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Assessment of target purity difference for a Louisiana sugar mill

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ASSESSMENT OF TARGET PURITY DIFFERENCE FOR A LOUISIANA SUGAR MILL

A Thesis
Submitted to the Graduate Faculty of the
Louisiana State University and
Engineering College
in partial fulfillment of the
requirements for the degree of
Master of Science in Chemical Engineering

In

The Department of Chemical Engineering

by
Luz Stella Polanco
B.S., Universidad del Valle, Cali-Colombia, 1989
August 2009
In memory of my loving mother:

Ana Maria Duque

April 27, 2007
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The main goals of a “raw” sugarcane factory are to have an efficient profitable operation with the required sugar quality and maximum sugar recovery. The loss of sugar to final molasses accounts for 45 to 47% of the total sugar losses. An estimation of the average cost of the manufacturing losses for the 2007 harvest season was approximately $9 million – the sugar lost to the final molasses accounts for approximately 50% of this cost (Salassi, 2008).

For final molasses, the “Target Purity” (or equilibrium purity) refers to the minimum level of sucrose that theoretically remains in solution for a fixed composition of non-sucrose substances. The “Target Purity Difference” (TPD), which is the difference between the actual “True Purity” and the “Target Purity,” is the non-bias measurement of factory performance. TPD was used for this research to define the operational goals of the low-grade station, toward reaching the physical maximum molasses exhaustion.

The approach to molasses exhaustion for the studied factory was focused on the low-grade station stages of the boiling house which include: 1) continuous cooling crystallizer’s performance; 2) molasses consistency; 3) supersaturation; 4) crystal size distribution and crystal content; and 5) purity drop and goals by stage.

The main recommendations are: 1) to increase the crystal content in the “C” massecuite at the exit of the continuous vacuum pan (CVP) by regulating the seed/massecuite ratio and Brix profile; and 2) to improve the flow pattern and cooling temperature control in the vertical crystallizers by modifying the internal arrangement (baffles and cooling coils arrangement) and placing more temperature probes. Complementary recommendations are: 1) implement procedures for seed preparation (slurry preparation by ball milling); 2) increase grain strike capacity according to the expected “C” massecuite rate; 3) grain batch pan automation; and 3) monitor and control crystal size distribution per stage according to standard desired values.

The establishment of a routine to measure purity drop/rise and crystal size distribution specifying achievable goals per stage at the low-grade station is the best tool to achieve the ultimate goal (zero target purity difference).
1. INTRODUCTION

1.1. Sugarcane Process

Raw sugar production from sugarcane uses several unit processes that generally begin with the extraction of the juice (milling or diffusion) followed by heating and clarification (flocculation and settling of suspended solids); then, water evaporation (concentration of dissolved solids); and finally the crystallization process (boiling house).

![Block diagram for the sugar cane process](image)

**Figure 1.1.** Block diagram for the sugar cane process

The boiling house consists of a series of crystallization stages where sugar purification goes in one direction and molasses exhaustion goes in the other direction. The boiling scheme (number of stages and streams distribution) depends on the purity of the syrup (cane juice clarified and concentrated) and the desired sugar quality. The most common boiling schemes are Rein (2007):

- Two-boiling
- Three-boiling
- Double-Einwurf
- VHP (“very high pol” sugar)

Each crystallization stage has to recover the maximum amount of sugar from the feed by conducting the crystallization to the point where the massecuite (mixture of crystals and mother liquor) still has flowability. Then, the massecuite is taken to centrifuges to separate the crystals from the mother liquor (molasses). If the mother liquor (molasses) has recoverable sucrose, it is taken to the following crystallization stage. When the sucrose content in the mother liquor is too low to be recovered, the mother liquor (final molasses) is stored and sold. Depending on the scheme, the raw sugar is produced by the first stage or by the two first stages.
1.2. Sugar Cane Industry at United States

Table 1.1 shows some production numbers for the US sugar cane industry from 1999 to 2005. The most important aspects of this information are that the cost to produce one pound of sugar is very close to the US sugar price; and the high variability of the sugar yield (10.44 to 12.95). The cost to produce sugar in the US is as high as twice the lowest cost in the world, and it is most of the time above the world’s average weighted cost (Stephen and Mir, 2007). One of the ways to reduce the cost is increasing the sugar yield; therefore, it is necessary to reduce the sugar that is lost with the different by-products such as bagasse, filter cake and final molasses.

Table 1.1. US sugar cane industry production, 1999–2005 (Stephen and Mir, 2007)

<table>
<thead>
<tr>
<th>States</th>
<th>Factories</th>
<th>Milling Capacity</th>
<th>Yearly Sugar Production</th>
<th>Sugar Yield</th>
<th>Sugar Costs cents/lb</th>
<th>Sugar Prices cents/lb</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>TCD *1000 tons raw value</td>
<td>Sugar%Cane</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Louisiana</td>
<td>13</td>
<td>164,630</td>
<td>1,413.2</td>
<td>11.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Florida</td>
<td>5</td>
<td>108,800</td>
<td>2,002.5</td>
<td>12.71</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Texas</td>
<td>1</td>
<td>10,000</td>
<td>180.8</td>
<td>10.44</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hawaii</td>
<td>2</td>
<td>10,500</td>
<td>259.4</td>
<td>12.95</td>
<td></td>
<td></td>
</tr>
<tr>
<td>US</td>
<td>21</td>
<td>293,930</td>
<td>3,855.9</td>
<td>12.06</td>
<td>12.55-20.08</td>
<td>18.40-21.76</td>
</tr>
</tbody>
</table>

For the sugar industry (sugar cane or beet) worldwide, final molasses exhaustion is one of the most important issues related to sugar recovery. The main goal of a factory that only produces sugar is to separate the sucrose which is in solution, to form crystals. The sucrose that stays in solution at the end of the process is a financial loss. For the sugar cane industry, the amount of sugar lost in final molasses is approximately 6 to 10% of the sugar that enters the factory with the cane. From a factory point of view, final molasses is defined as a by-product of the sugar process from which the sucrose left in solution can be neither physically nor economically recovered.

1.3. Target Purity Difference

The chemical process used for sugar production is based on crystallization from solution, where the sucrose solubility is the most important condition. To crystallize sugar from a solution, the equilibrium solubility (saturation) has to be exceeded (supersaturation). The supersaturation is the driving force of the crystallization process.
The equilibrium solubility (saturation) for an impure sucrose solution is not the same as that for a pure sucrose solution. On the other hand, type and composition of the impurities in solution not only affects the equilibrium solubility but also affects the crystallization process. The type and composition of the impurities may increase or decrease viscosity (diffusion mechanism) and/or change the crystal habit (sucrose attachment to a crystal site).

Decloux (2000) presented a very comprehensive literature review about molasses exhaustion for the sugar cane and the sugar beet industries. This document states that the type and composition of the non-sucrose substances in the sugar beet raw juice are different from that in the sugar cane raw juice; therefore, their influences in the crystallization process are different and the approach to the minimum level of sucrose in the final molasses is also different. While the beet industry uses the “polish test” (exhaustion test) the sugar cane industry uses “Target Purity” (or equilibrium purity).

The “Target Purity” is the minimum amount of sucrose in solution as a % of the total amount of dissolved solids, which can be physically achieved under specified controlled conditions. Several target purity formulas have been proposed in the literature, and most of them correlate the molasses “equilibrium” purity with the ratio between the reducing sugars (glucose and fructose) and the total amount of ash (Rein, 2007). The differences can be attributed to different analytical methods and to different exhaustion test conditions (cooling temperature and molasses viscosity). Large differences on non-sucrose composition can also affect this correlation.

The Target Purity formula (equation 1) used by Audubon Sugar Institute was determined in South Africa (Rein and Smith, 1981) at the laboratory level while controlling cooling temperature (40 °C), stirring (12 rpm), retention time (48 hours) and viscosity (above 300 Pa-s). The formula recognizes the factory equipment limitation for handling high viscosities and that there is no further improvement in sucrose recovery for viscosities of mother liquor above 300 Pa•s at 40°C (104°F) Rein (2007).

\[
\text{“Target Purity” } = \frac{P_{\text{Target}}}{G + F} = 33.9 - 13.4 \cdot \log \left( \frac{G + F}{\text{Ash}} \right) \tag{1}
\]

\( G + F \) = Glucose plus Fructose (reducing sugars, RS) concentration by HPLC

\( \text{Ash} \) = Conductivity Ash (A)

Rein (2007) mentions several important reasons that justify the broad applicability of the Target Purity formula; in addition, Saska (1999) tested its validity for Louisiana final molasses.
**Target Purity Difference (TPD)** is the difference between the actual **True Purity** and the **Target Purity** for a particular final molasses. The final molasses TPD can be used to compare the actual factory performance with other factories, showing the opportunity for improvement with a correspondent financial gain. Applying the same Target Purity formula used in South Africa to Louisiana, a TPD=0 is not common, rather, average factories have a TPD between 4 and 5 and the best factories have a TPD between 2 and 3 (Rein, 2007). An analytical survey for 15 sugar cane producing countries showed that the most frequent TPDs were between 3 and 7 (Sahadeo and Lionnet, 1999).

A limitation of TPD is not taking into account what happened before the final molasses formation. Nevertheless, Target Purity Difference is a very good tool to evaluate the performance of the “low grade station” and also to evaluate the performance of the whole boiling house (non-sucrose changes on this stage are lower than the ones that have been produced downstream).

### 1.4. A Louisiana Sugar Mill – M. A. Patout & Son, Ltd. – “Enterprise Factory”

M. A. Patout & Son, Ltd. – “Enterprise Factory” was founded in 1825 (the oldest sugar factory in United States), and it is located in Patoutville, Louisiana, approximately 6 miles southwest of Jeanerette, LA (M. A. Patout & Son, Ltd., 2009).

![Figure 1.2. “Enterprise Factory” –M. A. Patout & Son, Ltd. (2009)](image)

4
“Enterprise Factory” has a milling capacity of 22,000 tons of cane per day (TCD), from which 10,000 tons of cane are processed in a diffuser. Figure 1.3 shows a block diagram which approximately describes the stages and streams distribution for the double-Einwurf boiling scheme used at Enterprise.

