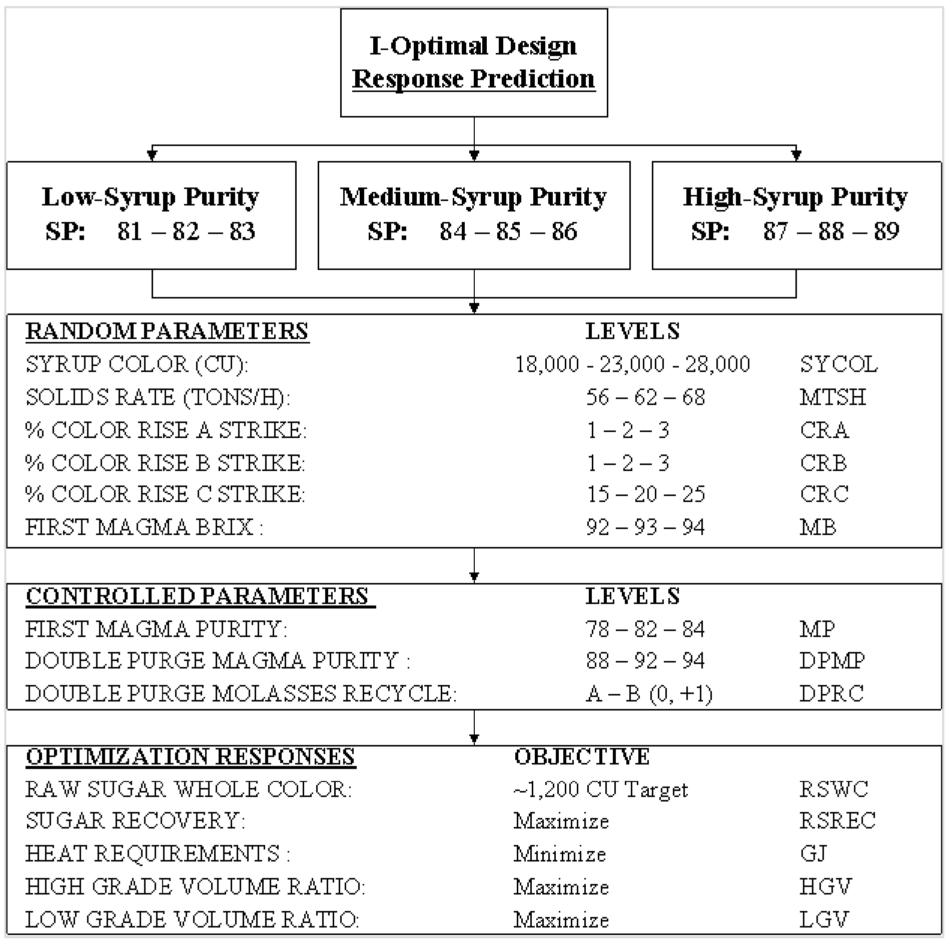


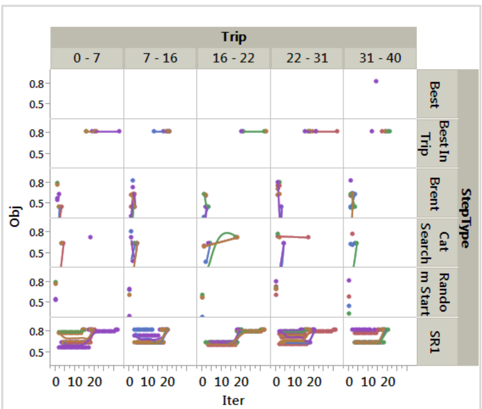
Figure 4.5 Block diagram for the computer aided I-Optimal experimental design adding center points to create response surface models (surrogate models) using JMP® (257 simulations)

Simulation results were evaluated using JMP multivariate analysis and model screening (SAS 2014). The multivariate analysis includes different tools to generating reports with basic statistical analysis of the distribution, scatter plots, histograms and outliers. The model screening tests the significance of main factors, interactions and aliasing (parameters confounding), and highlights the main effects for model creation. After analysis of the data, each surrogate response model is created and the adequacy of the model is evaluated by statistical means and graphs of residuals. Finally the multiple response optimizations for each scenario and case are performed with the JMP graph custom profiler (SAS 2014). Equation 4.16 shows the expression used by the JMP algorithm to maximize the objective function (overall desirability) for two responses RSWC and RSREC with equal importance (response weight). The values inside the first bracket for the individual desirability defines the user's choice for the response

range while the second bracket defines also assigned desirability between 0 and 1 for the respective response values inside the range.

max Obj= Exp ((1 * Log (Desirability ([800,1200,1600],[0.0183,1,0.0183],Pred_Formula_RSWC))+
1* Log (Desirability ([88,90,92],[0.066,0.5,0.9819],Pred_Formula_RSREC))) / 2) (Equation 4.16)

Figure 4.6 illustrates the results, optimization steps (methods) and sequence to maximize the overall desirability, for two responses RSWC and RSREC. The JMP algorithm does multiple searches (trips) generating random values for all the parameters at the start of the trip (first iteration) followed by a sequence of optimization strategies (steps) in cycles to explore the design space and to exploit the neighborhood for the best results of the search. On each trip the algorithm compares the best value found (best in trip) with the best of the all old searches (new – old). The search stops when the difference between new and old best values for a determined number of trips is lower than the convergence criteria (convergence tolerance = 0.000001).



Optimization sequence and results

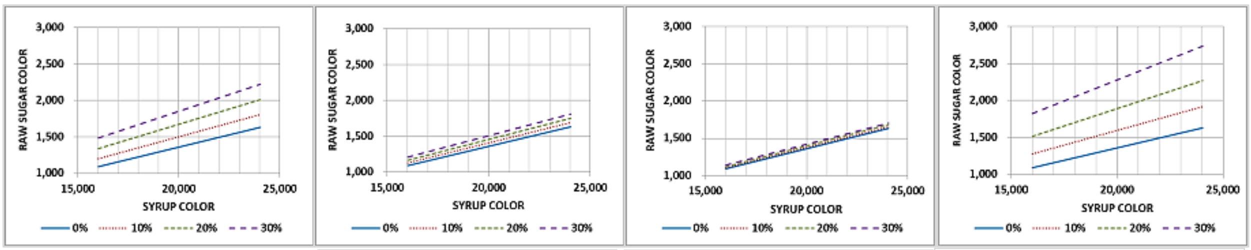
	Step Type	N	Max
1	Random start	40	0.81969
2	Brent	63	0.82521
3	Cat search	42	0.81227
4	SR1	680	0.82572
5	Best in trip	40	0.82572
6	Best	1	0.82572

Figure 4.6 Optimization plot: objective versus iteration number by trips and by step type (JMP optimization algorithm). Optimization sequence and statistical summary for each step to find the maximum overall desirability. Optimization settings: number of trips=40, maximum number of iterations=500, Convergence tolerance= 0.000001 and maximum cycles=50 (JMP(R) 2014)

4.3. Results and Discussion

Crystallization is a very effective liquid-solid physical separation used to obtain purified sucrose from impure sucrose solution. However, like other impurities, a small amount of colorants are not separated from the crystals by this process. Colorants can be retained within and on the sucrose crystals by different mechanisms. Some may bond to a crystal face, be trapped between crystal layers and/or contained in an external layer of mother liquor (Bento 2003). The sources of colorants are colorants input with the syrup and colorants formed during each crystallization stage. In a sugar mill these are carried back with the crystal seed to the first strike. Although the chemical nature and color intensity of the various components found in raw sugar are different, the balance of the clustered colorants behaves like other impurities (e.g. conductivity ash). Crystallization and separation schemes can be modified to improve the color of raw sugar by reducing the recycling of impurities, as in the crystal seed, like with very high pol (VHP) sugar production, double magma and high graining crystallization schemes (van Hengel 1983; Wright 1996; Madho and Davis 2008). Raw sugar color can also be reduced chemically by the addition of more water and hydrogen peroxide on centrifuges, or by double centrifugation (called also double purge and affination). Any improvement of raw sugar quality, as required by polarization and color specifications, increases the material throughput and the required energy, affecting boiling house performance (Wright 1989; Madho and Davis 2008). A raw sugar factory, to be efficient, needs to produce the specified quality sugar while maintaining a good crystal yield. There is information about the causes (factors) of color on raw sugar and the factors that affect operability and crystal yield (sugar recovery) but, there are no quantification of the effects and their interactions. This research evaluates the importance of the various factors and their interactions and combines different responses for quality, operability and sugar yield to optimize the operation of a boiling house, specifically for the implementation of double centrifugation or double purge of C-magma. The methodology used can be applied to other boiling schemes for optimization of desired responses.

Figure 4.7 shows the whole color response from the preliminary simulations of the double purge system, accounting for the amount of color in the syrup and the individual and combined levels of % of color rise on A, B and C strikes. It can be seen that after syrup, the color rise on A strike has the greatest impact on raw sugar color and that color rises by strike do not interact, but their effects are additive to the final color of raw sugar. From color analysis and models, it was determined that color rise was around 2% for A and B and 20% for C strikes. The selection of a real range of variation is a key factor for the creation of surrogate models representing the behavior of the whole system; a wide or unreal range will exaggerate the importance of a given factor.



a) Raw sugar color by % color rise in A strikes	b) Raw sugar color by % color rise in B strikes	c) Raw sugar color by % color rise in C strikes	d) Raw sugar color by % color rise in ABC strikes

Figure 4.7 Raw sugar whole color versus syrup color for different percentages of color rise – 0%, 10%, 20% and 30%, on: a) A strikes, b) B strikes c) C strikes and d) combined same level of color rise on each strike. Simulations using a double purge model created on Sugars™

4.3.1. Two Level Full Factorial Experimental Design for Factors Screening

The primary objective of the experimental design was to screen the raw sugar color response to determine factors and interactions. The syrup purity input ranged between 84 and 88 and the ratio of syrup color on non-sucrose (SCNSR) ranged between 16 and 20 * 10^4 cm^2/kg non-sucrose (19,200 CU to 32,000 CU → cm^2/kg soluble solids). Figure 4.8 shows a scatter plot matrix for model parameters and simulated raw sugar color. This data was used to estimate additional responses, such as sugar recovery (RSREC), heating requirements (GJ), pans capacity installed/required ratio for high grade (HGV) and low grade (LGV) pans. The scatter plots show differences between the responses when recycling double purge molasses. There was no significant difference for raw sugar color, when comparing recycle of double purge molasses (DPRC) to A molasses and to B molasses but, there were clear interactions of the recycle point with the purity of the double purge magma (DPMP), the purity of the first magma (MP) and the purity of the syrup (SP) for the other responses. These plots demonstrate that the controlled product parameters (DPMP, MP and DPRC) have also important effects on other boiling house responses.

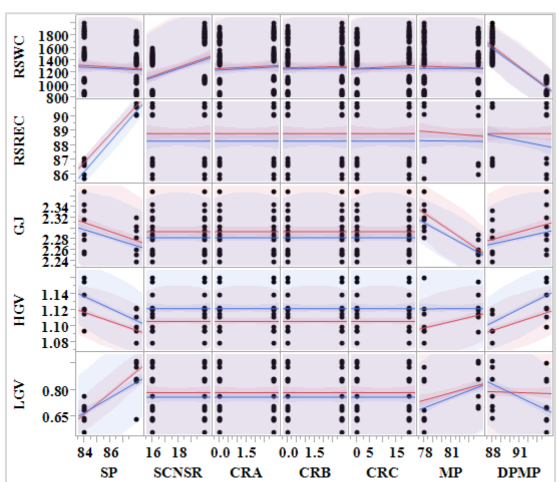


Figure 4.8 Scatter plot matrix for the two level full factorial (2^8=256 simulations) parameters (horizontal axis) and responses (vertical axis) to screen effects mainly on raw sugar color for the integration of double purge of C-magma (red fit lines for recycle to A molasses and blue fit lines for recycle to B molasses)

A two level experimental design shows the importance of controlled and random parameters and the sensitivity of several responses to changes in these parameters. An experimental design created specifically for model prediction was required to build the surrogate models that were used for optimization. The half normal plot (Figure 4.9) and the Pareto plot presented on Table 4.4 shows that the controlling factors affecting the color of raw sugar are the syrup color/non-sucrose ratio and the purity of the double purge magma (DPMP). Color rise on each strike was not as significant, at least for the specified variation range. The syrup color/non-sucrose ratio (SCNSR) was used because it was shown more important at high syrup purity range. For instance, syrup color/non-sucrose ratio of 20 * 10^4 cm^2/kg non-sucrose at 88 syrup purity is equivalent to a color of 32,000 CU and at 84 syrup purity is equivalent to a color of 24,000 CU.

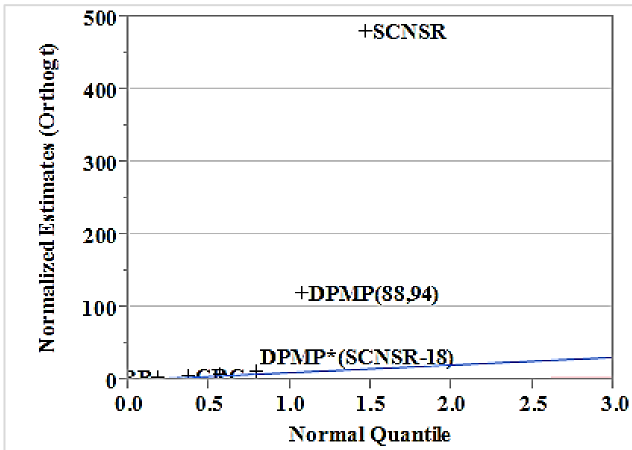


Figure 4.9 Half-normal probability plot indicating most important parameters for raw sugar whole color. Factors outside the blue line have a significant effect on raw sugar color. The slope of the line 17.96 is the Lenth's PSE-pseudo error (t-test scale)

Table 4.4 Pareto plot for raw sugar whole color model estimates from analysis of a two-level full factorial experimental design. The factors are syrup color/non-sucrose ratio (SCNSR), double purge magma purity (DPMP) and the color rise on A, B and C strikes (CRA, CRB and CRC)

Term	Orthog. Estimate	Pareto Plot
SCNSR	1295.704	
DPMP(88,94)	-327.953	
DPMP*(SCNSR-18)	-36.445	
CRA	23.453	
CRC	14.637	
CRB	9.657	

Table 4.5 indicates the level of importance (+) of each parameter on the respective responses, the more (+) signs the higher is the importance of the parameter. For example, the assessment of the importance (or impact) of SP main effect can be determined by measuring the variation of RSWC at each value in the range of variation of SP (variance=Var(E(RSWC|SP)), that is equivalent to the variation of the estimated (E) mean value of RSWC for a fixed value of SP. The sensitivity of RSWC to the parameter SP is determined in reference to the whole variance of RSWC (Var(E(RSWC)). Thus the sensitivity of RSWC to the main effect SP is the ratio Var(E(RSWC |SP))/Var(E(RSWC)). The evaluation of the total effect integrates all main effects, interactions and high-order effects for the parameter. For example, the total effect importance index of DPMP is determined by [Var(E(RSWC |DPMP))+Var(E(RSWC|DPMP,DPRC))]/Var(E(RSWC)) (SAS 2014).

Table 4.5 Variable importance for each response according to simulations using a two level full factorial experimental design (JMP(R) 2014)

Parameter	Minimum	Maximum	RSWC	RSREC	GJ	HGV	LGV
SP	84	88		++++		++	+++
SCNSR	16	20	+				
CRA	0	3					
CRB	0	3					
CRC	0	20					
MP	78	84			++		+
DPMP	88	94	+++++		++	+++	+
DPRC	A	B		++	++	++	+

Factors screening for the specified design space (range of variation of each factors) indicated that product parameters of the double purge of C-magma have significant effect not only for raw sugar whole color but also for other operational and performance indices.

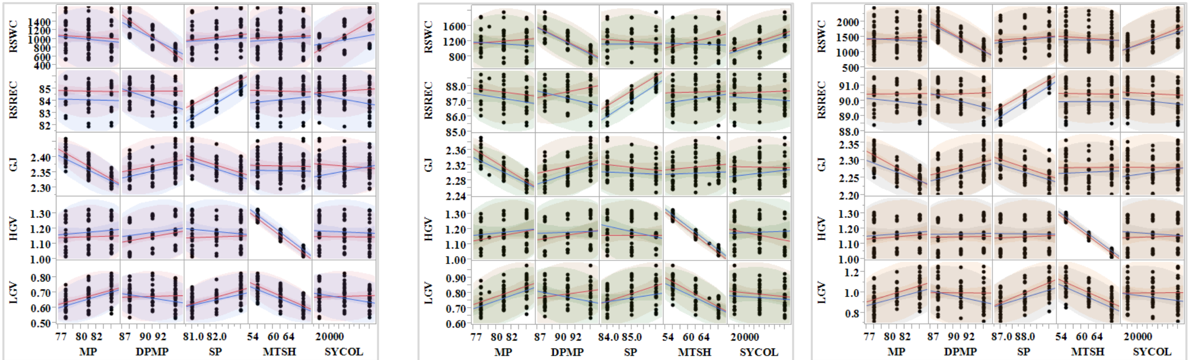
4.3.2. I-Optimal Experimental Designs for Model Prediction (Metamodeling)

An analysis of simulation results, applying a two-level full factorial experimental design, shows the importance and the direction of the effects on each evaluated parameter for raw sugar color and boiling house responses. The objective was to create approximate models representing the behavior of the desired responses. The experimental design was divided in blocks to show the responses for different syrup purity scenarios and the variability of the parameters that define boiling house responses. Table 4.6 shows a statistical summary for input parameters and simulation responses. For the same mean syrup color input (on soluble solids), the color non-sucrose ratio was higher for high purity syrup than for low purity syrup, producing a higher raw sugar color for the high syrup purity scenario.

Table 4.6 Statistics summary for input parameters and simulation responses at low, medium and high syrup purity scenarios including number of computer simulations (N), mean and standard deviation (Std. Dev.)