![Figure 1.3. Double-Einwurf boiling scheme](image)

In 2008, Enterprise expanded the factory capacity in the back end of the boiling process – low-grade station, Figure 1.4. The expansion is intended to reduce the molasses target purity difference and to increase sugar recovery.

The capacity expansion consisted in the installation of:

- two (2) new vertical crystallizers of 227 m$^3$ (8,000ft$^3$) each, for a total capacity of 878 m$^3$ (31,000ft$^3$);
- a new vertical reheater (finned tubes) - heat transfer area of 1,208 m$^2$ (13,000ft$^2$) and capacity of 23 m$^3$ (825ft$^3$); and
- two (2) additional continuous centrifuges for a total of 7 machines (centrifuge screens with 0.04 x 2.18 mm of slot size and 9.6% of open area).
1.5. Thesis Objectives and Overview

The purpose of this thesis was to assess the molasses exhaustion at the low-grade station in Enterprise factory to obtain “zero” target purity difference. Following recommendations given by trusted sources in the literature, specific goals for each stage were determined and monitored on weekly basis. Special attention was given to cooling crystallizers’ performance, crystal size distribution, molasses consistency and supersaturation.
The sugar boiling process chapter gives the fundamental theory of sugar crystallization – supersaturation as a driving force, variables that affect the crystal growth, and how the new nuclei are formed; then, it gives the crystallization techniques that are applied at the boiling house in a sugar cane factory; and finally, the technical recommendations that the factory could implement to achieve a good performance respect to sugar recovery and molasses exhaustion. The materials and methods chapter describes the procedures and sampling frequency used to collect the data.

The results and discussion chapter first shows the historical (6 years) and actual (2008 season) variation of parameters related to molasses exhaustion and sugar recovery. Then, the final molasses exhaustion is approached by focusing in the low-grade station stages (vacuum pan, cooling crystallizers, reheater, and centrifuges) and the main variables that affect the final molasses exhaustion process, such as: vertical crystallizers retention time; molasses – massecuite consistency (viscosity); crystal content, surface area, and size distribution; supersaturation; and the average stage performance. Next, the proposed goals were compared with the actual values and the economical return was estimated.

Conclusions and recommendations discuss the findings of this evaluation and what are the actions that may help this factory and what actions can be applied to other sugar cane factories.
2. THE SUGAR BOILING PROCESS

2.1. Crystallization Theory

_Crystallization_ is the process of formation of solid crystals precipitating from a solution or melt. Crystallization is also a chemical solid-liquid separation technique, in which mass transfer of a solute from the liquid solution to a pure solid crystalline phase occurs. The crystallization process requires **nucleation** and **crystal growth**. Nucleation is the step where the solute molecules dispersed in the solvent start to gather into clusters, on the nanometer scale (elevating solute concentration in a small region), that become stable under the operating conditions. These stable clusters constitute the nuclei. The clusters must reach a critical size in order to become stable nuclei, and this is dictated by the operating conditions (temperature, supersaturation, etc.). It is at nucleation that the atoms arrange in a defined and periodic manner that defines the crystal structure. The **crystal growth** is the subsequent growth of the nuclei (Wikipedia, 2009).

The driving force for the sucrose crystallization is supersaturation; the growth of the crystal depends on several factors such as temperature, crystal surface area, fluid dynamic conditions and the nature and concentration of the impurities (Vaccari et al., 2003). The solubility of a solute in a solvent at determined conditions, such as pressure, temperature and the presence of other substances is the maximum amount of this solute that can be dissolved and stay in solution (saturation); the solute concentration above the saturation concentration is the supersaturation. Depending upon the conditions, either nucleation or growth may predominate, and as a result, crystals with different sizes and shapes are obtained. Once supersaturation is exhausted, the solid-liquid system reaches equilibrium and the crystallization is complete (van der Poel et al., 1998).

2.1.1. Solubility and Supersaturation

The solubility of sucrose in water is very high (2 parts of sucrose for 1 part of water at saturation at 25°C and increases very sharply at higher temperatures); hence, water is considered one of the most melassigenic (molasses producer) non-sucrose substances in the crystallization process. In order to crystallize sucrose, the sucrose concentration must be increased to a concentration above the saturation conditions. Control of this process requires knowledge of the sucrose concentration at saturation for the working conditions (Rein, 2007).
The solubility of sucrose for a range of temperatures from 0 to 90 °C in pure sucrose solution is well represented by the Charles (1960) equation (Rein, 2007):

\[
w_{S,Sat} = 64.397 + 0.07251 * t + 0.002057 * t^2 − 9.035 * 10^{-6} * t^3
\]  \hspace{1cm} (2.1)

\(w_{S,Sat}\): g sucrose/100g solution at saturation t;
\(t\): temperature in °C.

\[Sat\, Coefficient = q_{Sat, p} = \left( \frac{w_{S}}{w_{W}} \right)_{Sat, p} = \frac{w_{S, Sat}}{100 - w_{S, Sat}}\]  \hspace{1cm} (2.2)

\(q_{S, Sat}\): g sucrose/g water at saturation in pure solution.

The saturation coefficient is used only for pure sucrose solution; the impurities in the sugarcane juice alter the sucrose solubility.

The solubility coefficient (SC) is used to express the ratio of sucrose concentration at saturation between the impure and the pure sucrose solutions:

\[Solubility\, Coefficient = SC = \frac{q_{Sat, i}}{q_{Sat, p}}\]  \hspace{1cm} (2.3)

\(q_{Sat, i}\): g sucrose/g water at saturation in impure solution

The supersaturation coefficient (y) is the degree of supersaturation obtained at determined conditions of temperature and purity and is determined by the ratio of the sucrose/water ratio at supersaturation and the sucrose/water ratio at saturation.

\[Supersaturation\, Coefficient = y = \frac{q_{Supersat}}{q_{Sat}}\]  \hspace{1cm} (2.4)
Figure 2.1 shows a pure sucrose solubility chart with the most important lines (y=1, y=1.2, y=1.3)

- y<1 Under saturated sucrose solution.
- y=1 Saturated sucrose solution.
- y>1 Supersaturated sucrose solution.
- 1.0<y<1.2 Metastable region. Crystal grows without formation of new nuclei.
- 1.2<y<1.3 Intermediate region. Crystal grows and formation of new nuclei.
- y>1.3 Labile region. Spontaneous formation of nuclei (nucleation).

![Figure 2.1. Pure sucrose solubility chart](image)

### 2.1.2. Crystal Growth

Van der Poel et al. (1998) refers to the crystal growth kinetics as a complex subject since it involves phenomena occurring on the surfaces of the different crystal faces and at the crystal-solution interface. There are different theories explaining crystal growth, but the fundamentals of the crystal growth are well described by **Adsorption Layer Theory**, which describes the processes that take place at the crystal surface, and **Diffusion Theory**, which describes the processes that take place at the crystal-solution interface.
Basically, crystal growth is a rate process where the supersaturation is the driving force. The sucrose molecules in solution are transported by diffusion to the crystal and then located and incorporated to the sucrose lattice (Rein, 2007).

![Figure 2.2. Crystal growth representation](image)

**Diffusion:**
\[
\frac{dm}{d\tau} = k_D \times A \times (c - c^*) 
\]  
\hspace{2cm} (2.5)

**Surface reaction:**
\[
\frac{dm}{d\tau} = k_R \times A \times (c^* - c_{eq}) 
\]  
\hspace{2cm} (2.6)

- \(\frac{dm}{d\tau}\): change on crystal mass with time, crystallization rate;
- \(k_D\): diffusion rate constant;
- \(k_R\): surface reaction constant (combined rate constant which involves adsorption, migration to the proper site and attachment to the crystal lattice);
- \(A\): crystal surface area;
- \(c, c^*\ and \ c_{eq}\): bulk solution concentration; crystal/solution interface concentration and equilibrium concentration.
Combining these two equations (2.5 & 2.6):

\[
\frac{dm}{d\tau} = K_G \star A \star (c - c_{eq})
\]  

(2.7)

\(K_G\) is the overall crystal growth constant (overall mass transfer coefficient).

\[
K_G = \frac{k_D \star k_R}{k_D + k_R}
\]

(2.8)

It had been found that this reaction is the first order at high concentration differences (pan conditions) and from order 1 to 2 for low concentration differences and in the presence of non-sucrose components. A more realistic representation of this rate process is given by (Rein, 2007):

\[
\frac{dm}{d\tau} = K_G \star A \star (c - c_{eq})^n
\]

(2.9)

When \(k_D \gg k_R\) then \(K_G \sim k_D\), the driving force for the growth process is diffusion. When \(k_R \gg k_D\) then \(K_G \sim k_R\), the driving force for the growth process is surface reaction. Crystallization conditions such as temperature, stirring or natural circulation and the presence of non-sucrose components affect the values of \(k_D\) and \(k_R\), and hence, affect the value of \(K_G\) (Rein, 2007).

### 2.1.2.1. Temperature and Crystal Growth

The dependence of the crystal growth constant on temperature is represented by the following Arrhenius Equation (Rein, 2007):

\[
K_G = K_{G0} \cdot \exp\left(-\frac{E_A}{RT}\right)
\]

(2.10)

- \(R\): universal gas constant;
- \(T\): absolute temperature; and
- \(E_A\): activation energy.
At low temperature $t<40^\circ C$ ($104^\circ F$), $E_A$ has a high value and surface incorporation is controlling the growth rate process ($k_R \gg k_D$, $K_G \approx k_R$) and at high temperature $t>50^\circ C$ ($122^\circ F$) pan conditions, $E_A$ has a low value and diffusion is controlling the growth rate process ($k_D \gg k_R$, $K_G \approx k_D$) (Rein, 2007).