	Parameter/Response	Low Purity Syrup			Medium Purity Syrup			High Purity Syrup		
	Abbreviation	**N**	**Mean**	**Std. Dev.**	**N**	**Mean**	**Std. Dev.**	**N**	**Mean**	**Std. Dev.**
Parameters	**MP**	103	81.0	2.2	66	81.1	2.4	88	80.9	2.7
	DPMP	103	91.1	2.5	66	91.0	2.5	88	91.0	2.6
	MB	103	93.0	0.8	66	93.0	0.8	88	93.0	0.9
	CRA	103	2.0	0.8	66	2.0	0.8	88	2.0	0.9
	CRB	103	2.0	0.8	66	2.0	0.8	88	2.0	0.9
	CRC	103	20.0	4.1	66	20.0	4.1	88	19.9	4.4
	MTSH	103	62.0	4.4	66	61.8	4.8	88	61.9	5.3
	SP	103	82.1	0.9	66	85.0	0.8	88	88.0	0.8
	SYCOL	103	22,952	4,112	66	23,000	4,114	88	23,057	4,321
Responses	**RSWC**	103	1,028	341	66	1,170	331	88	1,438	446
	RSREC	103	84.48	1.09	66	87.39	0.90	88	90.24	0.88
	GJ	103	2.36	0.04	66	2.31	0.03	88	2.27	0.04
	HGV	103	1.16	0.09	66	1.17	0.09	88	1.16	0.10
	LGV	103	0.67	0.07	66	0.78	0.08	88	0.98	0.13

Figure 4.10 shows the scatter plot matrices that relate the input parameters to raw sugar color response and boiling house responses, estimated from simulations for three different syrup purities. The effects of color rise for each strike (CRA, CRB and CRC) and the Brix of the first magma (MB) for the assigned variation range were not significant in terms of the variation of the responses.



a) Low syrup purity (82) | b) Medium syrup purity (85) | c) High syrup purity (88)

Figure 4.10 I-Optimal experimental design scatters plot matrices for different syrup purity scenarios a) Low, b) Medium and c) High. Red fit lines for recycle to A molasses and blue fit lines for recycle to B molasses (DPRC)

The models relating each response to the product parameters can be controlled in a double purge system by the input purity of the first magma (MP), the output purity of the double purge magma (DPMP) and the point of recycle of wash or double purge molasses (DPRC). Parameters with random variation are included in the model when the effect of the parameter is significant. The models were built using first model selection for screening designs (JMP(R) 2014). The Lenth's t-ratio and the simultaneous p-value<0.0001 are the criteria used to pick significant effects. Once the effects were selected, the model was created by choosing the standard least squares model fit with emphasis on leverage (isolation of the effect of a single parameter). Coefficients of determination (R^2 and adjusted R^2), prediction plots, residual plots and predicted error sum of squares (press) are among the statistical tools used to check linearity and validity of the models (SAS 2014). Complete statistical reports for each surrogate model are presented on Appendices B to F.

4.3.3. Raw Sugar Whole Color (RSWC)

The general surrogate model for RSWC is given by the following expression (β is the regression coefficient – estimator and the categorical parameter DPRC is +1 for A and -1 for B):

(Equation 4.17)

$$RSWC=\beta_0 + \beta_1 \cdot DPMP + \beta_2 \cdot SYCOL + \beta_3 \cdot SP \pm \beta_4 \cdot DPRC + \beta_5 \cdot DPMP \cdot SYCOL$$

The assessment of variable importance for RSWC shows that the most important parameter to control raw sugar whole color was DPMP in all the scenarios (Table 4.7). At the same SYCOL input, the effect of a variation on DPMP had more effect on RSWC at the low syrup purity than at the high syrup purity scenario. For the specified range of SYCOL variation, the total effect of this parameter is greater at high syrup purity because of a higher amount of color compounds in relation to the total amount of non-sucrose compounds (higher color/non-sucrose ratio).

Table 4.7 Summary of parameters importance according to total effect (dependent resampled inputs) and model fit evaluation: adjusted R-square, model error (root square mean error) and prediction error of surrogate models for raw sugar whole color (RSWC) at each syrup purity scenario

Importance - Total Effect	**Syrup Purity Scenario**		
Parameter	**Low**	**Medium**	**High**
DPMP	0.324	0.296	0.291
DPRC	0.175	0.321	0.252
SYCOL	0.136	0.182	0.188
SP	0.023	0.015	0.025
Adjusted R-square model	0.998	0.997	0.993
RSM – model error	15.81	18.71	36.69
Press – prediction error	16.30	20.08	38.41
Mean - RSWC	1,028	1,170	1,438

The whole color of raw sugar and the prediction error is greater for high syrup purity than for low syrup purity. The higher value for the RSWC at high syrup purity is due to a higher color/non-sucrose ratio input considering the same color input (bases on soluble solids) at high and a low syrup purity. To achieve the target color at high syrup purity, a higher purity of the double purge magma (DPMP) compared to low syrup purity, at the same syrup color input (SYCOL), is required. The recycling of double purge molasses (DPRC) has less effect on the raw sugar whole color. The optimization for a single raw sugar whole color response (RSWC) gives values for DPMP ranging from 88 for low syrup purity to maximum 94 for high syrup purity, depending on the color input with the syrup (SYCOL).The parameter DPRC is A for low and medium and also for the high syrup purity scenario when SYCOL=20,000 CU, but DPRC=B for high syrup purity when SYCOL=26,000 CU (Figure 4.11 (a, b), Figure 4.12 (a, b) and Figure 4.13 (a, b)). The desirability function depends on the response goal, which for raw sugar whole color is to match a target of 1,200 CU. The size and position of the triangles indicates the sensitivity and direction of the effect on the response. The intensity of the highlighting is determined based on the assessment of the importance of the parameter effect on the response.

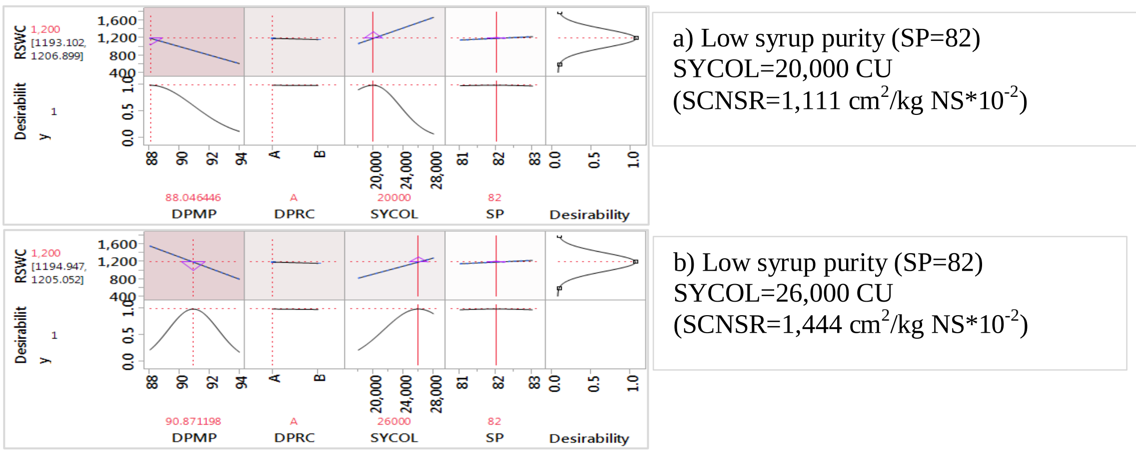


Figure 4.11 Raw sugar whole color (RSWC) response prediction and desirability plots at low syrup purity (SP=82) for main model parameters. Fixed syrup color (SYCOL) input a) 20,000 CU and b) 26,000 CU. SCNSR (syrup color/non-sucrose ratio) = SYCOL/(100-SP)

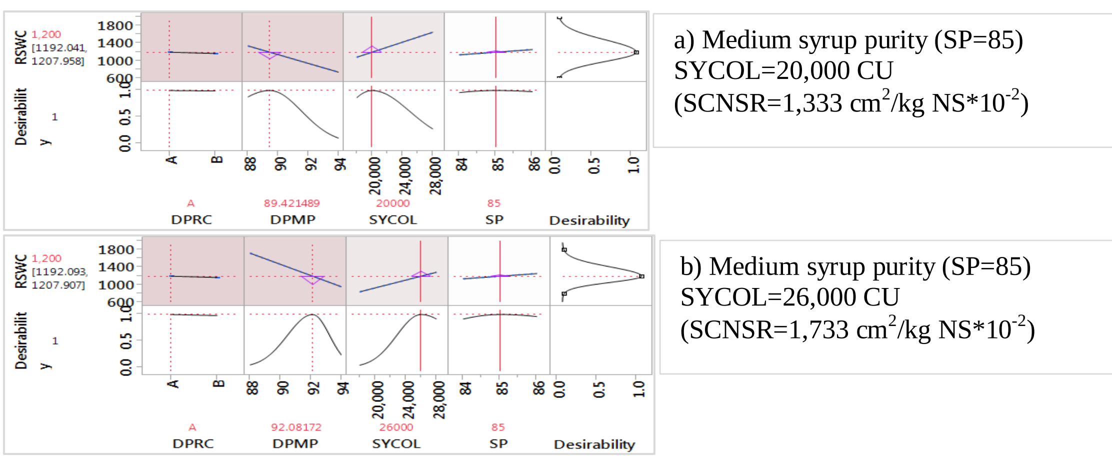
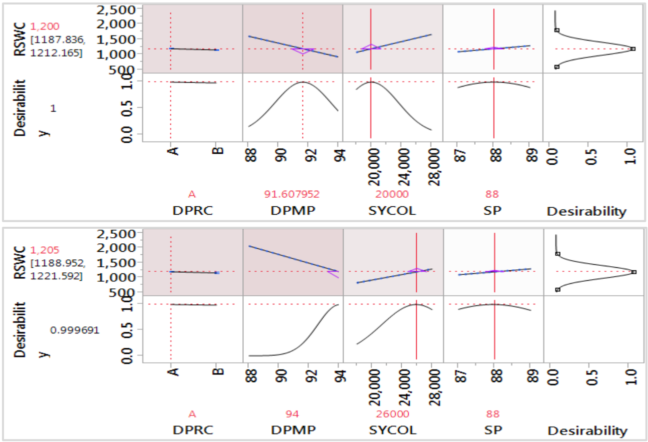


Figure 4.12 Raw sugar whole color (RSWC) response prediction and desirability plots at medium syrup purity (SP=85) for main model parameters. Fixed syrup color (SYCOL) input a) 20,000 CU and b) 26,000 CU. SCNSR (syrup color/non-sucrose ratio) = SYCOL/(100-SP)



a) High syrup purity (SP=88)
SYCOL=20,000 CU
(SCNSR=1,667 cm^2/kg NS*10^{-2})

b) High syrup purity (SP=88)
SYCOL=26,000 CU
(SCNSR=2,166 cm^2/kg NS*10^{-2})

Figure 4.13 Raw sugar whole color (RSWC) response prediction and desirability plots at high syrup purity (SP=88) for main model parameters. Fixed syrup color (SYCOL) input a) 20,000 CU and b) 26,000 CU. SCNSR (syrup color/non-sucrose ratio) = SYCOL/(100-SP)

Figure 4.14 illustrates that for the high syrup purity scenario, to achieve a target RSWC of 1,200 CU (contour line), the product parameter DPMP (double purge magma purity) needs to be controlled around 90 for low SYCOL values (~18,000 CU), but when the values of SYCOL are high (~ 28,000 CU), DPMP has to be controlled around 94.

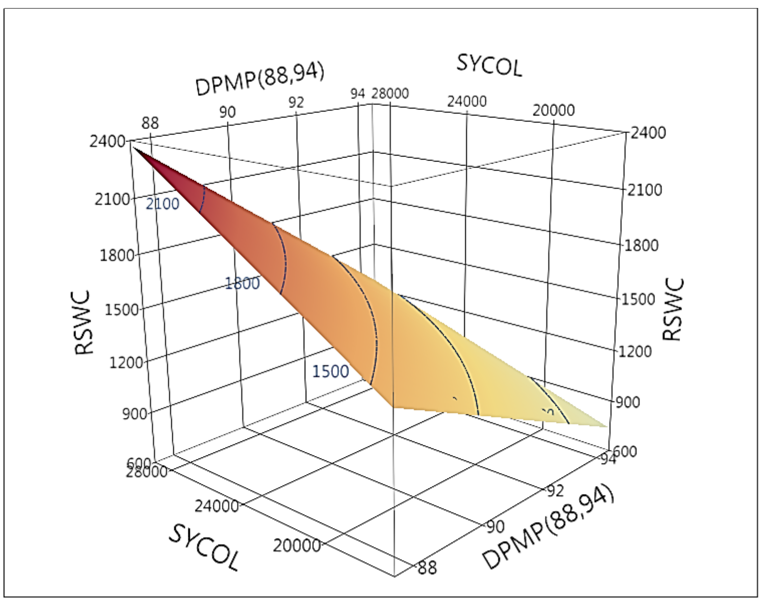


Figure 4.14 Response surface plot for RSWC by DPMP and SYCOL at high syrup purity scenario (fixed SP=88 and DPRC=A)

It is known that the color of the syrup (SYCOL) is related to the purity, or better expressed relative to the impurities or non-sucrose component concentration; however, color related to the soluble solids has more meaning within the sugar industry. The optimal settings determined for the single response optimization of raw sugar whole color do not account for the effects on other boiling house operational and performance responses.

4.3.4. Sugar Recovery (RSREC)

The general surrogate model for RSREC is given by the following expression (β is the regression coefficient – estimator and the categorical parameter DPRC is +1 for A and -1 for B):

(Equation 4.18)

$$RSREC=\beta_0 + \beta_1 \cdot SP \pm \beta_2 \cdot DPRC + \beta_3 \cdot DPMP + \beta_4 \cdot MP + \beta_5 \cdot MB \pm \beta_6 \cdot DPRC \cdot DPMP \pm \beta_7 \cdot DPRC \cdot MB$$

The assessment of the importance of each parameter for RSREC shows that SP and DPRC have the greatest effect (Table 4.8). The greater importance of SP in the high purity scenario confirms that, at the boiling house, more sugar can be recovered as the syrup purity rises. DPMP, MB and MP have a small effect on sugar recovery (RSREC).

Table 4.8 Summary of parameters importance according to total effect (dependent resampled inputs) and model fit evaluation: adjusted R-square, model error (root square mean error) and prediction error of surrogate models for raw sugar recovery (RSREC) at each syrup purity scenario

Importance-Total Effect	**Syrup Purity Scenario**		
Parameter	**Low**	**Medium**	**High**
SP	0.341	0.343	0.427
DPRC	0.226	0.198	0.228
DPMP	0.056	0.074	0.038
MB	0.032	0.016	0.009
MP	0.022	0.020	0.053
Adjusted R-square model	0.998	0.997	0.993
RSM - model error	0.089	0.079	0.068
Press - prediction error	0.094	0.086	0.072
Mean - RSREC	84.48	87.39	90.24

There are some significant interactions between DPRC, DPMP and DPRC with MB that increase the effect of DPRC on raw sugar recovery (RSREC). Figure 4.15 (a, b, c) show the amounts and direction of the variation of the RSREC response for each parameter, under different syrup purity scenarios. Fixing the values of SP and MB=93 for each syrup purity scenario, the optimal settings to maximize RSREC were DPRC=A, MP ~ 78 and DPMP~88 for low and medium SP and DPMP=94 for high syrup purity. The importance of MP is higher than DPMP at high syrup purity. At the high syrup purity scenario (Table 4.8), the total effect of MP is ~1.8 % higher than the total effect of DPMP respect to the combined total effect for all 5 parameters (0.836).

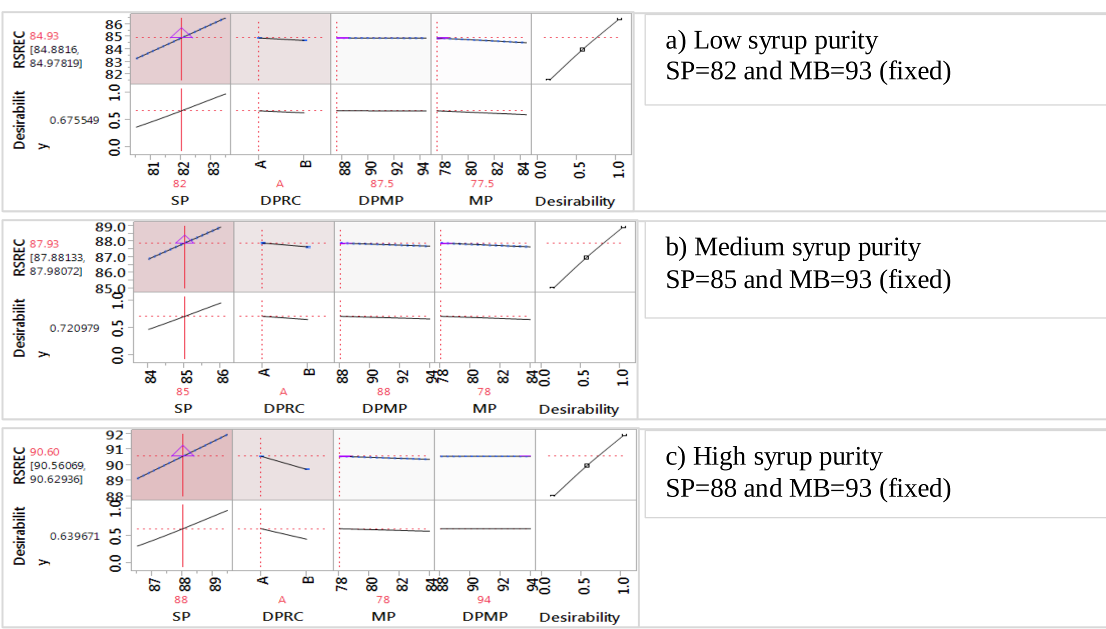
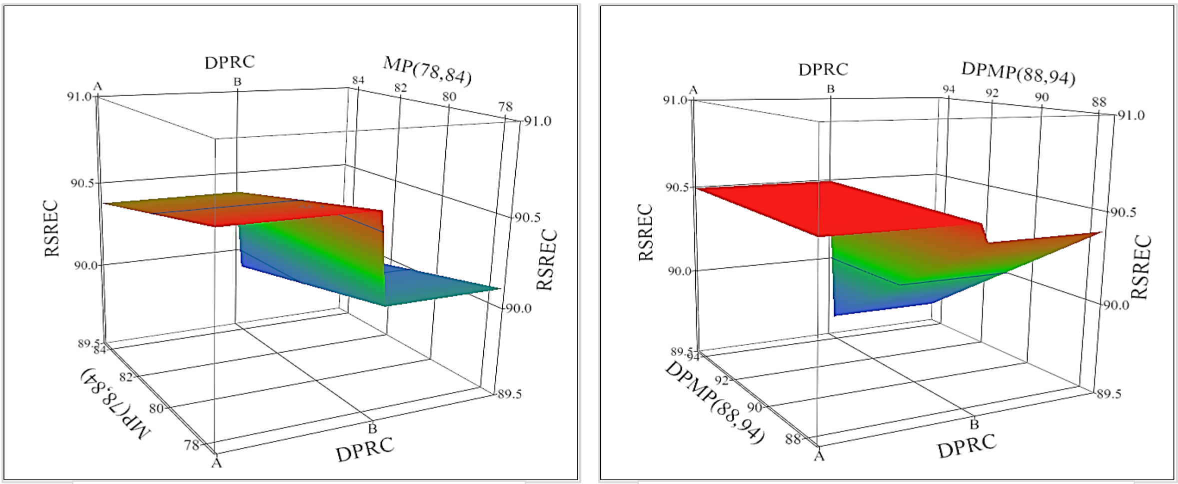


Figure 4.15 Raw sugar recovery (RSREC) response prediction and desirability profiles at different syrup purity scenarios: a) Low, b) Medium and c) High. Controlled double purge parameters DPRC, MP and DPMP and, fixed parameters SP and MB

Figure 4.16 (a, b) illustrates the effect of the point of recycling DPRC on the RSREC response surface at high syrup purity. The maximum sugar recovery (RSREC) is obtained when DPRC=A and that there is no significant effect of the DPMP parameter when DPRC=A.



a) RSREC versus DPRC and MP | b) RSREC versus DPRC and DPMP

Figure 4.16 Response surface plots for RSREC at high syrup purity by a) DPRC and MP and b) DPRC and DPMP (fixed SP=88, MB=93)

The maximum desirability just for sugar recovery on these conditions is 0.6 (SP=88), the desirability will be greater for a higher SP according to the sensitivity and importance of this parameter on the surrogate model.

4.3.5. Heating Requirements (GJ)

The general surrogate model for GJ is given by the following expression (β is the regression coefficient – estimator and the categorical parameter DPRC is +1 for A and -1 for B):

(Equation 4.19)

$$GJ = \beta_0 + \beta_1 \cdot MP + \beta_2 \cdot SP + \beta_3 \cdot DPMP \pm \beta_4 \cdot DPRC + \beta_5 \cdot MB + \beta_6 \cdot MP \cdot MP + \beta_7 \cdot MP \cdot SP + \beta_8 \cdot MP \cdot DPMP + \beta_9 \cdot DPMP \cdot MB$$

Assessing the importance of parameter effects it is determined that MP, DPRC and DPMP have significant effects on the variability of GJ. Minimization of heating requirements is an optimization goal for the integration of the double centrifugation or double purge. At low syrup purity, MP is the product parameter with the highest total effect. DPRC has the highest importance at high syrup purity according to the total effect of this parameter – 0.32, which is almost 3 times higher than the total effect at the low syrup purity scenario – 0.13 (Table 4.9).

Table 4.9 Summary of parameters importance according to total effect (dependent resampled inputs) and model fit evaluation: adjusted R-square, model error (root square mean error) and prediction error of surrogate models for heating requirements (GJ)

Importance-Total Effect	**Syrup Purity Scenario**		
Parameter	**Low**	**Medium**	**High**
MP	0.280	0.264	0.194
SP	0.132	0.078	0.085
DPRC	0.131	0.123	0.320
DPMP	0.111	0.123	0.097
MB	0.060	0.058	0.014
Adjusted R-square model	0.993	0.975	0.951
RSM - model error	0.003	0.005	0.008
Press - prediction error	0.004	0.006	0.009
Mean of Response - GJ	2.364	2.307	2.274

Figure 4.17 (a, b, c) show the amount and direction of the variation of the GJ response for each parameter at different syrup purities. Fixing the values of SP and MB=93 at a low syrup purity, the optimal settings to maximize GJ are DPRC=B, MP ~ 84 and DPMP~88. The setting of MP=84 minimizes the energy requirements because it reduces the recycling of impurities that have high effects on processing volumes (Hessey and Manning 1949).

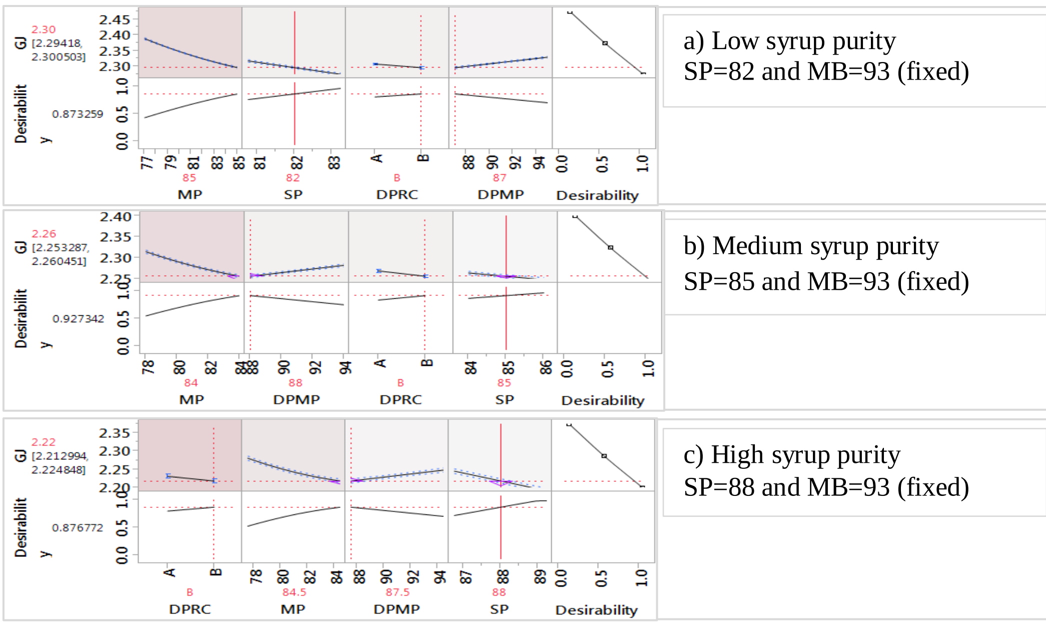
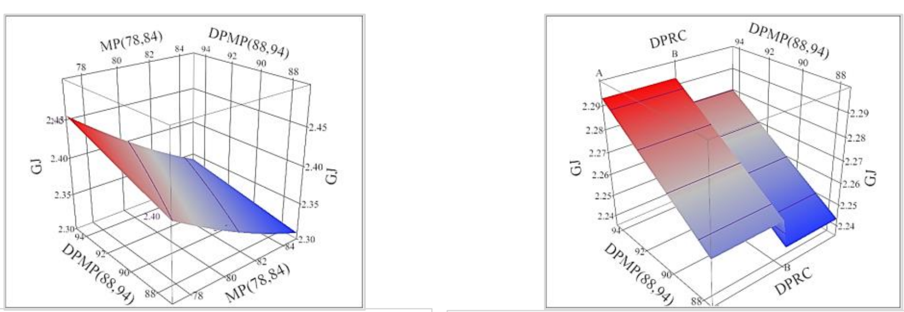


Figure 4.17 Heating requirements (GJ = gigajoules/ton syrup solid solids) response prediction and desirability profiles at different syrup purity scenarios: a) Low, b) Medium and c) High. Controlled double purge parameters DPRC, MP and DPMP (fixed SP and MB)

Rein (2007) reports steam use of approximately 87.7 – 93.0 (kg steam/kg syrup soluble solids) for a three-boiling scheme and approximately 99.0 to 99.4 for VHP and double magma boiling schemes using a constant pan factor of 1.25 (required steam/water evaporated). The management of the steam usage by the process is necessary for efficient operation of a sugar factory and possibly for cogeneration from surplus bagasse as well (Broadfoot 1999; Broadfoot 2001; Rein 2007). The estimated usage of 2.20 to 2.45 gigajoule/ton of syrup soluble solids is equivalent to the use of vapor from evaporators in the order of 103 to 111 kg/ 100 kg of syrup soluble solids (vapor-I pressure= 177.2 kPa, latent heat=2,212.27 kJ/kg) with an estimated pan factor of 1.30. The individual maximum GJ response desirability ranges from ~ 0.85 to 0.93. Figure 4.18 (a) shows on a 3D plot the shape of GJ response at low syrup purity (SP=82), indicating the higher effect of MP than DPMP inside the design space. Figure 4.18 (b) shows that heating requirements are higher when DPRC=A and the effect of DPRC is more important for the high syrup purity scenario.



a) GJ versus DPMP and MP at low syrup purity (82)

b) GJ versus DPRC and DPMP at high syrup purity (88)

Figure 4.18 Response surface plots for heating requirements (GJ=gigajoules/ton syrup soluble solids) at a) Low syrup purity GJ by DPMP and MP and, b) High syrup purity GJ by DPRC and DPMP (fixed MB=93)

At the boiling house, crystallization by water evaporation is the core process to transfer sucrose from solution to solid sucrose crystals. Evaporation increases the concentration of sucrose, driving it to supersaturation. The energy required for this operation is obtained mainly from the latent heat in the water vapor produced by the first effect evaporators (juice concentration process stage); this low pressure steam is denominated vapor-I. If the heat demand is not satisfied, the evaporation rate at the boiling house is reduced which in turn may lead to a slowdown in the milling rate or a factory stop. The energy balance is crucial for a steady state operation of the whole factory. The relevance of the heating requirement for soluble solids input (GJ) is higher for low syrup purity, demanding more vapor-I from evaporators or the need to use make-up steam.

4.3.6. High Grade Volume Ratio (HGV)

The general surrogate model for HGV is given by the following expression (β is the regression coefficient – estimator and the categorical parameter DPRC is +1 for A and -1 for B):

(Equation 4.20)

$$\mathrm{HGV} = \beta_0 + \beta_1 \cdot \mathrm{MTSH} + \beta_2 \cdot \mathrm{DPMP} \pm \beta_3 \cdot \mathrm{DPRC} + \beta_4 \cdot \mathrm{MP} + \beta_5 \cdot \mathrm{MTSH} \cdot \mathrm{MTSH}$$

The variation and effect of MTSH is high (56 – 68 tons syrup solids/hour) because it depends on the milling rate and quality of the cane. Both milling rate and cane quality increase from the beginning to the end of the harvesting season. The average milling rate ranges from 9,600 to 11,600 metric tons of cane per day (10,600 – 12,800 short tons per day). Cane quality also affects the quality of the juice, which normally changes from a low solids concentration and purity at the start to a high solids concentration and purity at the end. However there are fluctuations in the quality due to weather, harvesting and cane cultivation abnormalities. A single optimization goal is to maximize the high grade volume ratio (HGV). MTSH and DPRC are the most important parameters for the HGV response in all syrup purity scenarios (Table 4.10).

Table 4.10 Summary of parameters importance according to total effect (dependent resampled inputs) and model fit evaluation: adjusted R-square, model error (root square mean error) and prediction error of surrogate models for high grade volume ratio (HGV=installed / required capacity) at each syrup purity scenario

Importance-Total Effect	**Syrup Purity Scenario**		
Parameter	**Low**	**Medium**	**High**
MTSH	0.443	0.460	0.438
DPRC	0.235	0.225	0.239
DPMP	0.025	0.018	0.017
MP	0.013	0.039	0.006
Adjusted R-square model	0.997	0.994	0.995
RSM - model error	0.005	0.007	0.007
Press - prediction error	0.005	0.007	0.008
Mean of Response - HGV	1.163	1.172	1.157

The double purge parameters DPMP and MP have very little effect on high grade processing volumes, Figure 4.19 (a, b). Double purge parameters that maximize the HGV response are DPRC=B, DPMP=94 and MP=84. These settings render a minimum recycle of impurities to the high grade massecuites. Required high grade pan capacity at MTSH=68 is as high as the installed capacity (installed/required=1.07) affecting pan operation and sugar yield. Massecuite processing rates are related to the sugar yield per strike (% crystal content), since it affects directly the crystallization time (time required for the sucrose transference from the solution to the crystal). Cycle time for batch vacuum pans includes the required time for crystallization (depending on massecuite purity) and the "death time" required for loading, unloading and preparing the pan (Wright 1996).

The maximum desirability for an HGV response is achieved by setting the double purge parameters to DPRC=B, MP=84 and DPMP=94 where the expected response is minimizing the recycling of impurities in the first strike (Figure 4.20 a, b, c).

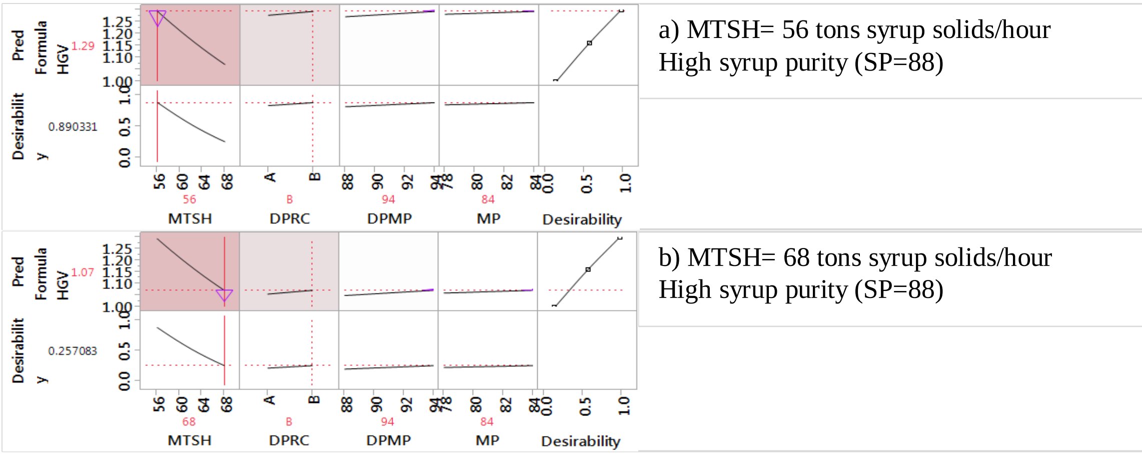


Figure 4.19 High grade volume ratio (HGV=installed / required capacity) response prediction and desirability profile at high syrup purity for different hourly rate input of syrup soluble solids: a) MTSH=56 and b) MTSH=68. Controlled double purge parameters DPRC, MP and DPMP. (fixed SP=88)

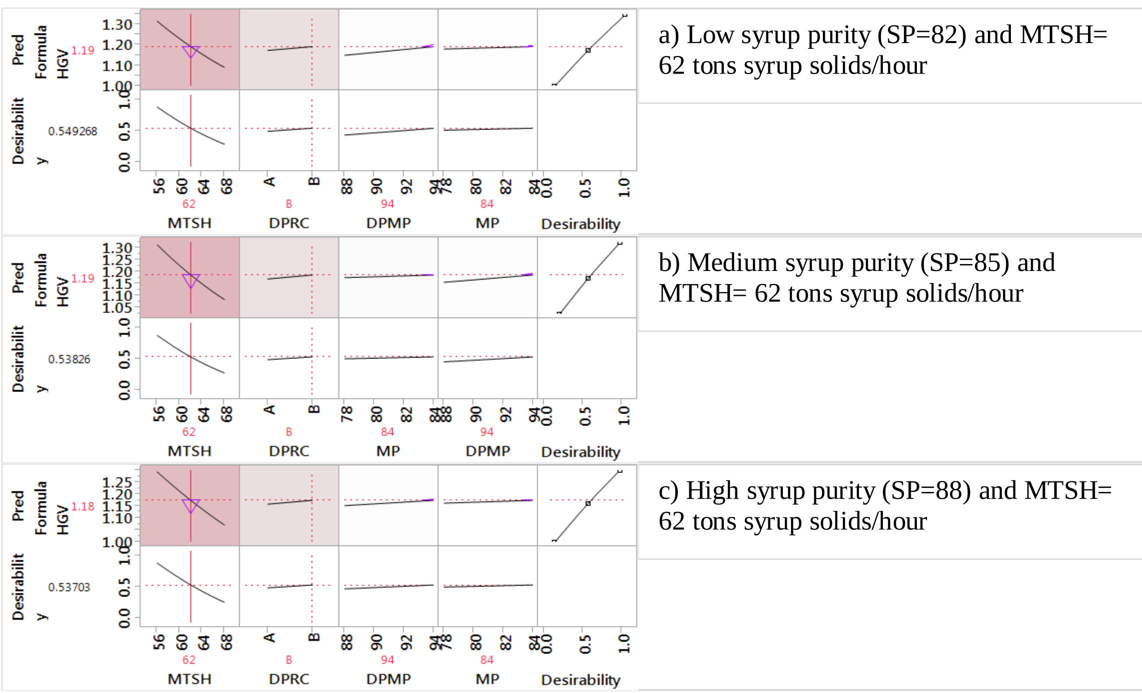


Figure 4.20 High grade volume ratio (HGV = installed/required volume capacity) response prediction and desirability profiles at different syrup purity scenarios: a) Low, b)Medium and c) High. Controlled double purge parameters are DPRC, MP and DPMP (fixed MTSH and SP)

4.3.7. Low Grade Volume Ratio (LGV)

The general surrogate model for LGV is given by the following expression (β is the regression coefficient – estimator and the categorical parameter DPRC is +1 for A and -1 for B):

(Equation 4.21)

$$\mathrm{LGV} = \beta_0 + \beta_1 \cdot \mathrm{MTSH} + \beta_2 \cdot \mathrm{SP} + \beta_3 \cdot \mathrm{MP} \pm \beta_4 \cdot \mathrm{DPRC} + \beta_5 \cdot \mathrm{DPMP} \pm \beta_6 \cdot \mathrm{DPRC} \cdot \mathrm{DPMP}$$

The low grade streams are composed of the massecuites output from the grain and C-strikes. The purity and volume rate of the C-massecuites are related to sugar losses to final molasses. C-massecuite purity depends on the exhaustion achieved from the high grade massecuites, because it determines the purity of the B-molasses used to

make C-massecuites. Equipment availability is a factor that limits the degree of exhaustion (sucrose on crystals % total sucrose on the massecuite) of high grade and low grade massecuites, because it determines the time given for each crystallization stage (strike). Amounts of impurities in the syrup and recycle are the most important factors affecting C-massecuites volume rates (Broadfoot and Pennisi 2001; Broadfoot and Pennisi 2002; Rein 2007). A single optimization goal is to maximize the low grade volume ratio (LGV). Analyzing LGV (installed/required capacity) response for each purity scenario determined that the random parameters, MTSH and SP, have the greatest effects, but the recycling of double purge molasses (DPRC) also has a large effect in the low syrup purity scenario. The parameter MP has also a significant effect for every syrup purity scenario (Table 4.11).