### 2.1.2.2. Stirring or Circulation and Crystal Growth

The effect of the stirring or circulation on the growth rate constant is related to the thickness of the boundary layer. As the relative crystal solution motion increases, the thickness of the boundary layer decreases, which decreases the resistance for the sucrose diffusion. The boundary layer thickness can be evaluated from the relationship between the following dimensional numbers (van der Poel et al., 1998):

\[
Reynolds = Re = \frac{v \cdot l \cdot \rho}{\eta}
\]

\[
Schmidt = Sc = \frac{\eta}{\rho \cdot D}
\]

\[
Sherwood = Sh = \frac{l}{\delta} = \frac{2}{3} \cdot Re^{1/2} \cdot Sc^{1/2}
\]

Boundary Layer Thickness =

\[
\delta = \frac{3}{2} \cdot l \cdot \left(\frac{v \cdot l \cdot \rho}{\eta}\right)^{-1/2} \cdot \left(\frac{\eta}{\rho \cdot D}\right)^{-1/3}
\]  

(2.11)

- $l$: crystals’ linear dimension
- $\rho$: solution density
- $v$: relative crystal-solution velocity
- $\eta$: dynamic viscosity
- $D$: diffusion coefficient
2.1.2.3. Non-Sucrose Components and Crystal Growth

Non-sucrose components can affect the characteristics of the sucrose solution (such as viscosity and solubility) and/or can interact with the growing faces of the crystals (modifying the crystal morphology). Some of the modifications of the solution characteristics are changes in the supersaturation conditions for crystal growth (solubility increase or decrease depending on the non-sucrose component) and changes in the viscosity of the solution (viscosity increase or decrease which affects the relative crystal-solution velocity, which is related to the thickness of the boundary layer). Some non-sucrose components may also “poison” specific crystal faces affecting the growth rate on these faces, causing morphologic modifications. Non-sucrose components which produce habit modifications and viscosity changes in sucrose crystallization are: oligosaccharides, dextran, glucose and fructose (van der Poel et al., 1998). Other important non-sucrose components are the salts, most of them increasing the sucrose solubility and viscosity but also they can act as catalysts on different reactions such as inversion or color formation (Vercellotti et al., 1996).

The effects of the non-sucrose components are most evident in the final stages of the sucrose crystallization, where the concentrations of the non-sucrose components with respect to sucrose are higher. An equation to represent the effect of the non-sucrose components in crystal growth was developed in Australia by Wright and White (1974), but Love (2002) in South Africa determined a higher value for the constant affecting the non-sucrose/water ratio (2.4 against 1.75), taken from (Rein, 2007).

\[
K_G = K_{G0} \cdot \exp \left( -\frac{E_A}{RT} - 1.75 \cdot q_{NS/W} \right) \quad (2.12)
\]

\( q_{NS/W} \): sucrose/water ratio

2.1.3. Nucleation

Nucleation is the formation of crystal nuclei and it may occur spontaneously or induced by an external stimulus.
2.1.3.1. Primary Nucleation

This term refers to the nucleation which occurs spontaneously in the labile zone (homogeneous), Figure 2.1; or is induced by particles of different species (heterogeneous). Since the formation of crystal nuclei cannot be controlled, primary nucleation promotes the formation of false grain, affecting the crystal size distribution, and has to be avoided (van der Poel et al., 1998).

2.1.3.2. Secondary Nucleation

Mullin (2001) defines secondary nucleation as the nucleation that occurs specifically in the presence of crystals of the same material that is crystallizing. Secondary nucleation occurs in the intermediate zone of the solubility curve. In sugar crystallization secondary nucleation may occur during the seeding point when the smalls seeds are introduced to the pan and may occur in the tightening stage in the pan (evaporative crystallization) and in the crystallizers (cooling crystallization) due to high supersaturation and low crystal content.

2.2. Crystallization Techniques for Raw Sugar Production

In general, crystallization can occur from any phase, solid, liquid or gaseous. In sugar production, crystallization from the solid phase is important for the conditioning and storage of sugar. Crystallization from aqueous solution is the basis for the manufacturing of sugar. Supersaturation is the driving force for the crystallization from solution, and it can be driven either by evaporation or by cooling. During crystallization of a substance from the solution, the concentration of the crystallizing substance in solution decreases. Supersaturation level must be attuned with the crystallization rate to maintain crystallization (van der Poel et al., 1998).

2.2.1. Evaporative Crystallization

Supersaturation in evaporative crystallization is obtained by removing water from the solution by evaporation under vacuum – to avoid high temperatures that may destroy the sucrose, degrade reducing sugars and promote color formation – increasing the dry substance content of the solution above the saturation within the safe metastable zone. Slurry or seed magma is injected into
the supersaturated solution and the seeded crystals are induced to grow. Evaporative crystallization can be conducted in batch or continuous vacuum pans (van der Poel et al., 1998). Figure 2.3.

2.2.1.1. Batch Evaporative Crystallization

Batch crystallization in the sugar industry is conducted in batch vacuum pans (Figure 2.4) and consists of seven steps (van der Poel et al., 1998).
- **Feed:** The feed (syrup or molasses) is drawn into the pan (by vacuum, 0.2 Bar) until the level covers the calandria (required for natural circulation). Some steam is applied to increase the feed temperature and accelerate the process. In the case of a pan with stirrer, it is turned on as soon as it is covered by the feed.

- **Concentration:** Increasing the steam pressure, the water is evaporated in the calandria (flash-evaporation) and the vapor bubbles produce turbulence, which favors the heat transfer to the feed. The level (covering the calandria) is kept constant by the addition of more feed.

- **Seed point:** Concentration ends when a specific **supersaturation** is reached and then crystal seed is drawn into the pan by vacuum. For seeding with slurry (alcoholic suspension of very fine sucrose crystals obtained by wet milling), the region of supersaturation is between 1.1 and 1.2.

- **Crystal formation or graining:** The supersaturation coefficient is kept as constant as possible for the required time (~10 min) to obtain the desired crystal size that can be seen by a naked eye (~50µm). Crystal content at the end of this step is ~10% dry substance. This step is not applicable when the pan is seeded with magma.

- **Crystal growth:** In this phase the steam pressure is increased in order to increase the evaporation of water and the supersaturation is sustained in the metastable zone (y=1.2) by drawing more feed or water into the pan. As the level rises and although the pressure is kept constant (~0.15 Bar), the boiling temperature increases due to the decreasing purity of the mother liquor. For example, depending also on the temperature probe position, the boiling temperature for “A” strike may rise from ~ 69 to 73°C.

- **Tightening:** This phase begins when the maximum working volume of the pan is attained. Concentration continues until the crystal content and viscosity of the material reaches the desired or maximum workable limits. The end of this step is determined by direct dry substance measurement (soluble solids content measured by refractometric index or density – Brix) or by indirect measurement of the consistency (stirrer current).

- **Discharge:** When the maximum allowable consistency is reached, the vacuum is released by injecting air or water vapor (low pressure vapor from the 1st, 2nd or 3rd effect evaporator) to the pan body, and then the discharge valve is opened releasing the pan contents to a receiver. When the pan is empty, the residual crystals adhered to the walls are removed by injecting vapor.
Pan time index (batch time) is used in South Africa to determine pan capacity, which is the total volumetric (m$^3$ or ft$^3$) amount of massecuite divided by the massecuite volumetric flow rate (m$^3$/h or ft$^3$/hr). Depending on the boiling scheme the pan time index is: for “A” massecuite 4.5 hours for VHP scheme and 3.75 hours for three-boiling scheme; for “B” massecuite 6.0 hours for VHP scheme and 4.5 h for three-boiling scheme; for “C” massecuite 9.0 hours for both schemes (Rein, 2007).

2.2.1.2. Continuous Evaporative Crystallization

There are several different types of commercially utilized continuous pans – horizontal multiple compartment pans, true plug flow type pans, multiple batch pan type – and they each may have different control philosophies (direct or predictive control). The start of a continuous evaporative crystallization in a horizontal multiple cell pan, with good boiling and plug flow, which is fed with seed magma or grain to the first cell, is equivalent to the 3rd step of the batch crystallization. The seed flow is approximately 22 to 33% of the final massecuite flow (Rein, 2007).

In a 12 cell pan, the first two cells are used to condition the seed, and as in the batch pan the crystal content in this stage is just a little lower than the required final crystal content, in order to avoid false grain formation. From cell 3 to 9, the seeded crystals grow gradually increasing the crystal content; rapid changes may induce false grain formation. The crystal growth is controlled keeping supersaturation constant, while the massecuite rate production is adjusted by water evaporation with the increment or reduction of the steam pressure according to factory load.
requirements. Tightening is conducted through cells 10 and 11. This last stage is very important to achieve the target molasses exhaustion, since the higher the Brix the lower the molasses purity (higher crystal content) (Rein, 2007).

Sugar deposition rate is high when the Brix (dry substance) of the massecuite ranges from 91 to 92.5; at higher massecuite Brix, the rate slows. Therefore, in order to obtain an optimum molasses exhaustion, a final high Brix is not the only required condition, but a low tightening rate is also important (Thelwall, 2002).

In continuous vacuum pans with direct control, the Brix of the massecuite is controlled at a fixed value using individual probes on each cell. The massecuite throughput is controlled by the variation of the steam pressure in the calandria, and the feed of seed is a function of the seed/final massecuite crystal size ratio (Thelwall, 2002).

The crystal residence time in the continuous vacuum pan is longer than the nominal retention time (pan volume/massecuite volumetric flow). The ratio between the actual and the nominal residence time in a continuous vacuum pan is approximately between 1.4 and 1.8. Nominal residence time in continuous vacuum pan for “A” strikes varies between 2.5 and 3 hours, for “B” strikes between 3.5 and 4 hours and for “C” strikes between 6 and 6.5 hours (Rein, 2007).