Table 4.11 Summary of parameters importance according to total effect (dependent resampled inputs) and model fit evaluation: adjusted R-square, model error (root square mean error) and prediction error of surrogate models for low grade volume ratio (LGV) at each syrup purity scenario

Importance-Total Effect	**Syrup Purity Scenario**		
Parameter	**Low**	**Medium**	**High**
DPRC	0.228	0.188	0.243
MTSH	0.198	0.252	0.188
SP	0.132	0.076	0.140
MP	0.089	0.096	0.083
DPMP	0.015	0.039	0.022
Adjusted R-square model	0.963	0.988	0.988
RSM - model error	0.013	0.009	0.014
Press - prediction error	0.013	0.010	0.015
Mean of Response - HGV	0.669	0.782	0.978

The maximum desirability for the LGV response is achieved by setting the double purge parameters to DPRC=A, MP=84 and DPMP=88 which is the expected response that minimizes the recycling of impurities (Figure 4.21 a, b, c).

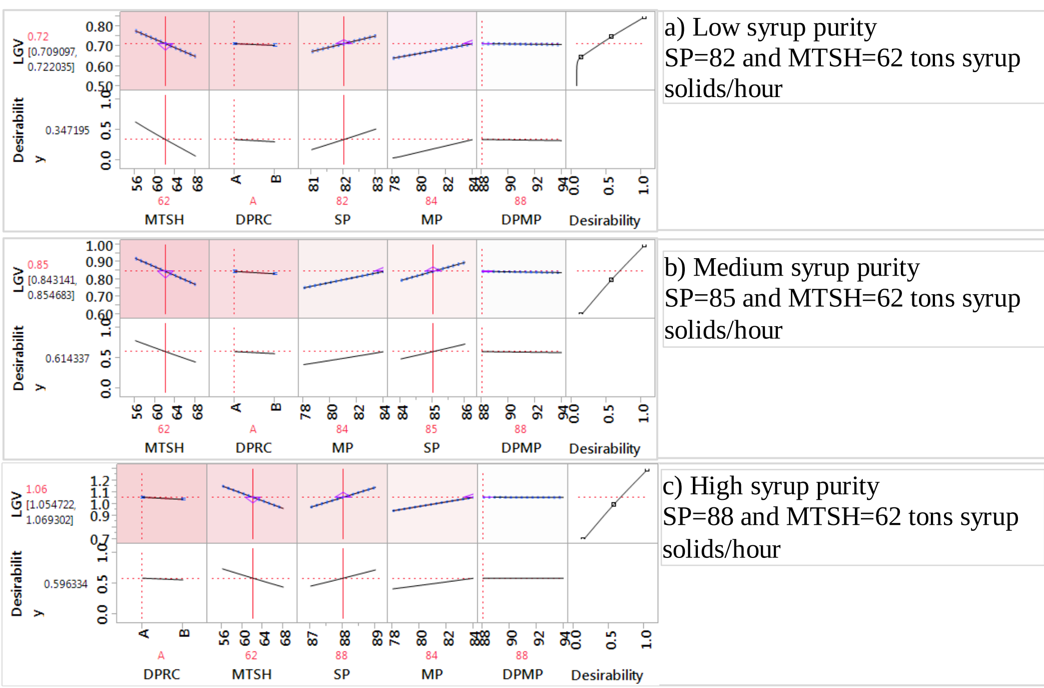
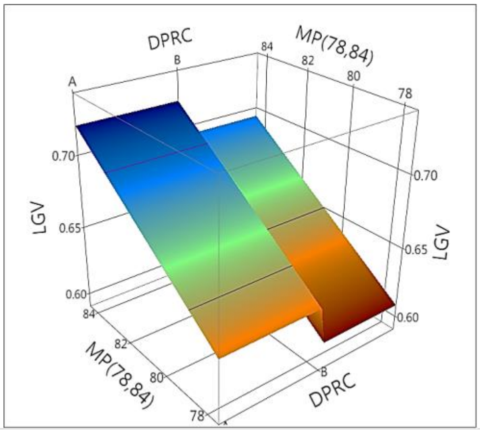


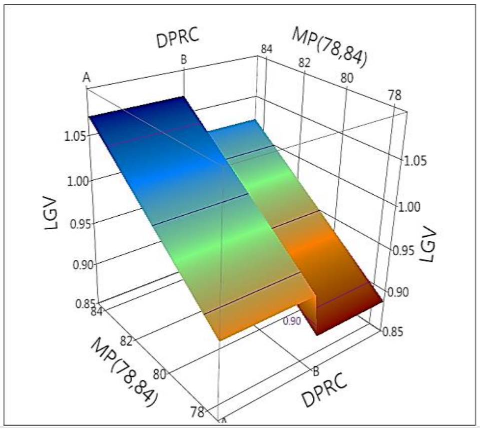
Figure 4.21 Low grade volume ratio (LGV = installed/required volume capacity) response prediction and desirability profiles at different syrup purity scenarios: a) Low, b)Medium and c) High. Controlled double purge parameters are DPRC, MP and DPMP (fixed MTSH and SP)

Notice that the mean values for the LGV responses indicate that, for an average solids input (MTSH=62), the required pan capacities are below the installed capacity for each scenario, which affects the exhaustion of final

molasses. This response is pertinent for the optimal integration of the double purge of C-magma system. To maximize the low grade volume ratio response (LGV) the best double purge parameters are the MP=84 and DPRC=A. Figure 4.22 (a, b) illustrates that there are more effective uses of the low grade pans when the recycle point is DPRC=A.



a) LGV versus DPRC and MP at low syrup purity (82)



b) LGV versus DPRC and MP at high syrup purity (88)

Figure 4.22 Response surface plots for low grade volume ratio (LGV=installed/required volume capacity), LGV by DPRC and MP at : a) Low syrup purity and b) High syrup purity (fixed MTSH=62 tons syrup solids/hour)

Sugar cane factories in Australia, producing raw sugar using a three boiling scheme and single purge, control the purity of the single magma between 87 and 90, while a purity of 82 is considered a poor purging efficiency (Broadfoot and Pennisi 2002). Jullienne (1982) performed tests on continuous centrifuges in South Africa proving that the purity of final molasses increases on 0.1 for one point of purity increase on C-magma. The objectives of the double purge system are not only to reduce color on raw sugar but also to reduce sugar losses on final molasses, hence minimizing the amount of water required to produce high purity magma in a single purge system.

4.3.8. Single Response Optimization Summary

The primary conclusion from the evaluation of the individual surrogate models is that they reflect the behavior of each response for both the double purge controlled parameters and for the random parameters. In general, main parameters and interactions are the same in all scenarios. Random parameters variation has significant effects on the responses, reducing the desirability of the response for a fixed value of the random parameter. For instance, syrup purity is important for LGV, RSREC and GJ. At high syrup purity, the desirability of RSREC is higher at 89 syrup purity (0.87) than for 87 syrup purity (0.42). MTSH is important for HGV and LGV (Table 4.12). The values of the parameters change with the desired response. Thus, to define the optimal values a decision must be made as to which responses will be used for the multiple criteria optimization and the weight of each response.

Table 4.12 Single response optimization of double purge product parameters for low syrup purity (LSP=82) and for high syrup purity (HSP=88). SYCOL=20,000CU, MTSH=62 and MB=93

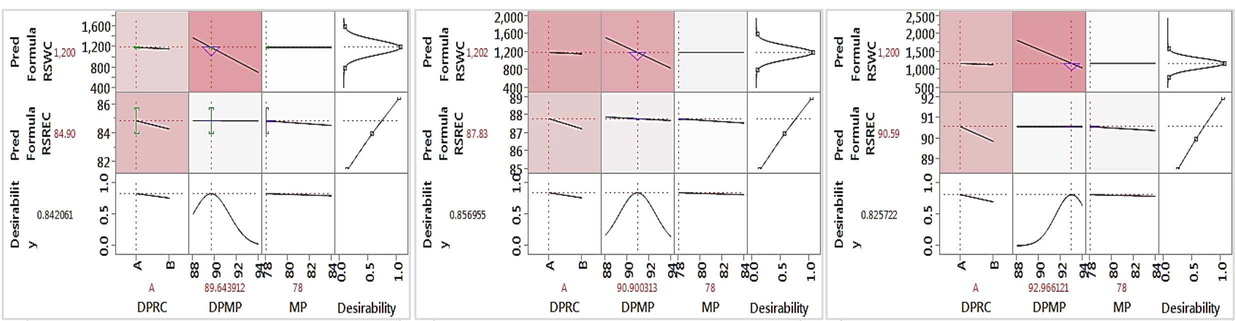
Response\Parameter	Scenario	DPMP	MP	DPRC	Response	Desirability
RSWC	LSP	88	**-**	A	1,200	1.00
Raw Sugar Whole Color, CU	HSP	92	**-**	A	1,200	1.00
RSREC	LSP	88	78	A	84.9	0.68
Sugar Recovery, %	HSP	94	78	A	90.6	0.64
GJ	LSP	87	85	B	2.30	0.87
Heating, gigajoules/ton solids	HSP	88	85	B	2.22	0.88
HGV	LSP	94	84	B	1.19	0.55
High grade volume ratio	HSP	94	84	B	1.18	0.55
LGV	LSP	88	84	A	0.72	0.35
Low grade volume ratio	HSP	88	84	A	1.06	0.60

4.4. Multiple Response Optimization for Different Syrup Purity Scenarios

Sugar quality, sugar yield, energy and volume process capacity of the crystallization station (denominated boiling-house) are important responses for a combination of multiple factors (input, process and product parameters) for raw sugar production. Some factors produce simultaneously positive or negative effects for each response. Traditionally, trial and error is used by engineers to select the product parameters at each crystallization stage and centrifugal separation, observing a complete cycle may last from 3 to 5 days Decisions are made in the factory based on the experience. Computer simulations and optimization can be developed as tools to improve decision making. Data collection, statistical analysis, modeling, experimental design, simulation and optimization (multiple criteria decision making) were applied to optimize integration of double centrifugation of C-magma to improve the quality of raw sugar to a target value defined by refinery requirements at a Louisiana sugar mill. The evaluation of the multiple-response optimization was performed sequentially first for RSWC and RSREC, then responses RSWC with RSREC and GJ and RSWC with RSREC and LGV were combined, and, finally all responses, RSWC with RSREC, GJ, HGV and LGV were combined. The random variables SP, SYCOL, MTSH and MB were fixed to isolate the effect of the controlled variables. The color intensity and the slope of the response lines in the plot matrix indicate the importance and sensitivity of the response for each parameter (SAS 2014).

4.4.1. Multiple-Response Optimization for RSWC and RSREC

For the same syrup color input (SYCOL=23,000 CU), the maximum desirability for two responses RSWC and RSREC was achieved by setting the system with recycle to A molasses (DPRC=A) and the lowest first magma purity (MP=78) for all simulation scenarios, assigning the same importance to the responses (Figure 4.23). The solution was influenced by the importance and direction of the effect of the parameter DPMP on RSWC and the importance and direction of the effect of DPRC on RSREC. The purity of DPMP increases from low syrup purity (DPMP~90) to high syrup purity (DPMP~93), since the amount of color is related to the non-sucrose components, which increase from the low to the high syrup purity scenario.

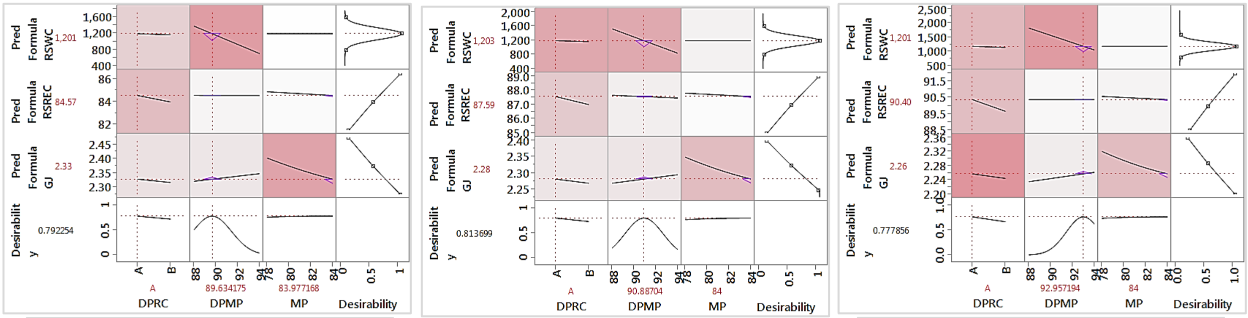


a) Low syrup purity (82) | b) Medium syrup purity (85) | c) High syrup purity (88)

Figure 4.23 Optimization profile plots for maximum desirability with two responses RSWC and RSREC at: a) Low, b) Medium and c) High syrup purity scenarios. Optimization settings are targeting RSWC=1,200 CU and maximizing RSREC with equal importance (w=1). (fixed SYCOL=23,000 CU, MB=93)

4.4.2. Multiple-Response Optimization for RSWC, RSREC and GJ

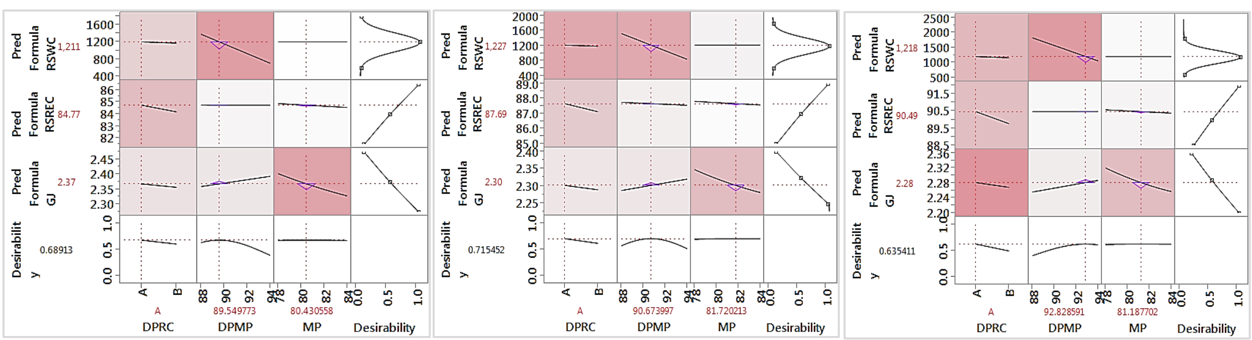
For the same syrup color input (SYCOL=23,000 CU), the maximum desirability for the RSWC (w=0.6), RSREC (w=0.3) and GJ (w=0.1) responses was achieved by setting the system with recycle to A molasses (DPRC=A) and the highest first magma purity (MP=84) for all simulation scenarios (Figure 4.24 a, b, c). The solution, adding the GJ response, changed because of the high effect of MP in heating requirements (GJ). The high setting for the first magma purity reduced significantly the recirculation of non-sucrose that, in turn, reduced the volumes of low grade materials. However, the sugar recovery (RSREC) is 0.33 points lower for the low syrup purity and 0.19 points lower for the high syrup purity scenarios, compared to the recovery by optimizing two responses (RSWC and RSREC) only.



a) Low syrup purity (82)
b) Medium syrup purity (85)
c) High syrup purity (88)

Figure 4.24 Multiple response optimization profile plots for maximum desirability with three responses RSWC, RSREC and GJ at: a) Low, b) Medium and c) High syrup purity scenarios. Optimization objectives are targeting RSWC(w=0.6)=1,200 CU, maximizing RSREC(w=0.3) and minimizing GJ(w=0.1) (SYCOL=23,000 CU, MB=93)

Assigning a higher importance to sugar recovery RSREC (w=0.7) and a lower importance to RSWC (w=0.2), the multiple response optimization solution increased the sugar recovery response (RSREC). Compared to optimizing two responses (RSWC and RSREC) the difference is 0.13 and 0.10 for the low and the high syrup purity scenario, respectively (Figure 4.25). The main change is that the optimal value for the product parameter MP is lower (MP~81) compared to MP=84. The optimal values for the parameter DPMP correspondingly for each scenario are 90, 91 and 93. In the long run, the decimal points on the RSREC responses are significant in terms of sugar produced and financial profit. The weight of the response can be adjusted to improve the optimization results.



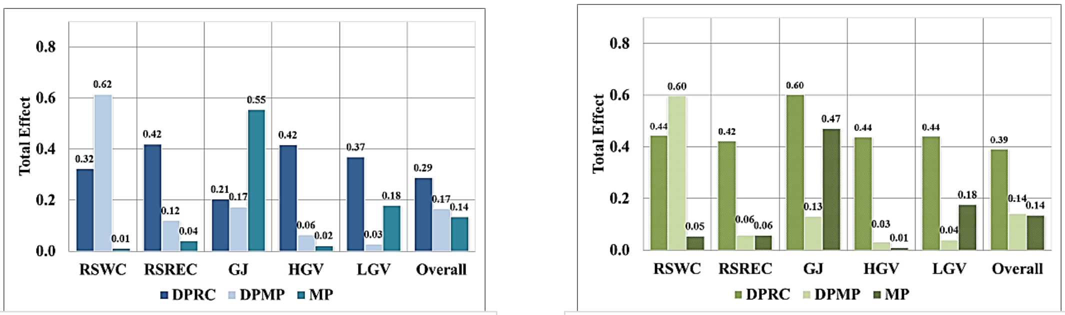
a) Low syrup purity (82)
b) Medium syrup purity (85)
c) High syrup purity (88)

Figure 4.25 Multiple response optimization profile plots for maximum desirability with three responses RSWC, RSREC and GJ at: a) Low, b) Medium and c) High syrup purity scenarios. Optimization objectives are targeting RSWC(w=0.2)=1,200 CU, maximizing RSREC(w=0.7) and minimizing GJ(w=0.1) (SYCOL=23,000 CU, MB=93)

4.4.3. Multiple-Response Optimization for RSWC, RSREC, GJ, HGV and LGV

The multiple response optimizations for five responses (RSWC, RSREC, GJ, HGV and LGV) will be studied focusing on the extreme scenarios for the boiling house operation: low syrup purity and high syrup purity. The importance assigned to each response is: w_{RSWC} =0.2, w_{RSREC}=0.65, w_{GJ}=0.05, w_{HGV}=0.05 and w_{LGV}=0.05)

Assessing the importance of the double purge product parameters (DPRC, DPMP and MP) on the five responses, it was found that DPRC has the greatest overall effect compared to DPMP and MP, and that the effect of DPRC was higher at high syrup purity (0.39) compared to the total effect of DPRC at low syrup purity (0.29). The effect of DPRC on the GJ response at high syrup purity is approximately three times greater than it was for the low syrup purity scenario. The highest effect of DPMP was for the RSWC response and the highest effect of MP was for the GJ response; MP can also be considered important for LGV response in both scenarios (Figure 4.26 a, b).

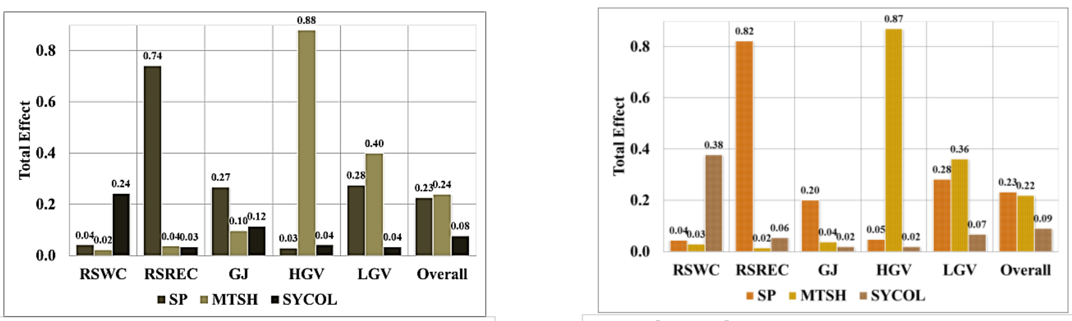


a) Low syrup purity scenario (82)

b) High syrup purity scenario (88)

Figure 4.26 Double purge total effect bar plots (dependent resampled inputs) for multiple response optimization with five responses RSWC, RSREC, GJ, HGV and LGV at: a) Low and b) High syrup purity scenarios. Controlled product parameters DPRC, DPMP and MP

Assessing the importance of the chosen random parameters (SP, MTSH and SYCOL) on the five responses, it was found that SP and MTSH have the highest overall effect (Figure 4.27 a, b). SYCOL is only important for RSWC; MTSH is important for HGV and LGV and; SP is important for RSREC, GJ and LGV.

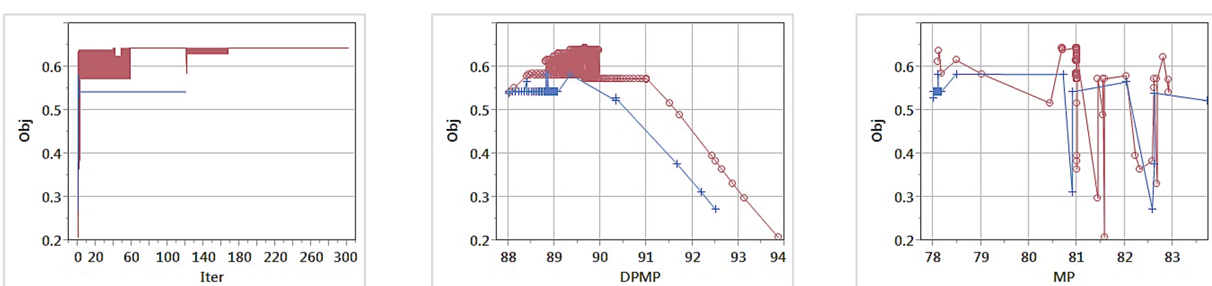


a) Low syrup purity scenario (82)

b) High syrup purity scenario (88)

Figure 4.27 Double purge total effect bar plots (dependent resampled inputs) for multiple response optimization with five responses RSWC, RSREC, GJ, HGV and LGV at: a) Low and b) High syrup purity scenarios. Random parameters SP, MTSH and SYCOL

Figure 4.28 (a) shows the convergence of the optimization results for the objective function (maximizing overall desirability). Figure 4.28 (b and c) show the exploration inside the design space (continuous parameters DPMP and MP and the categorical parameter DPRC). The student error and the interquartile range for the best in trip overall desirability are closer to the convergence tolerance of 0.000001.



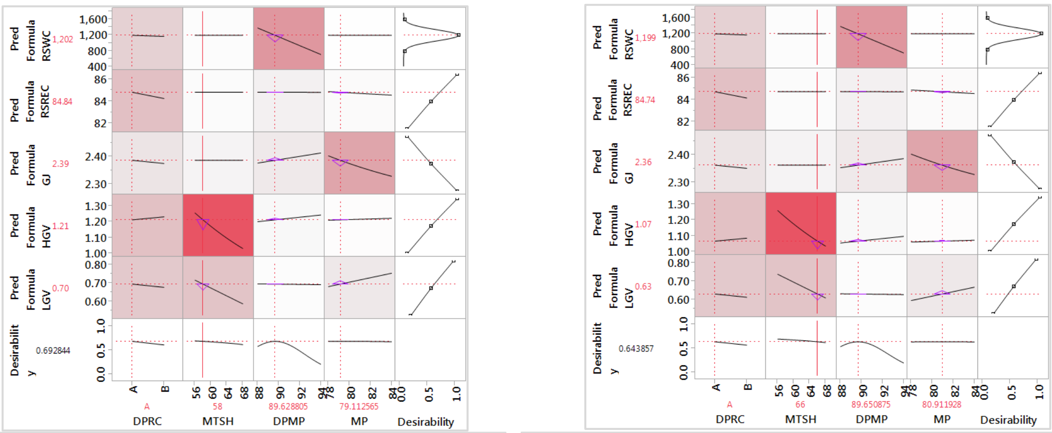
a) Iteration number versus maximization objective

b) DPMP versus maximization objective

c) MP versus maximization objective

Figure 4.28 Multiple response (5 responses) optimization convergence (SP=82 and MTSH=66) plots: a) iteration number versus objective, b) DPMP vs objective and c) MP versus objective. Red lines for DPRC=A and blue lines for DPRC=B are used to illustrate the different objective values for this parameter, these points are not in sequence. Best maximum desirability (objective) = 0.64388, best in trip student error (N=20) = 0.000003, best in trip interquartile range ($Q_{75\%}$-$Q_{25\%}$) = 0.000001 and total number of runs = 1,739 (JMP(R) 2014)

Comparing the optimization profiles for a low and high syrup solids input to the boiling house at the same low syrup purity, it was found that the optimal settings for the double purge of C-magma were recycle of double purge molasses to A molasses (DPRC=A), double purge magma purity, DPMP~89.6, changing only the purity of the first magma from MP~79 for the low (MTSH=58) to MP~81 for the high solids inputs (MTSH=66) (Figure 4.29 a, b). The increased purity MP, helped to reduce heating requirements and maximize the effective use of low grade pans (minimize GJ and maximize LGV) since this change reduces the recycle of non-sucrose components.

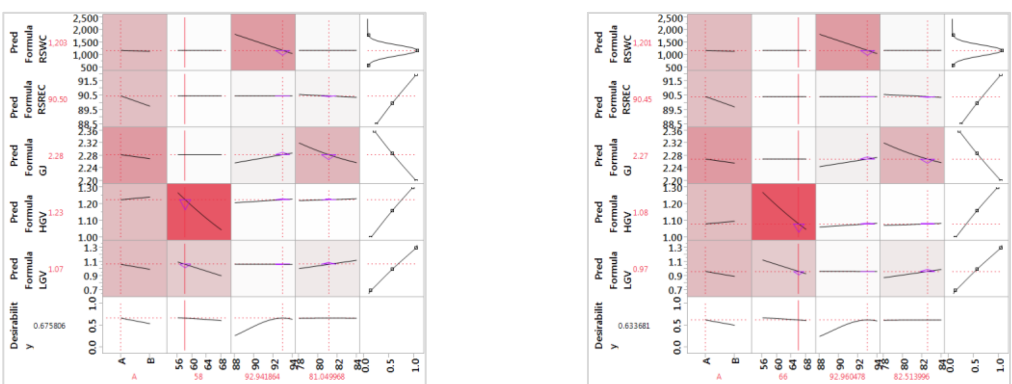


a) Low syrup purity (82) and low syrup solids rate (MTSH = 58 tons syrup solids/hour)

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

Figure 4.29 Low syrup purity multiple response optimization profile plots for maximum desirability with five responses RSWC, RSREC, GJ, HGV and LGV at a) MTSH=58 tons syrup solids/hour and b) MTSH=66 tons syrup solids/hour. Optimization objectives are targeting RSWC(w=0.2)=1,200 CU, maximizing RSREC(w=0.65), minimizing GJ(w=0.05), maximizing HGV (w=0.05) and maximizing LGV(w=0.05) (fixed SYCOL=23,000 CU, MB=93)

Compared with the optimal double purge parameters for the low syrup purity scenario, the solution is more highly influenced by the high level of color compounds in the syrup. The color/non-sucrose ratio for syrup with SP=82 and SYCOL=23,000 CU is 1,278 while for syrup with SP=88 and the same SYCOL=23,000 CU is 1,917. The optimal recycle of double purge molasses is to A molasses (DPRC=A) and the optimal purity of double purge magma is DPMP~93. The purity of the first magma increases from MP~81 for the low solids input (MTSH=58) to MP~82.5 for the high solids inputs (MTSH=66) (Figure 4.30 a, b).

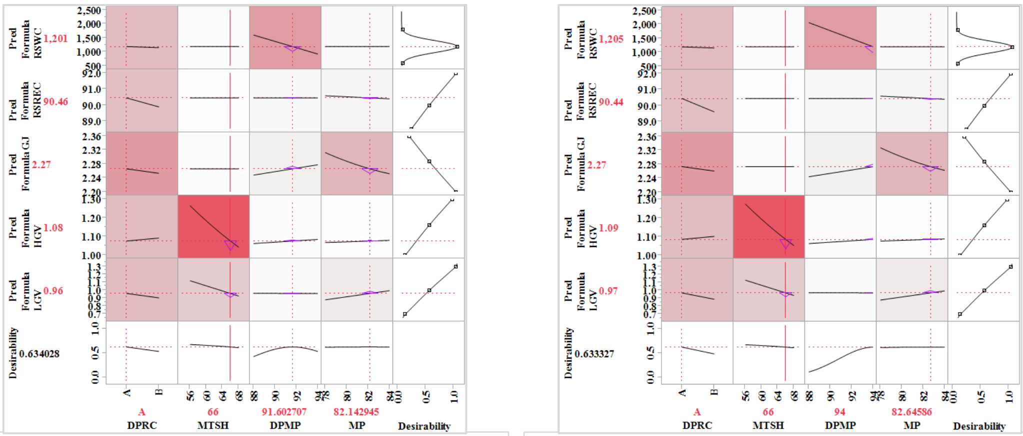


a) High syrup purity (88) and low syrup solids rate (MTSH = 58 tons syrup solids/hour)

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

Figure 4.30 High syrup purity multiple response optimization profile plots for maximum desirability with five responses RSWC, RSREC, GJ, HGV and LGV at a) MTSH=58 tons syrup solids/hour and b) MTSH=66 tons syrup solids/hour. Optimization objectives are targeting RSWC(w=0.2)=1,200 CU, maximizing RSREC(w=0.65), minimizing GJ(w=0.05), maximizing HGV (w=0.05) and maximizing LGV(w=0.05). (fixed SYCOL=23,000 CU and MB=93)

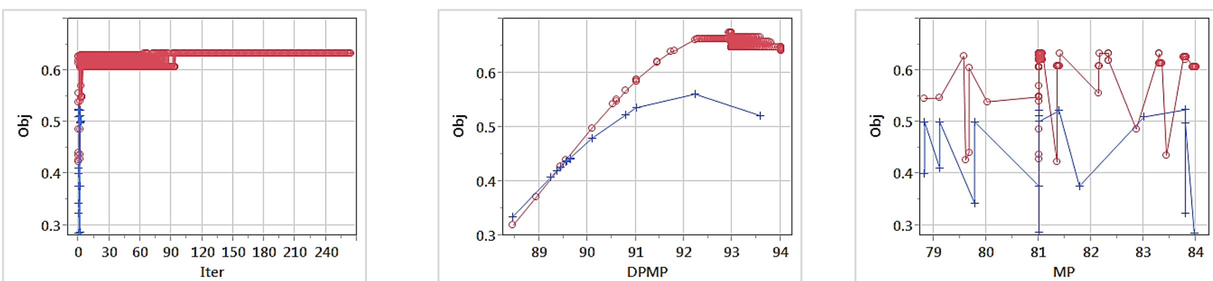
Comparing the optimization profiles for a high syrup purity scenario (88) by varying the color input from 20,000 CU to 26,000 CU (SYCOL) and fixing MTSH to 66, it was found that the optimal settings for MP were approximately the same (82.1 and 82.6), while the optimal value of DPMP increased from ~92 to 94 (Figure 4.31 a, b). The increased purity of DPMP caused a reduction of 0.02 points on the RSREC response. The 0.02 points reduction on RSREC when MTSH=66 and SP=88 is equivalent to the loss of 279 kg of sugar/day



a) SYCOL=20,000 CU (SP=88 and MTSH=66)　　b) SYCOL=26,000 CU (SP=88 and MTSH=66)

Figure 4.31 High syrup purity multiple response optimization profile plots for maximum desirability with five responses RSWC, RSREC, GJ, HGV and LGV at syrup color input a) SYCOL= 20,000 CU and b) SYCOL= 26,000 CU. Optimization objectives are targeting RSWC(w=0.2)=1,200 CU, maximizing RSREC(w=0.65), minimizing GJ(w=0.05), maximizing HGV (w=0.05) and maximizing LGV(w=0.05)

Figure 4.32 (a, b, c) shows the convergence of the optimization results for the objective function (maximum overall desirability) inside the design space (parameters range of variation). The student error and the interquartile range for the best in trip overall desirability are just over convergence tolerance of 0.000001.



a) Iteration number versus maximization objective　　b) DPMP versus maximization objective　　c) MP versus maximization objective

Figure 4.32 Multiple response (5 responses) optimization convergence (SP=88 and MTSH=66) plots: a) iteration versus objective, b) DPMP versus objective and c) MP versus objective. Red lines for DPRC=A and blue lines for DPRC=B are used to illustrate the different objective values for this parameter, these points are not in sequence. Best maximum desirability (objective) = 0.64368, best in trip student error (N=20) = 0.000045, best in trip interquartile range ($Q_{75\%}$-$Q_{25\%}$) = 0.000057and total number of runs = 2,099 (JMP[(R)] 2014)

4.4.4. Multiple Response Optimization Summary

The initial trials optimizing the double purge of C-magma product parameters and recycle of wash for RSWC and RSREC showed that the most important parameters for these responses were DPRC because of its effect on RSREC, and DPMP because of its effect on RSWC. The parameter MP does not have a significant effect on the responses RSWC and RSREC. Adding a third response (GJ), for which the parameter MP has a higher importance, allowed an assessment of the value of this parameter. The adjustment of the weight of the RSREC when using three responses (RSWC, RSREC and GJ) showed that it can be used to increase the value of this response (RSREC).

Table 4.13 summarizes the results for the combination of five responses (RSWC, RSREC, GJ, HGV and LGV). Although SP and MTSH have strong effects on the factory performance responses (depending on variation range) the double purge parameters have an equivalent importance for the overall system (combine responses).

Table 4.13 Multiple response optimization for five response of double purge product parameters for low syrup purity (LSP=82), medium syrup purity (MSP=85) and high syrup purity (HSP=88). Color input with syrup SYCOL=23,000 CU (fixed)

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

4.5. Conclusions

The response surface strategy is basically a methodology applied to a real or modeled system to create simple models that relate controlled parameters with one or several responses, such as product quality, process performance and process costs or revenues. The selection of the type of experimental design depends on the research goals – sensitivity analysis or optimization. Statistical analysis of experimental designs for model creation (analysis of variance – ANOVA) is based on the existence of random error; however, this approach has been applied to deterministic computer simulation recognizing the restrictions. Surrogate models are an approximation of the behavior of the response, or responses, that need to be evaluated. Surrogate models allow the determination of important parameters and the direction of the response; thus, they can be applied to optimize a system (Chen, Tsyum et al. 2003; Tekin and Sabuncouglu 2004; Kleijnen 2005; Kleijnen 2007; Vining 2008). The optimization of complex systems must take into account constraints or other responses that are important for the optimum performance of the whole system (Park and Kwang-Jae 2005; Cavazzuti 2013). The challenge of multiple response optimizations is that responses can be conflicting, or the overall objective function is non-linear or discontinuous – not differentiable. A desirability function transforms the results of an individual response into a scalar value that ranges from 0 to 1 depending on the desired type of response: meeting a target, maximization or minimization. The overall desirability combines individual response desirability by transforming the multiple response optimizations into a single response optimization – maximizing the overall desirability (Del Castillo, Montgomery et al. 1996; Baş, Arslan et al. 2010; Bera and Mukherjee 2010). Different optimization strategies have to be applied to guarantee the location of the best parameters on the whole design space – global optimal (hybrid algorithms).

The primary goal on this research was to find the optimal settings to integrate a double purge of C-magma system into a three-boiling crystallization scheme in producing raw sugar, for reducing whole color over a range

close to a target value. The surrogate models were produced through virtual experiments – simulations (Sugars™ 2014) programmed using the I-optimal experimental design for three different syrup purity scenarios and defined random and controlled parameters (JMP(R) 2014). Statistical analysis of simulation responses with respect to the controlled double purge parameters and random parameters was used to construct surrogate models for each response and syrup purity scenario. Primary effects and interactions utilizing individual responses in a scenario were practically the same for other scenarios. The main difference between models for each scenario was different regression coefficients for primary effects and interactions, changing the importance of the parameters in each scenario. The behavior of individual responses with respect to the variation of individual parameters confirmed the validity of the surrogate models.