**Figure 2.6.** Growth profile for a horizontal multi-cell continuous vacuum pan. (Thelwall, 2002)

**Figure 2.7.** Horizontal continuous vacuum pan (Fletcher Smith, 2001)
2.2.2. Cooling Crystallization

Cooling crystallization takes place when the temperature decreases such that the sucrose solubility is reduced. In this process, the supersaturation condition for sugar crystallization is increased or kept constant by the decrease of temperature produced by the heat transfer to the surroundings (natural cooling) or by the heat transfer to specifically designed heat transfer surfaces. The temperature drop is controlled by the flow and temperature of the cooling water. Ideally, the rate of cooling has to be proportional to the rate of sugar deposition in order to maintain a constant supersaturation and to avoid spontaneous nucleation (false grain formation), Figure 2.8. Good results are obtained when the massecuite is cooled at a rapid rate to the minimum temperature and then held at this temperature for the remainder of the time. Generally, cooling rates are between 1.5 and 2°C/hr (2 and 3°F/hr) (Meade and Chen, 1977)

![Sucrose Solubility Graph](image)

**Figure 2.8.** Cooling crystallization at constant supersaturation

Cooling crystallization has its most important application in the final stage of the sugar process, to maximize the sugar recovery from the final molasses (molasses exhaustion). During the final stage of the evaporative crystallization (tightening), the dry substance content of the massecuite is increased to a maximum value (~97 Brix), which is limited by the flowability of the massecuite (viscosity). At this point the sugar deposition rate is very low since massecuite viscosity is limiting the diffusion of sucrose, and is limiting the heat transfer in the vacuum pan. Massecuite viscosity is the limiting factor for molasses exhaustion in the sugar cane. Reduction of viscosity by the addition of water or diluted molasses in the massecuite receiver before the crystallizers is not recommended.
because of the dilution of the crystals, facilitated by the high temperature of the massecuite; instead, addition of final molasses at saturation temperature is a better option (Saska, 1990).

Low-grade massecuites are sent from the massecuite receivers to the crystallizers where temperatures oscillate between 65 and 70°C (149–158°F) with a supersaturation close to 1.2. The minimum temperatures that can be reached in the crystallizers vary depending on the workability of the massecuite. High density massecuites can be cooled to temperatures between 45 and 50°C (113–122°F), while low density massecuites are cooled to an approximate temperature of 35°C (95°F). The rate of cooling depends on the flow characteristics of the massecuite, nature and concentration of the impurities and the design and mechanical strength of the crystallizer. For low grade massecuites it is not possible to cool fast enough to produce false grain formation, but spontaneous grain formation may occur during the tightening stage in the pan and the small crystals will grow to a visible size during the cooling crystallization (Rein, 2007).

A significant purity drop is found at a residence time up to 30 hours. Between 30 and 45 hours the purity drop is reduced to 0.5, but it can be a little larger if the minimum temperature can be reduced (Rein, 2007). For refinery molasses, a purity drop of 9.3 points was found after 48 hours of residence time where 69% of this drop was reached in the first 24 hours. However, a small percentage (1%) of this purity drop is due to sucrose destruction during the time of the cooling process and another small percentage of this purity drop is due to losses during centrifugation (Meade and Chen, 1977)

Stirring is important in cooling crystallization since it improves the heat transfer and reduces the dead zones and channeling to achieve plug-flow in continuous systems (Rein, 2007).

As with evaporative crystallization, cooling crystallization can be conducted in batch or in continuous crystallizers (van der Poel et al., 1998).

2.2.2.1. Batch Cooling Crystallizers

Batch cooling crystallizers are used more with batch pans. “C” massecuite is discharged from the “C” batch pan to the batch crystallizer where the massecuite is water cooled to the required temperature while kept in movement. The retention time will depend on the number of crystallizers. Most of the batch crystallizers are horizontal stirred vessels with a U-shaped cross section. The operation of the horizontal batch crystallizers requires more labor because of the number of valves, which also make automation difficult (Rein, 2007).
2.2.2.2. Continuous Cooling Crystallizers

Ideal conditions to achieve a good performance in continuous crystallizers are to have the right massecuite flow pattern, i.e. every particle of the massecuite spending the same time through the crystallizer – plug-flow, and to have a good transfer coefficient, i.e. homogeneous temperature through the cross section, perpendicular to the flow direction (Rein, 2007), Figure 2.9.

![Ideal flow and temperature conditions required for continuous crystallizer operation.](image)

**Figure 2.9.** Ideal flow and temperature conditions required for continuous crystallizer operation. Plug-flow and homogeneous cross section temperature

With the change from batch to continuous low grade station operation (continuous pan and continuous centrifuges) and to ease automation (fewer valves), it was required to update cooling crystallization from batch to continuous. Then, U-shaped horizontal crystallizers were connected in series, using gutters at the top or pipes at the bottom and with the help of stirrers, baffles and in general convoluting the flow direction. But horizontal crystallizers have several disadvantages, such as (Rein, 2007):

- They require a head difference to favor the massecuite flow, but the changes in the process flow may cause repressing and overflowing.
- They occupy more space than the vertical and they require a supporting steel work.
- They easily develop problems involving channeling and dead zones.
- They require liquidation pumps to empty each crystallizer.

Vertical continuous crystallizers occupy less space, can be placed on the ground and outside the factory building, can be larger, have lower installation costs for the same size and have fewer leak problems (Rein, 2007).

To guarantee plug-flow for an optimum residence time distribution in vertical cooling crystallizers, the flow direction of the massecuite has to be from the top to the bottom of the vertical crystallizer because of the density increment with the temperature reduction. In contrast, when the
flow direction is from the bottom to the top, hot massecuite with lower density trends to flow upward, producing “rat-holing”; this behavior is promoted by the temperature differences (Rein, 2007). Good heat transfer coefficients and plug flow are conditions that optimize the performance of a vertical crystallizer. Walls and stationary heat transfer surfaces must be sheared by the stirring elements to prevent massecuite accumulations that reduce the heat transfer coefficient and to prevent “dead zones” inside the crystallizer that reduce the residence time. For crystallizer design, massecuite collection inside or between cooling elements has to be avoided. Baffles may prevent accumulation of massecuite rotating with the stirring elements, and fins may help to improve heat transfer coefficient (Rein, 2007).

Operation and control conditions for a good cooling crystallization are (Rein, 2007):

− Residence time between 30 and 45 hours. Keep the maximum level (overflow weir).
− Minimum temperature achievable. Correct amount of water to the right temperature, in countercurrent with the massecuite flow.
− Accurate measurement of massecuite temperature. Temperature probes with enough length to give a reliable and representative measurement, and right thickness to avoid massecuite accumulation in the element, which gives false low temperatures.
− Avoid aeration of the massecuite by the stirring elements. Aeration increases viscosity.

Problems with drivers due to massecuites with high Brix or temperatures lower than the minimum (mill breakdown or cool weather) can be solved by (Rein, 2007):

− Reducing stirring speed
− Adding some molasses or surfactants (such as sodium hydrosulphite)
− Heating up the water used for the cooling

It is important to emphasize that the optimum performance of the cooling crystallization cannot replace the work that has to be done in the pans. Pan work determines a good massecuite condition, such as dry substance content, non-sucrose/water ratio (purity), crystal content and crystal size (Rein, 2007).
2.3. Boiling House Procedures

In order to meet the primary goal of an efficient sugar boiling house, which is the production of sugar with the proper quality requirements, leaving a minimum amount of sugar in the final molasses, the sugar coming in the evaporator syrup has to be recovered by several crystallization stages with very specific efficiency targets. Different boiling schemes are employed to fit the sugar quality requirements from a determined syrup quality. The best boiling scheme is one which gives the required sugar quality with the efficient use of the steam and the equipment available. Achieving the crystallization efficiency targets (exhaustion) has an important effect on minimizing the use of steam and optimizing the equipment capacity (pan, crystallizers and centrifuges) (Rein, 2007).

2.3.1. Schemes or Systems

The recovery of sugar by crystallization cannot be achieved in one stage; the sucrose present in the syrup is recovered in several stages (strikes), which are required to reduce the purity of the mother liquor (exhaustion). Viscosity (better called consistency for non-newtonian fluids) of the massecuites limits the exhaustion in each stage. A common boiling scheme or system consists of 2, 3 or 4 boiling stages (strikes) where the last stage requires longer time than the former and, at the end the low grade massecuite is submitted to cooling crystallization to recover the maximum possible amount of sugar. In general, for sugar manufacturing in Louisiana, the different massecuite grades are designated by letters (“A”, “B”, “C” massecuite) or by numbers (first, second, third massecuite);
molasses and sugars (magmas) from the same massecuite take the same designation (“A” molasses, “A” sugar). (Chen and Chou, 1993).

Common boiling schemes (Figure 2.11) used for raw sugar production are: two-boiling scheme, three-boiling scheme, Double-Einwurf scheme and VHP scheme (Rein, 2007).

The choice of a boiling scheme depends on factors such as syrup purity and sugar quality, and this choice also affects the massecuite throughput and the steam requirement (Birkett, 1978).

![Figure 2.11. Boiling schemes](image)

### 2.4. Recommendations for Molasses Exhaustion

Molasses exhaustion is a topic that has been studied for many years. Several researchers across the sugar industry have discussed this subject, and they agree on the conditions required to achieve good molasses exhaustion. The recommendations taken from some of these researchers – Birkett and Stein (1988), Ninela and Rajoo (2006), Meade and Chen (1977), Chen and Chou (1993) and Rein (2007) – are as follows:
2.4.1. “Boil low purities “C” strikes” since “The lower the “C“ strike purity, the lower will be the final molasses purity.” (Birkett and Stein, 1988)

This goal can only be accomplished if the exhaustion in the “A” and “B” strike (crystal content) is kept under control. Ninela and Rajoo (2006) recommend increasing the crystal area for deposition of sucrose and the crystal content in the “A” strike by increasing the footing of Magma. An increase in the crystal content in the “A” strike will reduce the size of the crystal, but the crystal yield will increase. The target crystal content for “A” and “B” strikes in South Africa is calculated with the following equation (Rein, 2007):

\[
w_{C, ds} = 0.78 \cdot P_{Ma} - 10
\]

(2.13)

Target “C” massecuite purity can be estimated from the molasses target purity. High purity “C” massecuite leads to high volumes of “C” massecuite reducing the exhaustion capacity of pan, crystallizers and centrifuges. High purity “C” massecuite also leads to a high crystal content, which increases the massecuite consistency. For instance, increasing crystal content from 27 to 35%, “C” massecuite consistency may be approximately twice higher. However, “C” massecuite purity has to be high enough to assure a crystal content of 25% (Rein, 2007).