Optimization for individual responses produced different optimal parameters for a double purge system. The successive combination of the goal response RSWC (raw sugar whole color) with RSREC response (raw sugar recovery) and then the addition of the GJ (heating requirements) response showed that other controlled parameters were becoming important for the overall system, and that an adjustment of the weight of each response can be applied to improve the desired response (RSREC). A multiple response optimization including five responses for a low syrup purity scenario (same syrup color input) for low and high syrup solids inputs showed that increasing the value of the MP parameter (79 to 81) helped to manage high energy requirements (GJ) and made more effective use of low grade pans (LGV) while producing the same target whole color (1,200 CU). The same optimization comparison at high syrup purity showed that an increment on the MP parameter (81 to 83) would have the same effect on the GJ and LGV responses. The random parameter SYCOL had a high effect on RSWC and its effect changed with the syrup purity. Low values on SYCOL will require lower purities of DPMP to produce the same color target. The multiple response optimization approach to the integration of the double purge system gave the approximate best product parameters (DPMP and MP) and the best location for the recycle of double purge molasses DPRC. The optimal setting of DPRC was found to be A, this point of recycle does not appear on any publication about double purge of C-magma (Geerligs 1909; Jullienne 1989; Chou 2000; Madho and Davis 2008). Simulation models, surrogate models and optimization approach can be applied to optimize specific product parameters (Temperature, pressure, concentration, time, etc.) for individual stages or to define product parameter specifications on complex system such as the crystallization process that involves several stages. The obtained results are in agreement with actual factory data.

CHAPTER 5 SUMMARY AND CONCLUSIONS

The integration of double purge of C-magma to a three boiling crystallization scheme for the improvement of raw sugar whole color has been a comprehensive engineering project involving several stages, specifically:

1) Initial modeling to determine required equipment capacities and to best guess the values for the controlled product parameters (first and double purge magma purity)
2) Planning resources and required sampling and analysis
3) Trials and full scale plant implementation (execution)
4) Monitoring and evaluation of results through statistical analysis
5) Model improvement and validation with actual data
6) Application of a multiple response optimization strategy (experimental design and analysis of computer simulations, metamodelling and application of desirability functions)

These concluding remarks are divided into three groups: implementation, modeling and optimization.

5.1. Implementation

In the first year (2012), a new double purge system for C-magma reduced ~ 47% of raw sugar whole color compared to raw sugar produced by a single purge of C-magma (2,923 CU compared to 1,552 CU at 1.2μm, 8.5pH). Table 5.1 summarizes the results obtained at three factories (LU, WF and CT) in the 2013 Louisiana sugarcane crop season. Product parameters and performance of the system and boiling house were monitored continuously. It was shown that double purge produces lower raw sugar whole color and conductivity ash values avoiding the higher capital and energy investment required for other different boiling scheme. The same three factories continue to operate the double purge of C-magma system during the 2014 sugar cane crop season.

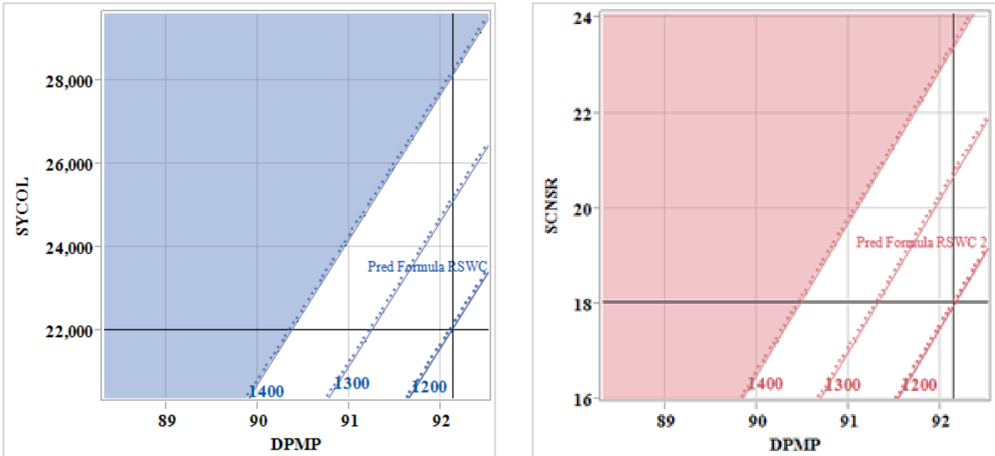
Table 5.1 Double purge system average product parameters and raw sugar polarization and color (2013) by factory: Lula (LU), Westfield (WF) and Cora-Texas (CT)

Product	Parameter	Factories (2013)		
		LU	WF	CT
C-Magma	Purity,%	81.3	81.4	81.2
DP Magma	Purity,%	92.3	90.0	90.5
DP Molasses	Purity,%	63.1	65.7	64.9
	Recycle Point	A	A	B
Raw Sugar	Polarization, °Z	99.2	99.3	99.2
	Whole Color, CU	1,164	1,536	1,675
	Affinated Color, CU	709	666	
*Color @ 8.5pH & 1.2 μm				

5.2. Modeling

The primary objective of the modeling stage was to create a simulation model specific for Lula factory in order to quantify the effects of double purge on the product parameters of color of raw sugar, sugar recovery, energy and massecuites processing volumes as well as to forecast the effects of random variables. A boiling house model created using a software specific for the sugar industry (Sugars^{TM} 2014) was adjusted according to real data of purity and color profiles. The correlation coefficient between observed and modeled color profiles was 0.997. The main adaptation of the model to integrate the double purge of C-magma was the addition of color separation units before the A and B centrifuges. The function of the color separation was to apply a small correction (~0.01%) on the color of A and B sugars according to the purity of the double purge magma. Weekly composite analysis of boiling house inputs and outputs (syrup, final molasses and raw sugar) were modeled to estimate correlations between double purge magma purity and the average (A and B) settings of the color separation units. It was found that double purge magma purity (DPMP) and syrup color (SYCOL) are linearly correlated to the whole color of raw sugar (adjusted R-square=0.91). Figure 5.1 (a, b) shows the contour lines for raw sugar whole color (1,200, 1,300 and 1,400 CU) by

syrup color and syrup color/non-sucrose ratio with double purge of C-magma purity, RSWC at the shaded region is > 1,400 CU. These graphs highlight the sensitivity of the color of raw sugar to the ratio between color and impurities (non-sucrose on soluble solids) on the syrup. The quality of the input is important not only for the quality of the product but also for the required double purge C-magma purity.



a) Contour plot: DP magma purity versus syrup color

b) Contour plot: DP magma purity versus syrup color/ns ratio*10^4

Figure 5.1 Raw sugar whole color contour plots for double purge magma purity (DPMP) versus a) syrup color (SYCOL) and b) syrup color/non-sucrose ratio (SCNSR*10^4). Composite weekly sampling analysis of syrup, raw sugar and weekly average of double purge magma purity for Lula and Westfield 2013 season

Statistical analysis was applied to define correlations between molasses and massecuites at each centrifugation stage. Historical data was used to fix or define the variation range of several boiling house parameters and to outline the responses that acted together and are important in terms of optimal boiling house performance. For instance, an inverse prediction (Figure 5.2) showed that if the whole color target is set to 1,200 CU the affined color may range (95% probability) from 590 to 850 CU. The raw sugar factory will receive and additional premium on the basis price for affined color below 750 CU. The financial impact on raw sugar price could not be evaluated since this information is confidential.

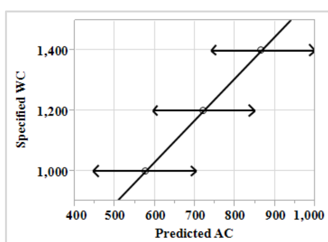


Figure 5.2 Inverse prediction plot for affined color (AF) versus specified whole color values, 95% confidence intervals (69 raw sugar samples Lula 2013)

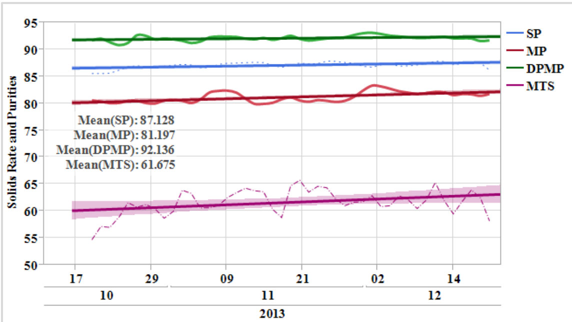
5.3. Multiple Response Optimization

The initial stage of any optimization strategy is to clearly define: goals (single or multiple criteria), optimization space (parameters, boundaries, constraints) and define one or multiple objective functions. The collected data from factory implementation and ad hoc simulations were combined to establish an initial hypothesis: there exists an optimal combination of double purge product parameters that need to be applied to produce raw sugar with a target whole color (1,200 CU); whereas, it is desirable to maximize the recovery of sugar, minimize heat requirements and maintain extra available installed equipment capacity (to account for death time and maintenance

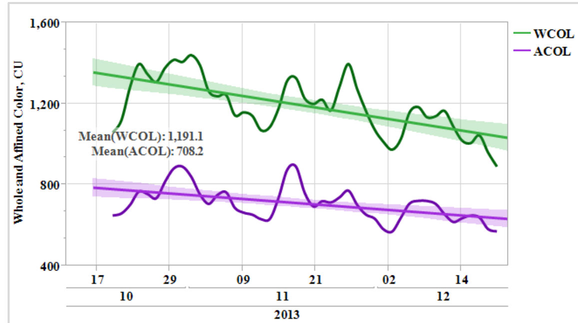
of pans). In addition, it was recognized that the optimal system setting are also depending on the processing scenarios, due to random variation on the quality of the input (linked to sugarcane quality). Simultaneous random variation of multiple factors, cyclic nature of the crystallization stages and long cycles (3 – 5 days) make impossible the straight application of surface response methodology at the boiling house of the factory. A complex boiling house model does not provide a direct relation among the double purge parameters and the desired quality and performance responses required for the direct application of optimization algorithms to the simulation model. These facts promoted the search for an alternative optimization strategy to approximate the behavior of the selected responses with prediction models (surrogate models), such as with response surface methodology applying computer simulations.

As with the surface response methodology, experimental designs were used to explore what were the main effects and interactions that the controlled product parameters (double purge system) and the boiling house random parameters were having on the wanted responses. Statistical analysis of deterministic computer simulation (like unreplicated experiments) was performed on the basis of the principle of "sparsity of effects" which declares that for "the majority of the systems some main effects and low order interactions are dictating while the high order interactions are unimportant". Linear surrogate models were created for the selected responses: color of raw sugar, sugar recovery, heating requirements and high grade and low grade massecuites processing volumes, related to actual installed capacity of the pans at Lula. The importance of the controlled and random parameters was evaluated for each response. Finally, the multiple-response optimization problem was transformed into a single optimization problem by the application of desirability functions. The individual desirability function transforms each response on a scalar value ranging from 0 to 1, while the geometric mean of the results is applied to obtain the overall desirability. The weight assigned to the individual desirability functions can be adjusted by the user according to the results. It needs to be pointed out that among the controlled parameters there was a categorical variable, the point of recirculation of double purge molasses requiring the application of mixed-integer optimization techniques. Statistical analysis, creation of predicted models, parameters importance evaluation and optimization were performed using the tools of an statistical software (JMP(R) 2014)

Among the controlling parameters double purge magma purity (DPMP) showed the greatest importance on the whole color of raw sugar, however, the point of recirculation of double purge molasses (DPRC) was the most important for the overall system at both the low and at the high syrup purity scenarios. The most important random parameters for the overall system were the syrup purity and the rate of soluble solids input to the boiling house. Two, three and five responses were combined individually to evaluate the effect on the optimal parameters selection. The number of responses used and the weight assigned to each response influenced the solution for the optimal settings of the double purge magma system. Using five (5) responses (RSWC, RSREC, GJ, HGV and LGV), the optimal double purge parameters simulating the same average syrup purity and rate of soluble solids input to the boiling house were in agreement with the parameters found on real operation of the system at Lula Sugar Factory in 2013. Figure 5.3 (a) shows the real trends and variation of the random (SP and MTS) and the controlled double purge product parameters (DPMP and MP), the recycle of double purge molasses was DPRC=A. The Figure 5.3 (b) shows the actual variation of whole and affined color of raw sugar. Figure 5.4 shows the optimal parameters determined by the optimization system (maximum desirability), simulating the system for the approximated mean values of syrup purity and soluble solids input to the boiling house from Lula 2013 data and assuming syrup color of 22,000 CU. The resulted optimal parameters to achieved 1,200 CU target whole color are: double purge magma purity of 91.9 (DPMP), magma purity of 80.6 (MP) and recycling double purge molasses to A molasses tank (DPRC). These optimal settings approximated match the actual settings of the double purge system (Lula 2013).



a) Controlled MP and DPMP, random SP and MTS – Actual Data 2013



b) Raw sugar whole and affined color (1.2 μm, 8.5pH) – Actual Data 2013

Figure 5.3 Line plots for a) random and controlled double purge product parameters daily average, and b) whole color and affined color daily average, Lula 2013

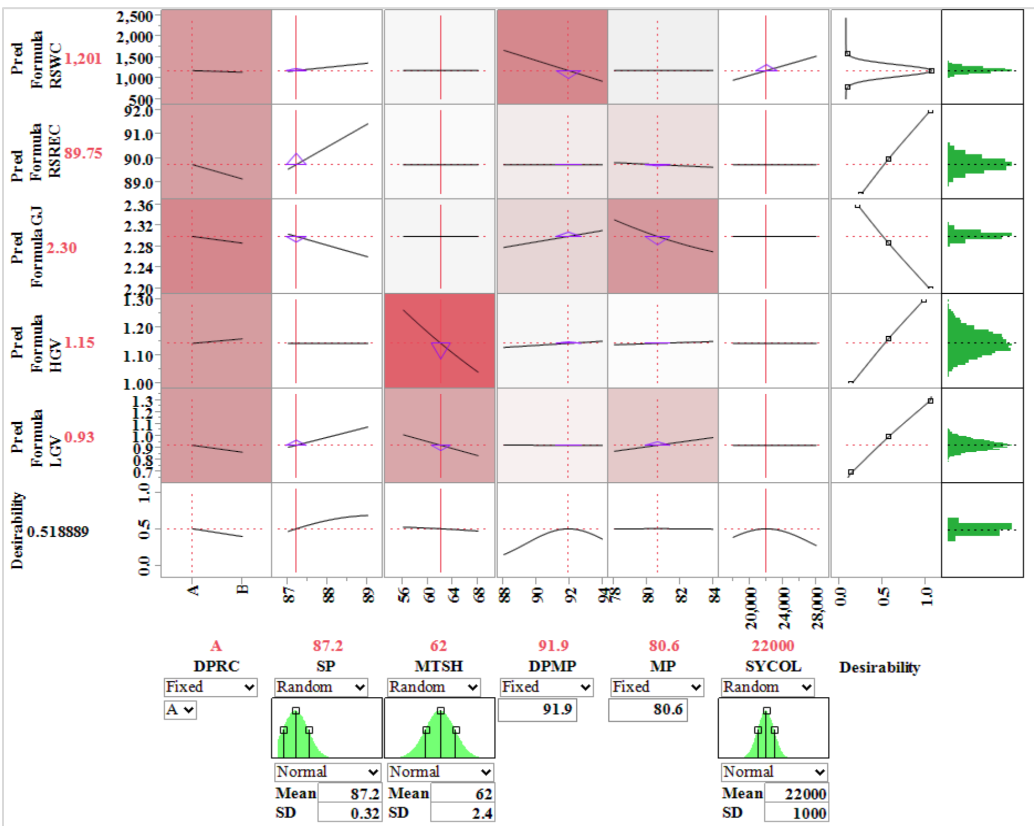


Figure 5.4 Responses and desirability plots for the product parameter optimization simulating the processing conditions at Lula in 2013 (assuming 22,000 CU mean syrup color)

Figure 5.5 illustrates on a contour plot what would be the optimal operation area (white) assuming low and high limits for the desired responses. There is still a need to identify what are the approximated maximum or minimum limits of each response. The definition of the response limits probably requires a multidisciplinary approach involving at least the financial and energetic goals of the factory.

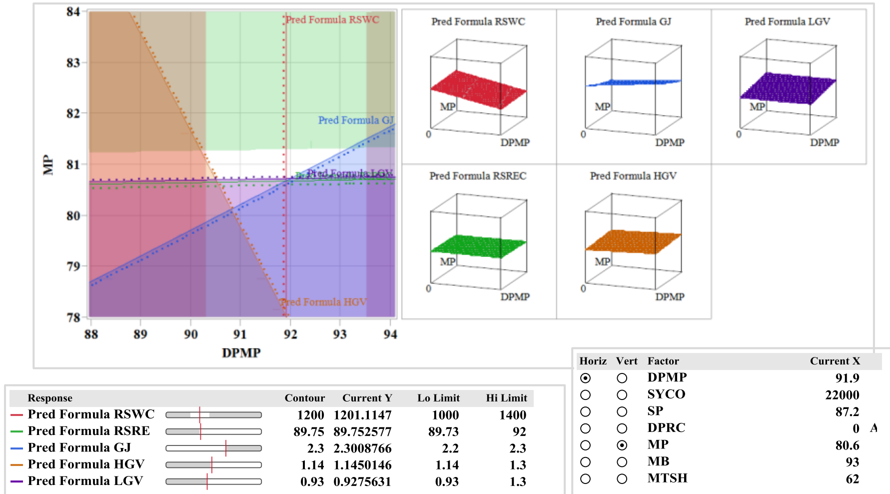


Figure 5.5 Contour and surface plots for double purge optimal product parameters. Assuming limits to the optimization responses: RSREC minimum of 89.73%, GJ maximum of 2.2 gigajoule/metric ton soluble solids (equivalent to 99 kg vapor-I/kg of soluble solid for a pan factor of 1.25), HGV minimum of 1.14 (Required/Installed capacity) and LGV minimum of 0.93

5.4. Conclusions

The whole crystallization stage (A, B, C) is a complex system that is affected by external and internal random variation. Part of the external and internal random variation was used besides the controlled double purge parameters to create experimental designs for running computer simulations. The experience gained from analysis of samples, analysis of factory reports and modeling of the boiling house with actual implementation data gave the required information to define the design space and the multiple optimization objectives. The surrogate models were used to assess the importance of the selected parameters on each response. Desirability functions were used to transform the prediction (surrogate) models into uniform scale objective functions to maximize, minimize and match a target. The desirability approach offers an easy and flexible way to adjust the objective of a particular response (changing the shape of the desirability function) and to adjust the weight of the responses. Finally, the optimization goal was to maximize the overall desirability a geometric mean of the individual responses desirability.