2.4.2. “Low cooled massecuite temperatures are necessary for maximum final molasses exhaustion.” (Birkett and Stein, 1988)

“C” massecuites are cooled in the crystallizers to a final temperature of 45–50°C (113–122°F) for high viscosity massecuites and can be as low as 35°C (95°F) for low viscosity massecuites (Chen and Chou, 1993). Optimum results on cooling crystallization are obtained when the massecuite is rapidly cooled to the minimum temperature required for equipment operation and held on this temperature for the rest of the time. Saska (1990) recommended a cooling rate of 1.5–2°C/hour (2–3°F/hr). Decloux (2000) refers to a minimum residence time of 24 hours in crystallizers to obtain good purity drop. It is important to determine residence time distribution (RTD), to check if the massecuite flow is closed to plug flow or if the retention time is reduced due to dead zones or channeling (Figure 2.12), which require design changes to improve the flow behavior. To determine the RTD, a tracer (zinc or lithium) has to be placed on the input and samples of the crystallizers output are taken and analyzed by atomic absorption.
2.4.3. “Reheat the final cooled massecuite back to the saturation temperature prior to centrifuging.” (Birkett and Stein, 1988)

To ease the separation of the molasses from the crystals on the centrifuges the “C” massecuite should be reheated to a temperature lower than its saturation temperature, 49–52°C (120–125°F). In extreme conditions, higher temperatures may be required in order to reduce massecuite consistency but cannot be higher than 60°C (140°F). Rapid reheating (15°C/hour) and rapid purging (maximum time 3 hours) are required to avoid the dilution of the sugar crystals. Reheater areas are approximately 4.5 m²/TC for a Δt (water to massecuite temperature difference) of 3°C (Rein, 2007).

2.4.4. Centrifugal Management.

To achieve good molasses exhaustion, a screen management program has to be implemented, hot water and steam have to be controlled, machine cleaning and maintenance have to be scheduled and a periodic evaluation of the purity rise from the reheater to centrifuges has to be conducted. Purity rise in the “C” centrifuges should be no higher than 3 and it is better if it is lower than 2 (Rein, 2007).
2.4.5. Slurry Preparation and Grain Strike

A standard procedure for the preparation of slurry must be established and the proper slurry to massecuite ratio determined. Although full seeding is the ideal to achieve an optimum crystal size distribution, some nuclei may form when the slurry is injected to the pan. The proper amount of slurry to be used has to be determined by experience (Rein, 2007).

The difference between seeding with powder sugar and seeding with slurry is the amount of crystals required to give the right crystal surface area for crystallization. The smaller is the size of the crystal, the bigger is the crystal surface area for the same number of crystals (Rein, 2007).

The purity of the grain strike has to be kept in a range between 64 and 68% using “A” molasses and combining with “B” molasses if it is required (Rein, 2007).

2.4.6. Crystal Size and Crystal size Distribution (CSD) are very Important Parameters to be Determined in Low-Grade Massecuite

The combination of crystal content and crystal size determines the crystal surface area available for sucrose deposition (avoiding the formation of false grain). As was stated before, for the same crystal content, smaller crystals have more surface area than larger crystals. However, small crystals reduce the purging capacity of the centrifuges and increase the molasses recirculation. Also, small crystals may pass throughout the centrifuges’ screen slots and increase the molasses purity. The proper size is established balancing the advantage of small size crystals (higher surface area) for the rate of crystallization versus large crystals (lower surface area) for the centrifugation. Crystal sizes recommended for grain (“C” massecuite seed) are between 180 and 200 µm, for “C” massecuite leaving the pan are between 250 and 300 µm and for “C” massecuite leaving the “C” crystallizers are between 300 and 350 µm. The purity rise in centrifuges depends mainly in the crystal size. Crystal width has to be 1.5 to 2.0 times larger than the width of the screen slot, which is 0.04 or 0.06 mm (40 or 60 µm). Minimum crystal width should be 120 µm (Rein, 2007, Chen and Chou, 1993, and Hugot, 1960).

Meade and Chen (1977) state that crystal uniformity is a desired quality because – “with a mixture of crystal sizes, the pore volume of sugar is reduced and drainage of molasses is slower and less complete in the centrifugal operation”. To achieve crystal size uniformity, the steady state parameters in a continuous pan are the key: constant production rate and regulated seed injection (Chou, 2000).
Ninela and Rajoo (2006) state that “the Crystal Size Distribution (CSD) is an important analytical tool that assists process management to make informed decisions”. This information can be used to adjust the seed/massecuite ratio to achieve the desired crystal content. The objective is to boil “C” massecuites with regular grains and free of false grain.

![Figure 2.13. Crystal picture and CSD analysis for “C” massecuite from CVP](image)

2.4.7. The “C” Massecuite has to be Boiled to the Maximum Density within the Limits of Workability for Crystallizers and Centrifuges

Mother liquor in the massecuite has to be concentrated to an optimum density (Brix) that will give – after cooling crystallization – a final molasses saturation of about 55°C (131°F) (Meade and Chen, 1977).

To achieve the target purity of the final molasses, the Brix (RDS) of the “C” massecuite should be at least 97 and a plan to achieve the optimum Brix of 98 must to be implemented in small increment. The molasses purity cannot be further reduced for molasses viscosities above approximately 300 Pa-s (Rein, 2007).

Maximum massecuite density is limited by the massecuite viscosity. A “C” massecuite viscosity limit about 2,000 Pa-s at 40°C (104°F) is recommended, which corresponds to approximately a 4.5 non-sucrose/water ratio. The maximum viscosity of the massecuite leaving the crystallizers can be close to 5,000 Pa-s at 40°C (104°F), and the design of the crystallizers can be addressed to handle 10,000 Pa-s at 40°C (104°F) (Rein, 2007).

Reduction of viscosity by the addition of water or highly diluted molasses in the massecuite receiver (before crystallizers) is not recommended because of the dilution of the crystals, which is
facilitated by the high temperature of the massecuite; instead, addition of final molasses at saturation temperature and surfactants is a better option (Saska, 1990).

The conductivity profile in a continuous pan has to be approximately linear from the first to the last compartment and can be adjusted according to the experience. The set-point changes in a continuous pan have to be done by small steps and the production rate has to be controlled with the evaporation rate (Rein, 2007).

2.4.8. The Lower the Quantity of “C” Massecuite, the Better the Molasses Exhaustion and the Lower the Quantity of Molasses.

The amount of “C” massecuite affects the retention time in crystallizers and the capacity of pans and centrifuges. Quantity of “C” massecuite depends on several factors (Rein, 2007).

- Juice purity (non-sucrose in juice). The higher the non-sucrose content in juice, the higher the “C” massecuite quantity. Values of 1.5 to 2.0 m$^3$ (53.0 to 70.6 ft$^3$) of “C” massecuite/ton of non-sucrose in juice have been found in practice
- Recovery of non-sucrose, which is the net effect of the elimination and formation of impurities. The lower the recovery of non-sucrose, the lower the “C” massecuite quantity. A normal range of non-sucrose recovery is between 0.97 and 1.06.
- “C” massecuite purity. The higher the purity of “C” massecuite, the higher “C” massecuite quantity.
- “C” sugar purity. The lower the purity of “C” sugar, the higher the “C” massecuite quantity (molasses recirculation).

2.4.9. Routine Measurement of Nutsch Molasses (“C” Massecuite Mother Liquor Separated from Crystals by Filtration under Pressure) (Rein, 2007).

A periodic monitoring of the “C” massecuite mother liquor has to be implemented in order to detect purity drop and crystal content changes after each stage for the low grade station, i.e. pan, crystallizer and reheater (Rein, 2007).

- It is expected that 60 to 75% of the purity drop is obtained in the pan (Meade and Chen, 1977).
- The crystal content goal for the massecuite leaving the pan is between 25 and 28% based on South Africa experience (Ninela and Rajoo, 2006).
The purity rise after the reheater must be zero “0” and sometimes can be negative (Rein, 2007).

2.5. Sugar Boiling Summary

Supersaturation, the driving force of the sucrose crystallization process, depends on the sucrose solubility, which is affected by parameters such as temperature, pressure and the composition of the non-sugars in solution. Keeping supersaturation in a specific value inside the metastable zone ($y=1.2$) and the right crystal surface area (crystal content and crystal size) are the keys to achieve an optimum crystal growth rate and to avoiding nucleation or sucrose dilution. Consistency is recognized as the most important process constraint since it limits the sucrose diffusion (crystal growth), the heat transfer and the final crystal content (sugar recovery). The consistency affects flow pattern in continuous vacuum pans and crystallizers, flowability in pipes, power consumption (pumps and stirrers), and centrifugal work. Consequently, the mechanical strength of the equipment bounds the consistency of the material. Evaporative and cooling crystallization (batch or/and continuous) are the techniques applied in the boiling house to produce raw sugar.

The recommendations for molasses exhaustion addressed equipment design and process parameters per stage. Equipment design focus on the flow pattern to give the required retention time (crystal growth) and the material movement (mass and heat transfer). Recommended process parameters refer to solids concentration, temperature, retention time, crystal content (purity drop) and crystal size distribution that improves the molasses exhaustion on each stage of the low-grade station. Cane quality and a good performance of the process stages before the low-grade station, are fundamental to maximize sugar recovery. This information was the basis to define the required materials and methods, and to analysis the results of this research.
3. MATERIALS AND METHODS

One important aspect of any research is to establish what materials and methods can be used to approach the proposed objectives. The analytical procedures described are implemented by most of the Louisiana sugarcane factories or are conducted by Audubon Sugar Institute – Louisiana State University Agricultural Center. The obtained results can be reproduced by other factories and can be repeated season-to-season allowing the comparison. Crystal-molasses separation, soluble solids concentration and sucrose concentration were the basis for this work. Determination of crystal size, consistency, non-sucrose components, and metal tracer analysis complemented the information used to evaluate each crystallization stage of the low-grade station.

3.1. Analytical Procedures

3.1.1. Nutsch Filtration

Nutsch filtration allows extracting the mother liquor (nutsch molasses) from the massecuite to evaluate the exhaustion (sugar recovered by crystallization) achieved in any stage of the low grade station. The nutsch filter used on this test was designed in South Africa at the Sugar Mill Research Institute (SMRI), Figure 3.1. The sample is poured into the nutsch filter as soon as possible after collecting, in order to conduct the filtration at a temperature close to the process temperature. The sample level must be a minimum 1 cm (1/2 inch) above the screen, and then the upper part of the device is put in place and the screws are tighten enough to avoid air leaks. A line with air at high pressure 6 – 7 bar (87 - 100 psi), is plugged into the top and the molasses sample (~100g) is taken as soon it crosses the screen (100 Mesh, 5.5 µm opening width, 32.8 % open area).

![Figure 3.1. Nutsch filter, SMRI (2009)](image-url)
3.1.2. Sample Preparation

For analysis dilute massecuites and molasses samples with water (1:1 by weight), weighting 50 g of sample ($samplewt$) and 50 g of DI water ($waterwt$):

$$\text{Dilution Factor} = DF = \frac{samplewt + waterwt}{samplewt}. \quad (3.1)$$

3.1.3. Pol (or Apparent Sucrose Content)

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 angle of rotation of the polarized light at a given wavelength and temperature is an intrinsic property of a pure chemical compound, and the values of this rotation can be negative (levorotatory rotation) or positive (dextrorotatory rotation). 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)

In the last stage of the boiling process, massecuites and molasses have more non-sucrose components than sucrose. The optical rotation for non pure materials is the result of the summation of the individual rotations which affects the determination of the sucrose concentration. The “pol” values normally give an approximation below the real values. However, due to low cost and low operator’s skill requirements this technique is used in the sugar industry for a quick assessment of each stage of the manufacturing sugar process.

The procedure followed for pol analysis of “C” massecuites and final molasses, is: 26.00g ($w_{sln}$) of the diluted sample (1:1 dilution) are weighed into a 200 ml volumetric flask and brought to volume with DI water; then the solution is transferred to a 200 ml jar and the required amount of Octapol™ (C massecuites 8 – 10g, final molasses 10 – 15 g) is added covering the jar with a lid. After shaking, the jar content is filtered through filter paper Whatman 91 (or equivalent) discarding the first ~10 ml of filtrate. Approximately 100 ml of the filtrated solution are collected in order to rinse and fill the 200 mm glass cell (for the continuous cell ~150 ml of filtrate). The cell is placed in
the polarimeter and the pol is read at 589 nm (the instrument should be zeroed before for the same cell with DI water).

For a more rigorous determination, the pol reading has to be corrected by the temperature of the solution which is determined placing a thermometer in the mouth of the cell (the continuous cell is implemented to give the solution’s temperature).

\[
Pol = Pol \text{ read} \cdot DF \cdot \frac{52}{w_{sln}}
\]  (3.2)

\[
Pol = Pol \text{ read} \cdot DF \cdot \frac{52}{w_{sln}} \cdot [1 - c \cdot (t - 20)]
\]  (3.3)

\(w_{sln} = \) weight the of diluted sample (1:1)
\(DF = \) dilution factor
\(t = \) temperature of the tested sample solution

\(c = 0.000467\) tube and flask: borosilicate (589 nm) ICUMSA 1/2/3/9-1 (2007)
\(0.000490\) tube and flask: borosilicate (880 nm) ICUMSA 1/2/3-2 (2005)
\(0.000455\) tube: steel; flask: borosilicate (589 nm) ICUMSA 1/2/3/9-1 (2007)
\(0.000478\) tube: steel; flask: borosilicate (880 nm) ICUMSA 1/2/3-2 (2005)

**Instruments:**
Optical activity polarimeter AA-10R, accuracy 0.02 °Z, wavelength 589 nm (Enterprise mill)
Rudolph Research Autopol 880 Saccharimeter, accuracy 0.01 °Z, wavelengths 589 and 880 nm (Audubon Sugar Institute)

**3.1.4. Refractometric Dry Substance (RDS % or Brix):** ICUMSA 4/3-13 (2007)

Dry substance (DS) is the mass percentage (%) of solids presents in a solution. The dry substance is the mass of solids remaining after the water is evaporated and it is determined by an oven drying method. For pure sucrose solutions the dry substance gives the mass of sucrose in
solution, but for impure sucrose solutions it gives the total mass of sucrose and non-sucrose in solution.

A refractometer measures the speed of the light in a substance compared with the speed of the light in the air (refractive index). The refractive index is related to the concentration for a given material and it depends on the temperature and the wavelength of the light; however, different materials may have the same refractive index at different concentrations. Brix is the scale used to convert the refractive index to sucrose concentration % mass, which is also called refractometric dry substance (RDS). Refractometric dry substance (RDS) for pan boiling products is generally higher than the dry substance by the oven dry method. The goodness of the prediction of the dry substance from refractive index depends on the purity of the material, ICUMSA SPS-3 (2000).

The °Brix or refractometric dry substance (%RDS) is read directly from the refractometer display after covering the refractometer prism with few drops of the diluted sample (1:1 dilution). Presence of air bubbles and suspended solids also affect the Brix values. The removal of the suspended solids by centrifugation improves the precision of the measurement.

\[ Brix = Brix\ read \cdot DF \] (3.4)

**Instruments:**
- Index Instruments PTR2 Refractometer, accuracy 0.1 °Brix (Enterprise mill)
- Bellingham and Stanley RFM340 Refractometer, accuracy 0.03 °Brix (Audubon Sugar Institute)

3.1.5. **Viscosity:** ICUMSA Method SPS-5 (1994)

In general, viscosity is a quantity which describes the resistance of a material to flow. The viscosity depends on the fluid material (pure and impure), temperature and sometimes on pressure. When the viscosity can be represented by a number which relates shear stress with velocity gradient (shear rate), the fluid is known as a Newtonian. But when the viscosity is a function of a mechanical variable such as shear stress or time, the fluid is known as a non-Newtonian. “C” massecuites and final molasses are shear-thinning (pseudoplastic) non-Newtonian fluids that follow the power law model, i.e. the apparent viscosity decreases when shear rate increases.

The viscosity of the nutsch molasses samples was determined at 40 and 60 °C using a Brookfield Programmable DV-II+ Viscometer, with a small sample adapter (coaxial cylinder, SC4-
28 spindle). The speed was chosen to give a torque reading between 20 and 80%. Three speeds were used with each sample, and for each speed the apparent viscosity was recorded every minute for five minutes. Assuming that the molasses follows the power law model, also known as the Ostwald-de Waele equation ($\mu_{app} = K \gamma^{n-1}$), the consistency $K$ and flow index $n$ are evaluated by plotting the natural logarithms of apparent viscosity against shear rate according to the equation:

$$\ln \mu_{app} = \ln K + (n - 1) \ln \gamma$$  \hspace{1cm} (3.5)

- $\mu_{app}$ = apparent viscosity of the material, Pa•sec (centipoises $\cdot 10^{-3}$)
- $K$ = consistency, Pa.s$^n$ = viscosity (or stress) at a shear rate of 1 sec$^{-1}$
- $\gamma$ = Shear Rate, sec$^{-1}$
- $n$ = flow index (dimensionless). Measure the extent of non-Newtonian behavior

3.1.6. Crystal Size and Crystal Size Distribution

Sucrose crystals, like any three-dimensional objects, are better described by length, breadth and height; therefore a single number cannot give a good definition of the crystal size. Some techniques used to determine crystal size and crystal size distribution are: sedimentation, wet sieving, image analysis, coulter counter and laser techniques (laser diffraction and back scattering) (van der Poel et al., 1998). Most of these sizing techniques approximate the crystal shape to a sphere where the size is given by the diameter. Although this approximation simplifies the representation of the crystal size distribution, when the crystal shape is far from the sphere the results have more variation (Cilas, 2004). The sucrose crystal habit which depends on the growth rate of each crystal face is affected by the temperature, supersaturation and the presence of non-sucrose components and for instance, the sucrose crystal can be elongated in some cases (van der Poel et al. 1998).

Particle size analysis based on laser diffraction and diffusion phenomena, relies on the fact that particles passing through a laser beam will scatter light at an angle that is directly related to their size. In laser diffraction, particle size distributions are calculated by comparing a sample’s scattering pattern with an appropriate optical model. Traditionally two different models are used: the Fraunhofer Approximation and Mie Theory (Cilas, 2004). The Fraunhofer approximation assumes that the particles being measured are opaque and scatter light at narrow angles. As a result, it is only applicable to large particles and will give an incorrect assessment of the fine particle fraction. Mie
Theory provides a more rigorous solution for the calculation of particle size distributions from light scattering data. It predicts scattering intensities for all particles, small or large, transparent or opaque (Cilas, 2004).

The crystal size and crystal size distribution for “C” massecuite are determined by laser diffraction (Cilas Particle Size Analyzer 1180, Figure 3.2). A saturated sugar solution (exactly 66.72 °Brix at 20 °C) is used to rinse the system 2-3 times and to fill the instrument cup. The sample is prepared by dispersing ~3-5 g of massecuite in ~5-6 ml of the saturated sugar solution with a spatula. After determining the background for the measurement, the prepared sample is added to the instrument cup until the Obscuration Number is between 9 and 14, then the crystal measurement is initiated. The software produces a graph for the particle size distribution and the results for mean size in μm (MA= \( D[4,3] \)), standard deviation in μm (\( \sigma \) = Deviation), size in μm for the first 10% of the distribution (d(10%)) and the size in μm for the 50% of the distribution (d(50%)). The coefficient of variation (CV) is calculated dividing the mean size (MA) by the standard deviation (\( \sigma \)). Figure 3.3 shows a crystal size distribution graph and results given by the report.

![Figure 3.2. Cilas Particle Size Analyzer 1180](image)

![Figure 3.3. Crystal size distribution graph and results](image)
3.1.7. **Sugars by High Performance Liquid Chromatography (HPLC):** ICUMSA GS7/4/8-23 (2002)

True Sucrose, Fructose and Glucose in final molasses are determined with high accuracy by liquid chromatography in Audubon Sugar Institute. An additional dilution is required to analyze the sample by HPLC, 1.000g of the final molasses 1:1 solution is weighed by duplicate into 100 ml volumetric flasks then they are taken to volume with DI water.

The diluted samples are filtered through a 0.45 µm filter into a glass sample vial. Three level of standards, containing sucrose (1000, 2000, and 3000 ppm), glucose (100, 300, and 600 ppm) and fructose (100, 300, and 600 ppm), are run at the beginning and the end of each six (6) samples set to verify accuracy.