In general, this optimization strategy applied to the integration of the double purge of C-magma system gave results that are in agreement with actual operation at Lula factory. The definition of the boundaries of the system (e.g. low and high limits for processing rates and energy requirements) requires a team work with factory engineers. This specific approach through surrogate models and application of desirability functions to perform a multiple-response optimization is novel for the sugar industry and can be improved and adapted to optimize other process stages. The double purge of C-magma system to improve whole color of raw sugar was successfully implemented, modeled and optimized by learning from the real process through the intensive use of collected information.

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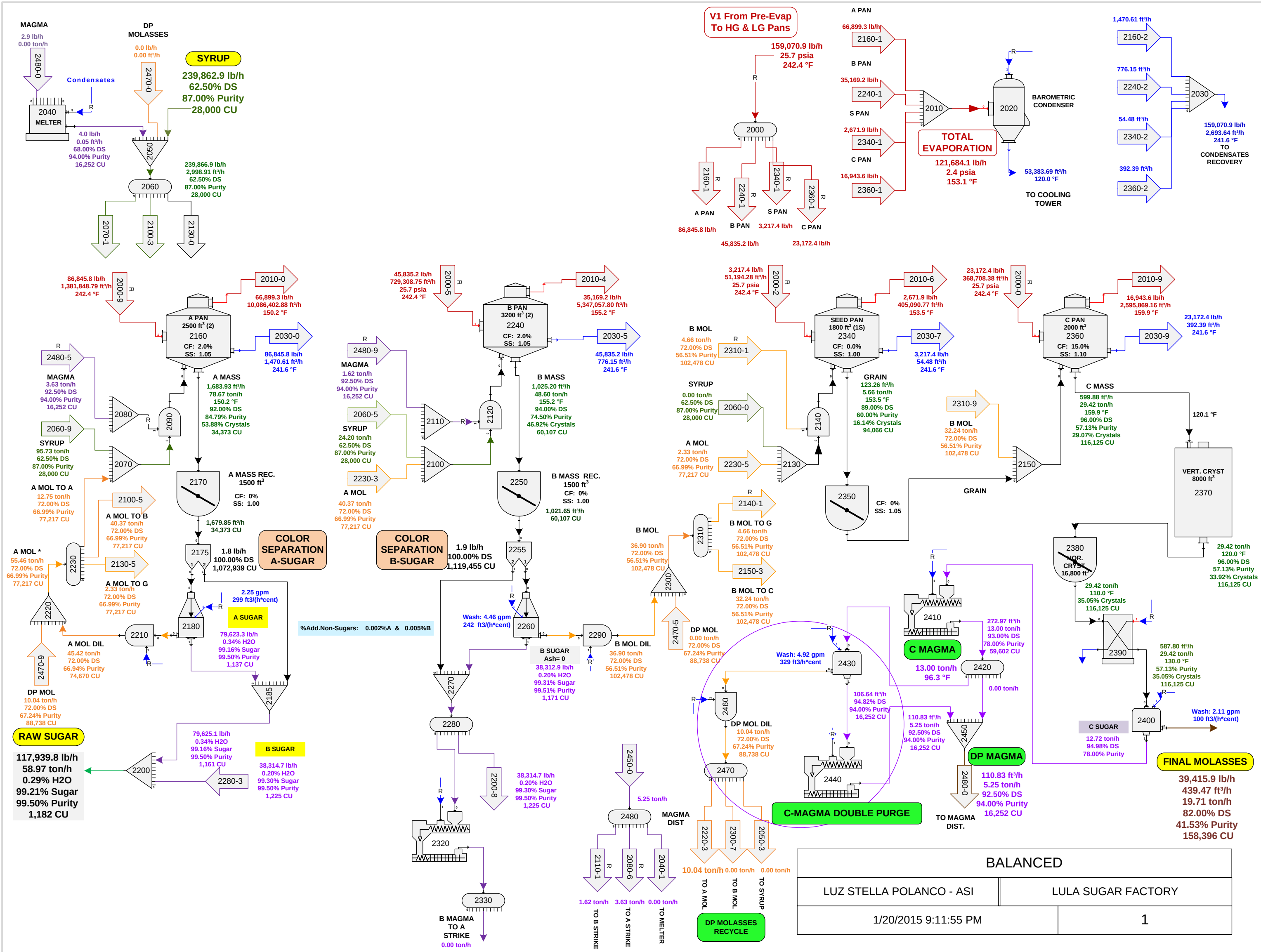
APPENDIX

A. Three-Boiling Scheme + Double Purge of C-Magma Simulation Model

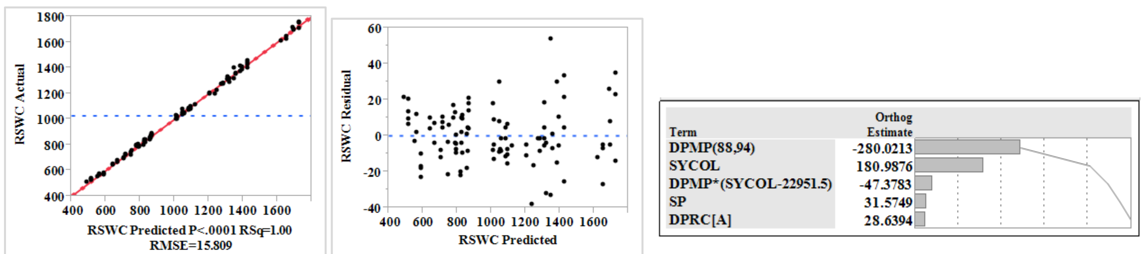


Conversion (SI units): 1 metric ton = 2,204.62262 lbs., 1.10231 short tons

B. Raw Sugar Whole Color (CU) – RSWC Model Summary

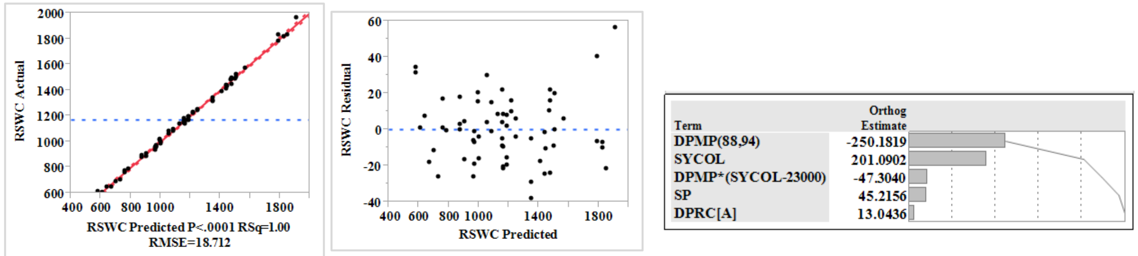
Table B.1 Parameters estimates and summary of model fit evaluation for raw sugar whole color – RSWC response at different syrup purity scenarios

Parameter Estimates	Low Syrup Purity			Medium Syrup Purity			High Syrup Purity		
Term	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**
Intercept	-3120.59	-20.82	<.0001	-5055.83	-20.30	<.0001	-8734.84	-20.99	<.0001
DPMP(88,94)	-338.30	-180.90	<.0001	-343.65	-119.20	<.0001	-388.31	-85.71	<.0001
SYCOL	0.05	118.88	<.0001	0.05	87.38	<.0001	0.06	67.19	<.0001
SP	37.86	20.81	<.0001	59.88	20.49	<.0001	99.47	21.18	<.0001
DPRC[A]	14.50	8.80	<.0001	14.28	6.12	<.0001	19.41	4.94	<.0001
DPMP*(SYCOL-SYCOL*)	-0.01	-30.41	<.0001	-0.01	-20.54	<.0001	-0.02	-14.48	<.0001
Summary of Fit									
R-Square	0.998			0.997			0.994		
Adjusted R-Square	0.998			0.997			0.993		
Root Mean Square - RMS	15.81			18.71			36.69		
Mean - RSWC	1,028			1,170			1,438		
Observations	103			66			88		
Prediction Error – (Press)	16.30			20.08			38.41		
SYCOL*	22,952			23,000			23,057		



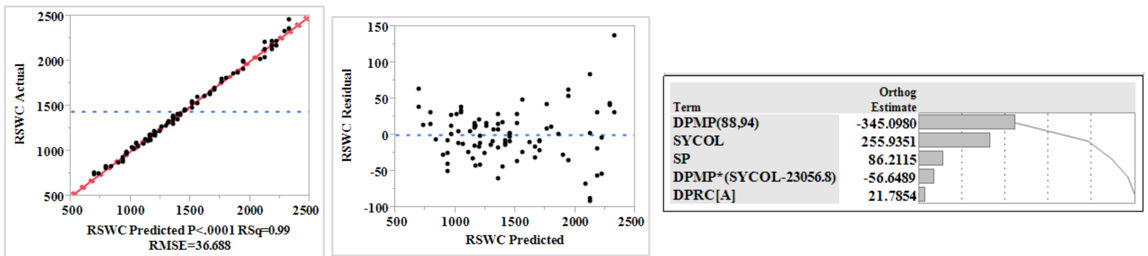
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure B.1 Low syrup purity scenario raw sugar whole color – RSWC plots



a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure B.2 Medium syrup purity scenario raw sugar whole color – RSWC plots



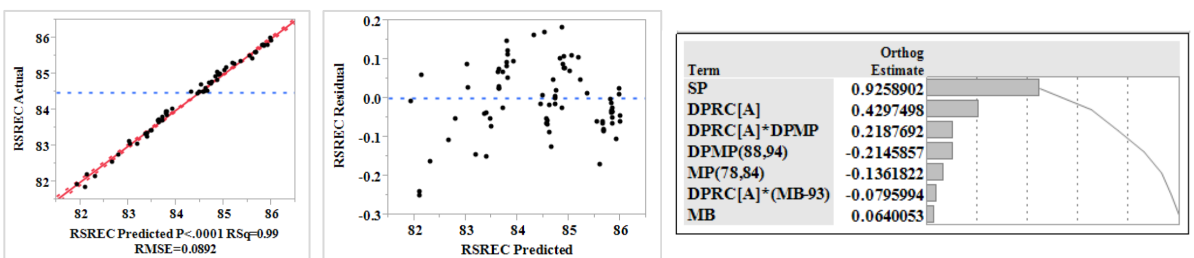
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure B.3 High syrup purity scenario raw sugar whole color – RSWC plots

C. Sugar Recovery (%) – RSREC Model Summary

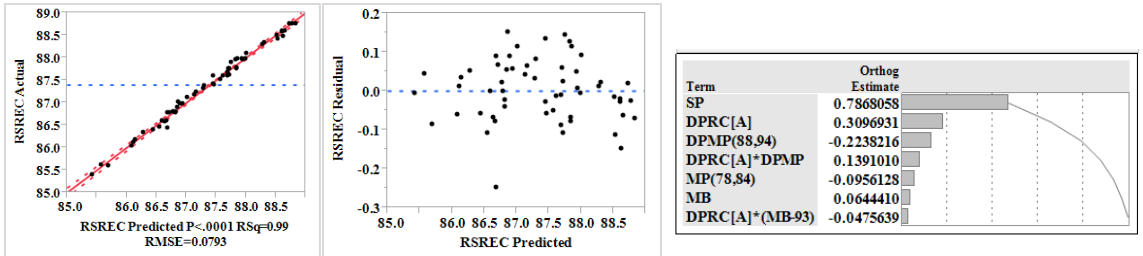
Table C.1 Parameters estimates and summary of model fit evaluation for sugar recovery – RSREC response at different syrup purity scenarios

Parameter Estimates	**Low Syrup Purity**			**Medium Syrup Purity**			**High Syrup Purity**		
Term	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**
Intercept	-14.33	-11.25	<.0001	-7.25	-4.94	<.0001	2.15	1.98	0.0506
SP	1.09	103.75	<.0001	1.02	79.03	<.0001	0.95	109.01	<.0001
DPRC[A]	0.40	45.60	<.0001	0.29	28.93	<.0001	0.25	34.15	<.0001
DPMP(88,94)	-0.27	-25.25	<.0001	-0.27	-21.80	<.0001	-0.17	-20.00	<.0001
MP(78,84)	-0.16	-13.40	<.0001	-0.12	-9.59	<.0001	-0.10	-11.80	<.0001
MB	0.10	7.99	<.0001	0.09	6.43	<.0001	0.05	6.29	<.0001
DPRC[A]*DPMP	0.26	24.77	<.0001	0.17	13.90	<.0001	0.17	20.16	<.0001
DPRC[A]*(MB-MB*)	-0.11	-9.06	<.0001	-0.06	-4.87	<.0001	-0.05	-6.00	<.0001
Summary of Fit									
R-Square	0.994			0.993			0.994		
Adjusted R-Square	0.993			0.992			0.994		
Root Mean Square - RMS	0.089			0.079			0.068		
Mean - RSWC	84.48			87.39			90.24		
Observations	103			66			88		
Prediction Error – (Press)	0.094			0.086			0.072		
MB*	93.0			93.0			93.0		



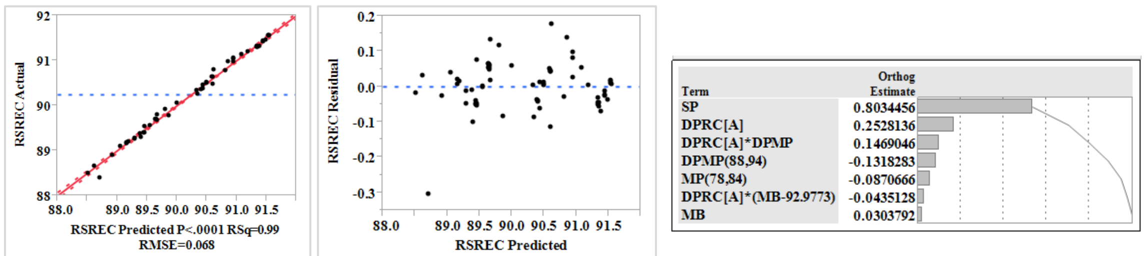
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure C.1 Low syrup purity scenario sugar recovery – RSREC plots



a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure C.2 Medium syrup purity scenario sugar recovery – RSREC plots



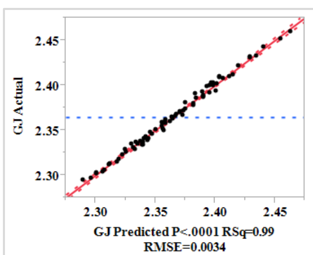
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure C.3 High syrup purity scenario sugar recovery – RSREC plots

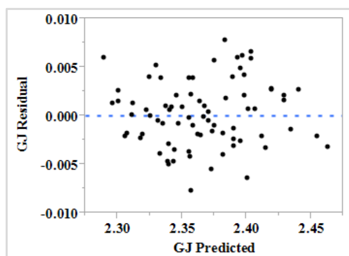
D. Heating Requirements (gigajoule/metric ton syrup solids) – GJ Model Summary

Table D.1 Parameters estimates and summary of model fit evaluation for heating requirements – GJ response at different syrup purity scenarios

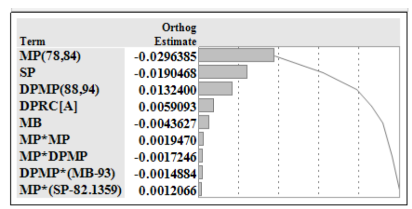
Parameter Estimates	**Low Syrup Purity**			**Medium Syrup Purity**			**High Syrup Purity**		
Term	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**
Intercept	4.5622	93.75	<.0001	3.7722	39.44	<.0001	4.5523	34.16	<.0001
MP(78,84)	-0.0383	-83.88	<.0001	-0.0338	-41.53	<.0001	-0.0299	-29.40	<.0001
SP	-0.0201	-50.34	<.0001	-0.0115	-13.98	<.0001	-0.0217	-20.32	<.0001
DPMP(88,94)	0.0165	41.53	<.0001	0.0177	22.56	<.0001	0.0159	15.33	<.0001
DPRC[A]	0.0057	17.23	<.0001	0.0064	9.97	<.0001	0.0064	7.17	<.0001
MB	-0.0059	-12.87	<.0001	-0.0052	-6.04	<.0001	-0.0040	-3.96	0.0002
MP*MP	0.0039	5.70	<.0001	0.0045	3.42	0.0012	0.0052	2.43	0.0172
MP*(SP-SP*)	0.0023	4.29	<.0001	0.0036	3.33	0.0015	n/a	n/a	n/a
MP*DPMP	-0.0030	-5.86	<.0001	-0.0046	-4.68	<.0001	-0.0025	-2.24	0.0276
DPMP*(MB-MB*)	-0.0023	-4.50	<.0001	n/a	n/a	n/a	n/a	n/a	n/a
Summary of Fit									
R-Square	0.993			0.978			0.955		
Adjusted R-Square	0.993			0.975			0.951		
Root Mean Square - RMS	0.003			0.005			0.008		
Mean - RSWC	2.364			2.307			2.274		
Observations	103			66			88		
Prediction Error – (Press)	0.004			0.006			0.009		
SP*	82.1			85.0			n/a		
MB*	93.0			n/a			n/a		



a) Prediction Plot

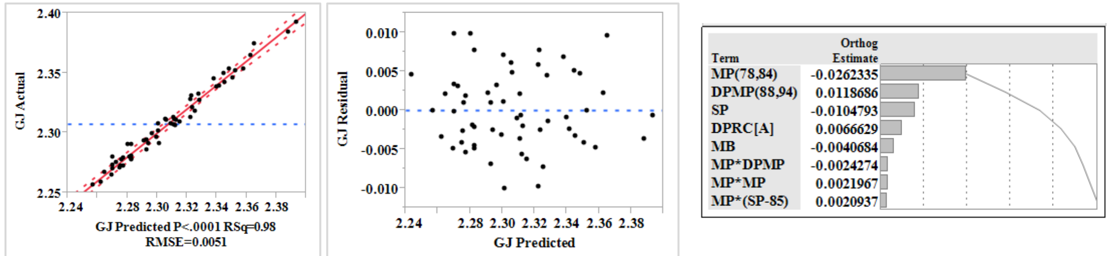


b) Residuals Plot



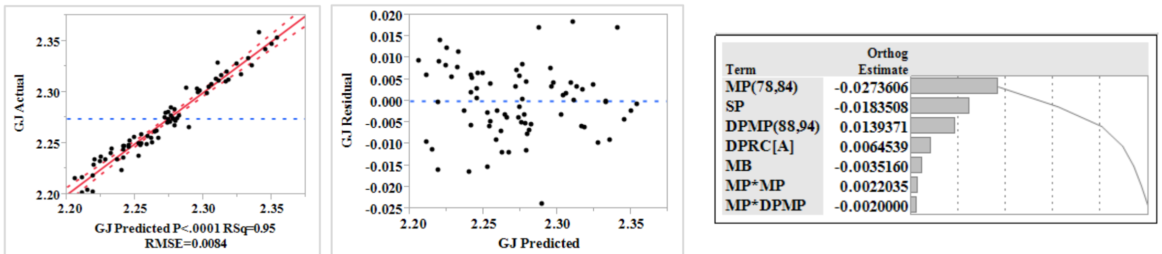
c) Pareto plot of transformed estimates

Figure D.1 Low syrup purity scenario heating requirements – GJ plots



a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure D.2 Medium syrup purity scenario heating requirements – GJ plots