The instrument and operating conditions are:

- **HPLC instrument:** Agilent 1200 HPLC System
- **Column:** Aminex HPX-87K 300 * 7.8 mm
- **Column Heater:** Agilent 1200 thermostatted column compartment (G1316A) at 80°C
- **Detector:** Agilent 1200 refractive index detector (differential refractometer)
- **Software:** ChemStation
- **Auto Sampler:** Agilent 1200 autosampler with temperature control at 5°C
- **Injection volume:** 20 µl
- **Pump:** Agilent 1200 Isocratic Pump
- **Flow rate:** 0.6 ml/min
- **Solvent:** 0.01 M K₂SO₄ solution (mobile phase)

3.1.8. **Conductivity Ash:** ICUMSA GS1/3/4/7/8-13 (1994)

Conductivity ash correlates the specific conductance of a solution to the sulfated ash concentration. The sample dilution is such that the conductivity measurement does not exceed 500 µS/cm so, 0.900 g of the 1:1 final molasses solution is weighed (wₛ) into a 200ml volumetric flask and then it is brought to volume with DI water.

The calibration of the conductivity meter is checked and 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.

The temperature correction for a temperature range of 20 ± 5 °C is:

\[
C_{20°C} = \frac{C_T}{[1 + 0.023 \cdot (T - 20)]}
\]

(3.6)

\(C_T\) = Conductivity in µS/cm at T °C

Corrected conductivity of the solution

\[C = C_1 - C_2\]

(3.7)

\(C_1\) = measured conductivity in µS/cm at 20 °C

\(C_2\) = specific conductivity of the water in µS/cm at 20 °C

\[Conductivity\ Ash(\%) = \left(16.2 + 0.36 \cdot D\right) \cdot 10^{-2} \cdot C \cdot f\]

(3.8)

\(D\) = dry substance concentration of the solution in g/100ml

\(S\) = mass of sample (g) in 100 ml \((S = \frac{w_S}{DF})\)

\(f\) = dilution factor of the solution in comparison with 5 g/100 ml, i.e. \(f = 5/S\)

3.1.9. Zinc Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

ICP-AES is a type of emission spectroscopy that uses the inductively coupled plasma to produce excited atoms and ions. As these atoms and ions return from the excited state to the ground state, they emit electromagnetic radiation at a determined wavelength which is according to the chemical character of the element. The intensity of the emitted energy is proportional to the element concentration in the sample. The element concentration can be quantified by comparing to a reference standard, Wikipedia (2009).

For trace analysis determination it is very important to have a sample pretreatment to: digest the organic matrix, decrease viscosity, increase homogeneity, release the analytes from various
compounds and phases and remove components which may affect the analytical signal. Wikipedia (2009)

For the sample preparation, microwave assisted acid digestion is used (EPA Method 3052), 0.5g of sample is weighed into teflon cup adding 9 ml of concentrated nitric acid and 1 ml of concentrated hydrochloric acid. The cup is sealed and placed in microwave where the temperature is ramped to 200 °C over 10 min., then held at 200 °C for 15 min., then cooled. The sample is transferred to 100 ml volumetric and brought to volume with DI water. Now it is ready for ICP-AES (EPA Method 200.7), AgChemistry (2009)

3.2. Evaluation of Purity Drop and Crystal Content for each Stage of the Low-grade Station (Nutsch Analysis)

**Sampling:** Once a week, samples of “C” massecuite and final molasses were taken in the following order (see Table 3.1 and Figure 3.4):

- “C” massecuite after crystallizers (reheater inlet);
- “C” massecuite after continuous vacuum pan – CVP (pan discharge, before “C” massecuite receiver);
- “C” massecuite after reheater (centrifuges feed) and;
- Final molasses from each centrifuge (7 machines).
- “C” massecuites from each individual crystallizer for nutsch analysis were taken 3 times during the season.
- Sampling and analysis were run and reported each Monday during 10 weeks

<table>
<thead>
<tr>
<th>Stage</th>
<th>Massecuites</th>
<th>Molasses</th>
</tr>
</thead>
<tbody>
<tr>
<td>Continuous Vacuum Pan (CVP)</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Continuous Crystallizers</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Reheater</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Centrifuges</td>
<td></td>
<td>70</td>
</tr>
</tbody>
</table>
3.3. Retention Time Distribution (RTD) Determination for the Continuous Vertical Crystallizers Using Zinc as Tracer

Since supersaturation is the driving force of the crystallization process (the higher the supersaturation, the higher the crystallization rate), cooling crystallization has to reduce the temperature to increase or sustain the supersaturation. In cooling crystallization the sucrose deposition is slow and the rate is controlled by lattice incorporation instead of diffusion. According to these conditions, the most important process parameters to optimize the exhaustion of the mother liquor in continuous crystallizers are: the retention time (crystallization rate), cooling temperature (level of supersaturation) and crystal surface (lattice incorporation) (Rein, 2007).

The flow behavior of the massecuite through the continuous vertical crystallizer, determines the retention time and the mixing characteristics that have important consequences in crystallization time and heat transfer. The ideal massecuite flow behavior within a continuous crystallizer should follow a “dispersed plug flow model with complete mixing in the radial direction, and practically no mixing in the axial direction”. Channeling or back mixing in the axial direction and, dead zones reduce the retention time in the crystallizer. The retention time and the degree of mixing are very important factors to determine in the design of continuous vertical crystallizers (Rouillard and Smith, 1981).

In tracer studies, the mixing and hydraulic characteristics of a vessel are evaluated by imposing a certain input stimuli at the inlet and by determining the results of the stimuli at the outlet. Some conditions for a good tracer test are:
− The tracer can be easily measured
− The tracer or property (e.g. absorbance, fluorescence, pH, and conductivity) is not modified by the operation conditions
− The tracer is completely mixed with the fluid exhibiting the same flow pattern as the fluid.

Various tracers have been used for the evaluation of crystallizers in the sugar industry. Some of them are: lithium chloride, radioactive iodine, zinc sulphate, and magnesium salts. The use of PVC rings as a tracer has also been mentioned to evaluate crystallizers design (Rouillard and Smith, 1981).

**Tracer Test Design:**

Cane Grinding rate........................................~ 22,000 tc/day
“C” massecuite/ton of cane...............................~1 ft³/tc
Continuous Vertical Crystallizers capacity ..........~31,000 ft³
Zinc Sulphate Technical grade...........................~95% ZnSO₄·H₂O
% Zn in ZnSO₄·H₂O........................................~34.6 %
Application of 95% ZnSO₄·H₂O .........................~9.072 kg
ZnSO₄·H₂O Solubility ......................................~0.7 g/mL H₂O
Solution of Zinc Sulphate (95%) .........................~9.072 kg/4glns H₂O
Injection Point............................................Compartment 11 continuous vacuum pan
Injection Rate ..........................................~1 gal/min

Zinc sulphate (~9.072 kg) was dissolved in 4 gals of water and then it was injected in the compartment 11 of the continuous vacuum pan using the same compartment’s vacuum. The “C” massecuite leaving the pan was sampled every 10 minutes during 2 hours. Five hours after, “C” massecuites hourly samples were taken from the top of each crystallizer during 44 hours. Zinc analysis was performed by AgChemistry using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).
3.4. Materials and Methods Summary

Nutsch filtration and determination of Brix and pol for the massecuite and the separated molasses were the most important methods used to measure the level of molasses exhaustion along the season at the low-grade station. Variation of the material purity, the purity drop and crystal content were determined and compared with recommended and calculated parameters to achieve a zero molasses target purity difference. Crystal size distribution, consistency and supersaturation were included to complement and explain the exhaustion results serving as a reference for future evaluations.

Crystallizers’ exhaustion and tracer tests were run for the 4 crystallizers to compare and evaluate the individual performance and the flow pattern inside of each crystallizer.
4. RESULTS AND DISCUSSION

The degree of molasses exhaustion season-to-season varies according with the quality of the cane, the weather, the milling rate, the equipment and the operation of the factory. A comparison with past performance tells if the actual factory operation and capacity gives satisfactory results taking into account the quality of the material processed. The Results and Discussion chapter begins with the comparison of past factory performance (6 years) and the influence of the material quality. For the studied season, the graphs show the variation in quality material and boiling house operation, analyzing the implications on molasses exhaustion. Then, the discussion is focused on each stage of the low-grade station according with the crystallization theory and the recommendations mentioned on chapter 2. The analysis is based on purity drops, crystal content, crystal size distribution, consistency and supersaturation. Individual flow pattern and performance of continuous vertical crystallizers were compared. Finally, a financial evaluation of the factory during the season supports the importance of this study.

4.1. Final Molasses and Syrup Parameters between 2003 and 2008

Quality parameters such as syrup purity, reducing sugars and ash have strong impacts on sugar production, molasses purity and factory performance. Low syrup purities reduce sugar production, and high non-sucrose content increases the volume of the “C” massecuities and final molasses, reducing the factory capacity. Figure 4.1 shows that the syrup purity at Enterprise factory improved in 2008 compared with the years 2004 - 2007. In 2008, reducing sugars showed the lowest value and conductivity ash the highest value (final molasses, Figure 4.2), producing the lowest reducing sugars/ash ratio (RS/A ratio) for this five-year period. This RS/A ratio is related to sugar recovery and to the final molasses purity.

Although there was a lower RS/A ratio at Enterprise mill in 2008, it had a lower average true purity than the year before. The average target purity difference (TPD) for 2008 was 7.1 close to the TPDs obtained in 2006 and 2003, Figure 4.3.
**Figure 4.1.** Syrup yearly averages: reducing sugars (RS), conductivity ash (A) and true purity

**Figure 4.2.** Final molasses yearly averages: reducing sugars (RS), conductivity ash (A) and reducing sugars/ash ratio (RS/A ratio)

**Figure 4.3.** Final Molasses yearly averages: true purity and target purity difference (TPD)
4.2. Factory and Boiling House Graphs 2008 Season

Boiling house operations were evaluated for a period of 10 weeks (from 10/12/2008 to 12/22/2008). The molasses survey (Audubon Sugar Institute) and factory reports were used to review the boiling house performance focus on molasses exhaustion for Enterprise mill. The quality of the trends and correlations is affected by the error involved in the traditional methods of analysis (Brix and pol) but also in the factory production numbers (sugar, cane and molasses) which are estimated.