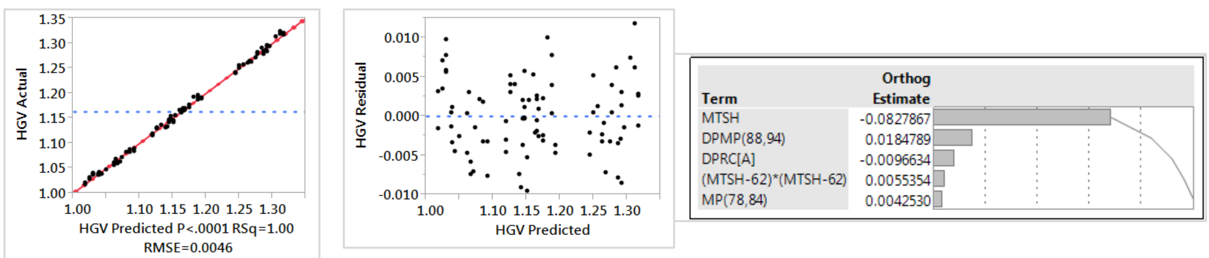
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure D.3 High syrup purity scenario heating requirements – GJ plots

E. High Grade Volume Ratio (Installed/Required Capacity) – HGV Model Summary

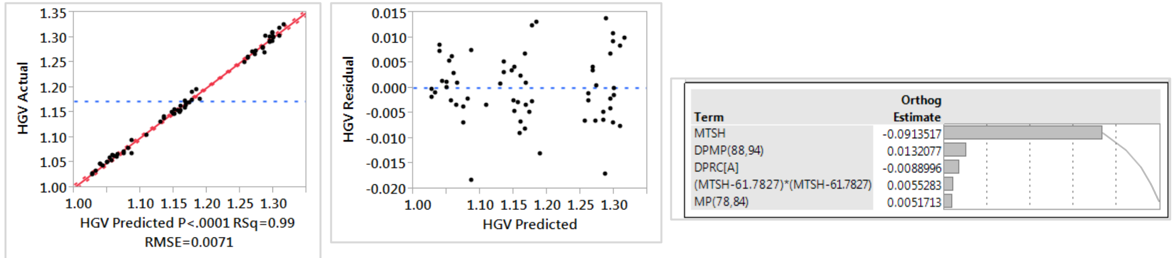
Table E.1 Parameters estimates and summary of model fit evaluation for heating requirements – HGV response at different syrup purity scenarios

Parameter Estimates	**Low Syrup Purity**			**Medium Syrup Purity**			**High Syrup Purity**		
Term	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**
Intercept	2.3220	355.55	<.0001	2.3503	204.76	<.0001	2.2984	248.49	<.0001
MTSH	-0.0188	-179.30	<.0001	-0.0192	-103.30	<.0001	-0.0185	-125.20	<.0001
DPMP(88,94)	0.0213	38.93	<.0001	0.0154	14.61	<.0001	0.0112	12.50	<.0001
DPRC[A]	-0.0094	-20.60	<.0001	-0.0085	-9.62	<.0001	-0.0080	-10.39	<.0001
MP(78,84)	0.0060	9.67	<.0001	0.0057	5.04	<.0001	0.0059	6.68	<.0001
(MTSH-MTSH*)^2	0.0003	12.18	<.0001	0.0003	6.35	<.0001	0.0002	4.32	<.0001
Summary of Fit									
R-Square	0.997			0.995			0.995		
Adjusted R-Square	0.997			0.994			0.995		
Root Mean Square - RMS	0.005			0.007			0.007		
Mean - RSWC	1.163			1.172			1.157		
Observations	103			66			88		
Prediction Error – (Press)	0.005			0.007			0.008		
MTSH*	62.0			61.8			61.9		



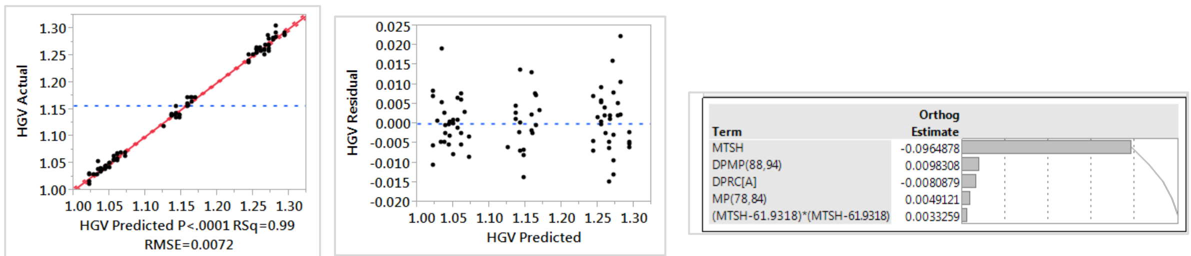
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure E.1 Low syrup purity scenario heating requirements – HGV plots



a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure E.2 Medium syrup purity scenario heating requirements – HGV plots



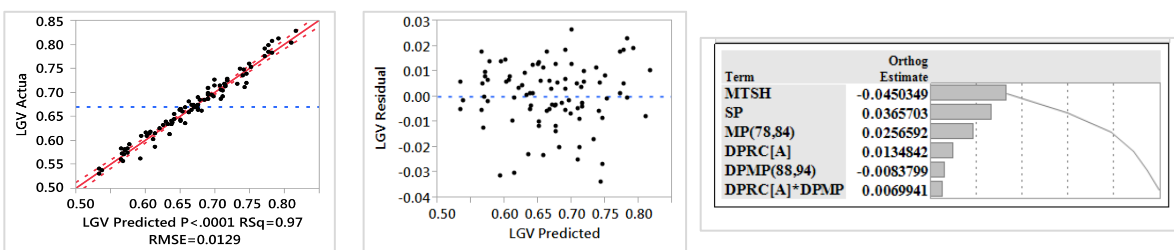
a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure E.3 High syrup purity scenario heating requirements – HGV plots

F. Low Grade Volume Ratio (Installed/Required Capacity) – LGV Model Summary

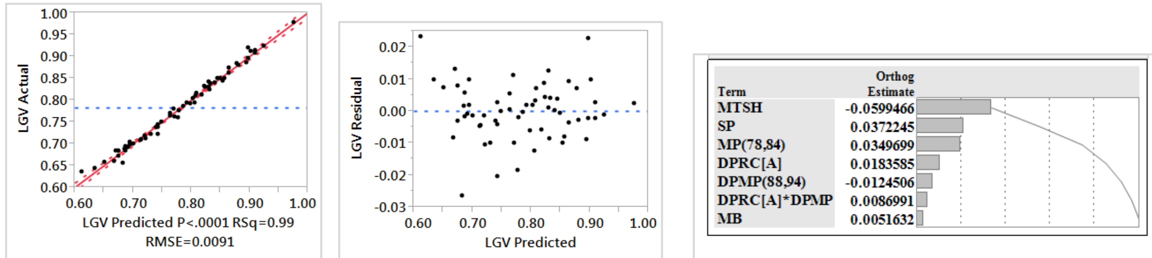
Table F.1 Parameters estimates and summary of model fit evaluation for heating requirements – HGV response at different syrup purity scenarios

Parameter Estimates	Low Syrup Purity			Medium Syrup Purity			High Syrup Purity		
Term	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**	**Estimate**	**t Ratio**	**Prob>|t|**
Intercept	-1.8868	-15.30	<.0001	-3.5553	-21.04	<.0001	-5.5002	-33.73	<.0001
MTSH	-0.0107	-36.52	<.0001	-0.0125	-51.70	<.0001	-0.0159	-54.11	<.0001
SP	0.0392	26.20	<.0001	0.0520	34.79	<.0001	0.0848	46.02	<.0001
MP(78,84)	0.0366	21.06	<.0001	0.0473	32.33	<.0001	0.0578	32.74	<.0001
DPRC[A]	0.0127	9.92	<.0001	0.0176	15.40	<.0001	0.0244	15.92	<.0001
DPMP(88,94)	-0.0106	-6.92	<.0001	-0.0143	-10.16	<.0001	-0.0163	-9.14	<.0001
DPRC[A]*DPMP	0.0084	5.51	<.0001	0.0107	7.74	<.0001	0.0154	8.68	<.0001
MB	n/a	n/a	n/a	0.0074	4.87	<.0001	n/a	n/a	n/a
(MTSH-MTSH*)*(SP-SP*)							-0.0018	-5.04	<.0001
Summary of Fit									
R-Square	0.965			0.989			0.989		
Adjusted R-Square	0.963			0.988			0.988		
Root Mean Square - RMS	0.013			0.009			0.014		
Mean - RSWC	0.669			0.782			0.978		
Observations	103			66			88		
Prediction Error (Press)	0.013			0.010			0.015		
SP*	n/a			n/a			88.0		
MTSH*	n/a			n/a			61.9		



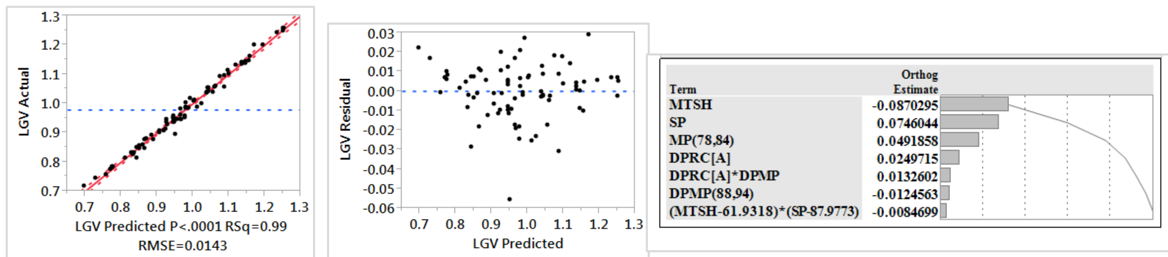
a) Prediction Plot | b) Residuals Plot | c) Pareto plot of transformed estimates

Figure F.1 Low syrup purity scenario heating requirements – LGV plots



a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure F.2 Medium syrup purity scenario heating requirements – LGV plots



a) Prediction Plot b) Residuals Plot c) Pareto plot of transformed estimates

Figure F.3 High syrup purity scenario heating requirements – LGV plots

G. Glossary of Sugar Industry Terms

Affination: sugar manufacturing stage that involves magma preparation and centrifugation to increase the purity of a sugar crystal by a further removal of the molasses layer attached to the surface of the crystal

Affined sugar: Sugar produced following an affination procedure

Back-boiling: Recycle of molasses that were separated from sugar crystals by centrifugation to a prior crystallization stage (strike) to adjust the purity of the massecuite (e.g. A molasses returned to feed the first strike).

Boiling house: Section of the raw sugar factory devoted to produce sugar crystals mainly by evaporative crystallization (boiling) under vacuum and separation of sugar crystals by centrifugation.

Brix: term equivalent to the mass percentage (%) of the total soluble solids. It is also referred as RDS (refractometric dry substance). The True or total solids (dry substance) are the actual content of solids in a sample determined by drying in a vacuum oven.

Clarification: Treatment applied to a sugar cane juice to precipitate and separate, by sedimentation/flotation and filtration, non-sucrose compounds to improve purity, color and turbidity of the final clarified juice.

Color/non-sucrose ratio: Color measurement referred to the total amount of non-sucrose compounds in a sugar product solution.

Color: Color measurement (absorbancy index) referred to the total amount of soluble solids in a sugar product solution (cm^2/kg dry substance).

- Whole color: color in solution for a raw sugar sample as it is delivered to the sugar refinery
- Affined color: color in solution on raw sugar after an affination procedure (affined sugar)

Conductivity Ash: Determination by electrical conductivity of inorganic compounds (cations and anions) present in a sample solution.

Crystal content: Mass percentage of crystals in a massecuite/magma based on the total solids or on the mass.

Crystallization scheme (boiling scheme or boiling system): Number and arrangement of crystallization stages to produce determined sugar quality. The crystallization stages are identified by letters or numbers (A, B, C… or first, second, third…)

Crystallizer: A vessel for crystallization

Cut-crush time: length of time between the moments of cutting the cane to the time the juice is extracted from the cane at the mill.

Cycle time: For evaporative crystallization in batch pans, it is the length of time that starts when the vacuum pan begins to be loaded and ends at the next batch starting point.

Double purge: Addition of a second centrifugation unit to separate (purge) the molasses from the crystals

Dry substance: mass of soluble solids remaining after the evaporation of the water of a sugar solution and it is determined by an oven drying method. It is denominated also as total solids by drying of dry solids.

Footing: Feeding a pan with seed (magma/massecuite) to start the boiling or crystallization

Grain Strike: Intermediate crystallization stage used to produce seed (magma/massecuite) which starts the last crystallization stage

ICUMSA: is the International Commission for Uniform Methods of Sugar Analysis which standardizes the analytical procedures for the sugar industry

Impurity: Term that groups all soluble solids compounds different from sucrose. It is the non-sucrose compounds percent soluble solids (Impurity = 100 – Purity)

Inverts (reducing sugars): The mixture of glucose and fructose produced by the hydrolysis of sucrose

Magma: Process product produced by the mingle of sugar crystals and a liquid (water, juice, syrup or molasses)

Massecuites: Process product from the end of a crystallization stage (strike) consisting of a mixture of sugar crystals surrounded by the mother liquor. For three crystallization stages (strikes), high grade massecuites are produced by the first two strikes and they are called A and B massecuites. Low grade massecuites are produced by the last strike and they are called C massecuites

Molasses: Liquid process product produced by centrifugal separation and washing of the sugar crystals that were grown by crystallization (strike). For three crystallization stages, the molasses are called A and B for the two first stages and 'final' for the last stage

Mother liquor: Syrup or liquor in which sugar crystals grow. It is the liquid part in a massecuite.

NAFTA: North American Free Trade Agreement between United States, Canada and Mexico

Non-sucrose: water soluble solid compound different from sucrose

Pan or vacuum pan: especial vessel used to evaporate (under vacuum) the water present in a sugar solution growing sugar crystals (crystallization) driven by the supersaturation of the solution

Polarization (Pol): Approximate measure of the mass percentage of sucrose in a sugar solution by the optical rotation of polarized light. The approximation to the true percent of sucrose in solution is lower as the percentages of non-sucrose compounds are higher. The measure is called apparent sucrose content. A more accurate measure of the true sucrose content has to be done applying high performance liquid chromatographic procedures (HPLC).

Purity: The term refers to the sucrose percent soluble solids in a sugar or sugar process sample. Depending on the analytical procedure the purity is called:

- Apparent purity: Pol % RDS (°Brix)
- True Purity: HPLC Sucrose % Dry Substance

Refining: It is the sugar cane manufacturing stage that produces white sugar through the purification of raw sugar applying several procedures, including clarification, filtration, decolorization and re-crystallization.

Refractometric dry substance (RDS): Mass percent of total soluble solids in a sugar solution measured by the refractive index of the solution

Remelt: Syrup prepared by dissolving low grade sugars (C-sugar). The remelt is returned to the first crystallization stage

Seed: Slurry generally prepared by blending fine sugar crystals with isopropanol. The fine sugar crystals are used as nuclei to grow sugar crystal to the final size (crystallization). Seeding is the introduction of the fine crystals slurry to start the crystallization. The term seed is also applied to the magma/massecuite used to start the crystallization process for any crystallization stage (A, B, C)

Strike: Massecuite at the end of an evaporative crystallization stage which is discharged from the pan.

Sucrose: Sweet crystalline organic compound with molecular formula $C_{12}H_{22}O_{11}$

Sugar: In the sugar industry the term sugar is applied to the crystal products which are largely sucrose. The production of white sugar from sugarcane is effected on two stages: raw sugar production (not for human consumption) and sugar refining. At the market worldwide, the common types and standards for raw sugar are:

- HP sugar: high pol raw sugar (98.0 ≤ Pol ≥ 99.3 °Z)
- HP sugar: high pol raw sugar (98.0 ≤ Pol ≥ 99.3 °Z)
- VHP sugar: very high pol raw sugar (Pol > 99.3 °Z)
- V-VHP sugar: very, very high pol raw sugar (Pol > 99.6 °Z)

Sugarcane (cane): Tropical and subtropical perennial grass mainly from the *Saccharum officinarum* species which is grown and harvested for sugar (sucrose) production. The sucrose is mainly located in the parenchyma cells of the sugarcane stalk.

Sugarcane trash: sugarcane leaves, tops, dead stalks and other vegetable matter which are delivered to the mill.

Supersaturation: Degree to which the concentration of sucrose in a solution is higher than the concentration of sucrose at saturation at the same temperature conditions

Syrup: Concentrated juice (~ 60 – 70 °Brix) delivered from the evaporators to the boiling house

Target Purity (TP) or equilibrium purity: is an empirical formula that estimates the lower purity of final molasses reached at standard conditions in a laboratory. The target purity formula allows for the effect of non-sucrose compounds, mainly invert sugars and ash, in the exhaustion of final molasses.

Majority of the definition terms have been adapted from Rein (2007) and van der Poel, Schiweck et al. (1998)

VITA

Luz Stella Polanco was born in Cali, Colombia. Luz Stella has a bachelor's degree in Chemical Engineering from Universidad del Valle at Cali, Colombia, and worked for eight years at a Colombian sugarcane factory as a Chief Chemist. Due of her experience in the sugar industry, in 2003 she was hired as a research associate by the Audubon Sugar Institute (LSUAgCenter).

In August 2005 she began her studies as a part-time student and in August 2009 she received a M.S. in Chemical Engineering at the Louisiana State University. Continuing her studies, in 2010 she began the PhD in Engineering Science concentrated in Chemical Engineering and in Biological and Agricultural Engineering. Since the beginning of sugar industry and research career, her research has been focused on statistics, quality control, chemistry, energy, modeling, integration and optimization of the sugar manufacturing process.

e-mail: luzstellapol@yahoo.com