Figure 4.4 shows that the true purity increases and the reducing sugars (RS) decrease in syrup along the 2008 season. This is a normal pattern related to the age of the cane (maturity), which increases through the harvest season.

![Figure 4.4](image)

**Figure 4.4.** Syrup weekly averages (2008): reducing sugars (RS), conductivity ash (A) and true purity

Figure 4.5 shows that the RS/A ratio in molasses decreased as the season progressed with a similar pattern as in syrup. The ash content in final molasses stays more or less stable after the fourth week.

![Figure 4.5](image)

**Figure 4.5.** Final molasses weekly averages (2008): reducing sugars (RS), conductivity ash (A) and RS/A ratio
Figure 4.6 shows the variation of the true purity and the target purity difference (TPD) on the final molasses. The molasses true purity had the highest value the last week of the sampling period.

![Figure 4.6. Final molasses weekly averages (2008): true purity and target purity difference (TPD)](image)

During the season, the grinding rate increased gradually from ~800 to 990 tons of cane per hour (TCH), but sugar yield (ton sugar 96 pol/100 ton cane) started to decrease from the third week (Figure 4.7). Sugar yield is an indicator of the overall sugar recovery relative to the cane and normalized for a sugar pol of 96, sugar yield involves mill extraction and boiling house recovery. The drop in the sugar yield may have two explanations: the reduction of retention (crystallization) time because of the increment of the massecuites flow rates; and the reduction of the “A” massecuite crystal content because of a low seed/massecuite ratio.

![Figure 4.7. Final molasses weekly averages (2008): Grinding rate – ton cane/hour (TCH) and sugar yield (ton sugar 96 pol/100 ton cane)](image)
Figure 4.8 shows that “A” and “C” massecuites volumetric flow rates increased with the grinding rate (TCH). Molasses exhaustion strongly depends on the volume that has to be processed by each stage (low purity materials require higher retention times).

![Image of Figure 4.8](image)

**Figure 4.8.** Weekly averages (2008): “A” and “C” massecuites volumetric flow rates (ft$^3$/hour)

Massecuites and molasses volumetric flow rates are also affected by the non-sucrose content of the syrup. Table 4.1 and Figure 4.9 indicate that molasses production (gal@79Brix/ton of cane) increased as the syrup apparent purity decreased (Enterprise mill 2008).

\[
gal\_mol@79\ Brix/tc = 44.57 - 0.4529 \cdot SyrupPurity
\]  
(4.1)

**Table 4.1.** Molasses production (gal@79Brix/ton of cane) correlated to syrup purity

<table>
<thead>
<tr>
<th></th>
<th>R Square</th>
<th>Signif F</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>gal/tc</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>df</td>
<td></td>
<td>9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coefficients</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Intercept</td>
<td>44.5744</td>
<td>0.0001</td>
<td>83.6</td>
<td>86.2</td>
</tr>
</tbody>
</table>

![Image of Table 4.1](image)

**Figure 4.9.** Molasses production versus syrup purity in 2008
Figure 4.10 shows that the sugar yield decreased as the “A” massecuite purity drop decreased. In crystallization, the sucrose goes from the solution to the crystal, reducing the purity of the solution (mother liquor or molasses) therefore, the difference between the purity of the massecuite and the purity of the molasses (purity drop) is roughly related to the massecuite exhaustion. “A” Massecuite exhaustion is important for sugar recovery and for the final molasses exhaustion.

Figure 4.10. Weekly averages (2008): A massecuite purity drop (massecuite purity – molasses purity) and sugar yield (ton sugar 96 pol/100 ton cane)

According to Rein (2007), crystal content for “A” and “B” massecuites is related to the purity of the massecuite (higher purities, higher exhaustions). Factories in South Africa estimated a target or goal crystal content for “A” and “B” massecuites with a correlation (crystal content% dry substance=0.78*purity-10) obtained from historical data. Figure 4.11 and Figure 4.12 show the variation of apparent purity, the actual crystal content and the goal crystal content for “A” and “B” massecuites during the 2008 season for Enterprise. The actual crystal content for “B” massecuite shows good correlation ($R^2=0.91$) with the “B” massecuite purity, but the correlation for “A” massecuite purity with crystal content is lower ($R^2=0.56$). The crystal content is also determined for the amount of footing (seed/massecuite ratio) used and is limited by the viscosity.
Figure 4.11. Weekly averages (2008): “A” massecuite apparent purity and % crystal (actual and goal)

Figure 4.12. Weekly averages (2008): “B” massecuite apparent purity and % crystal (actual and goal)

Figure 4.13 shows the changes of crystal content and purity of the “C” massecuite estimated from the pan nutsch sampling each week. Birkett and Stein (1988), Ninela and Rajoo (2006) and Rein (2007) recommend that the purity of the “C” massecuite should be as low as it can to give a crystal content between 25 and 28% at the exit of the pan, and the Brix as high as the maximum material viscosity that can be handled by the pumps and by the equipment of the low-grade station.

Figure 4.13. Weekly pan sampling (2008): C massecuite apparent purity and % crystal (actual)
Table 4.2 shows the statistics that establish the goodness of a model that explain the variation of the sugar yield. The model predicts that the sugar yield (tons sugar 96 Pol % tons cane) increases with the increment in syrup purity (~29%), “A” massecuite/ton of cane volume ratio in ft³/tc (~47%) and, “A” massecuite purity drop (24%).

\[
\text{Yield}(96\text{Pol}) = -27.05 + 0.30 \cdot \text{SyrPty} + 1.47 \cdot \text{Amass/tc} + 0.32 \cdot \text{Amass_PtyDrop} 
\]  \quad (4.2)

<table>
<thead>
<tr>
<th>Model</th>
<th>R Square</th>
<th>Signific F</th>
<th>Min</th>
<th>Max</th>
</tr>
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<tbody>
<tr>
<td>Yield</td>
<td>0.9334</td>
<td>0.0006</td>
<td>10.0</td>
<td>11.5</td>
</tr>
<tr>
<td>df</td>
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<td></td>
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<tr>
<td></td>
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</tr>
<tr>
<td></td>
<td>Coefficients</td>
<td>P-value</td>
<td>Min</td>
<td>Max</td>
</tr>
<tr>
<td>Intercept</td>
<td>-27.0521</td>
<td>0.0032</td>
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</tr>
<tr>
<td>Syr Purity</td>
<td>0.3033</td>
<td>0.0019</td>
<td>83.6</td>
<td>86.2</td>
</tr>
<tr>
<td>&quot;A&quot;mass/tc</td>
<td>1.4780</td>
<td>0.0017</td>
<td>4.1</td>
<td>4.7</td>
</tr>
<tr>
<td>Pty Drop</td>
<td>0.3225</td>
<td>0.0037</td>
<td>15.6</td>
<td>18.3</td>
</tr>
</tbody>
</table>

Figure 4.14 shows the plot between observed and predicted sugar yield values (R²=0.93). This model highlights the importance of the purity drop (equivalent to crystal content) in sugar recovery.

Figure 4.15 indicates with a probability of 97% that the target purity difference (TPD) drop is predicted in 67% by the increase in the “C” massecuite Brix. Rein (2007) states that to get a TPD close to zero “0” the Brix of the massecuite has to be at least 97%. Ninela and Rajoo (2006) recommend keeping the Brix in a range between 96.5 to 97.5%.

\[
\text{TPD} = -1.84 \cdot \text{CmassBrix} + 183.5 
\]  \quad (4.3)
Table 4.3. Molasses target purity difference (TPD) correlated to “C” massecuite Brix

<table>
<thead>
<tr>
<th></th>
<th>$R^2$</th>
<th>Signific F</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>TPD Model</td>
<td>0.6694</td>
<td>0.0038</td>
<td>5.4</td>
<td>9.4</td>
</tr>
<tr>
<td>df</td>
<td>9</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coefficients</td>
<td>P-value</td>
<td>Min</td>
<td>Max</td>
<td></td>
</tr>
<tr>
<td>Intercept</td>
<td>183.4903</td>
<td>0.0031</td>
<td>94.9</td>
<td>96.5</td>
</tr>
<tr>
<td>&quot;C&quot;massBrix</td>
<td>-1.8405</td>
<td>0.0038</td>
<td>94.9</td>
<td>96.5</td>
</tr>
</tbody>
</table>

Figure 4.15. Target purity difference versus Brix of “C” massecuite

4.3. Crystallizers Performance Evaluation

Exhaustion in a crystallizer depends on the crystal surface area (combination of crystal content and crystal size), final temperature of cooling and retention time. Crystallizer design, operation and material characteristics limit the exhaustion for a particular crystallizer. Figure 4.16 shows a picture of the 4 vertical cooling continuous crystallizers at Enterprise mill. From the right to the left, crystallizers 1 and 2 were installed in 1995 and crystallizers 3 and 4 were installed in 2008.

Figure 4.16. Enterprise continuous vertical cooling crystallizers (Honiron)
“C” massecuite from the pan at temperatures from 65 to 70 °C (149 to 158 °F) is dropped to the “C” massecuite receiver. From the receiver, ~ 48% is pumped with individual pumps to the bottom of crystallizers 1 and 2 and ~52% is pumped with a single pump to a distributor box, which feeds the bottom of the crystallizers 3 and 4. Figure 4.17 shows the massecuite flow for the cooling crystallization stage at Enterprise mill. The cooling temperature is measured and controlled in the middle and in the top for crystallizers 1 and 2 and it is measured in 5 points and controlled in 2 points for the crystallizers 3 and 4.

![Figure 4.17. “C” massecuite flow through the continuous cooling vertical crystallizers](image)

Table 4.4 shows the general assumptions for crystallizers’ calculations and Table 4.5 shows the operating conditions estimated for the flow distribution implemented during the tracer test. It may be noticed that with this flow distribution, crystallizers 3 and 4 have to remove 100,000 BTU/hr more than crystallizers 1 and 2.

### Table 4.4. General assumptions for crystallizer calculations

<table>
<thead>
<tr>
<th></th>
<th>units</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>C Mass. volume per ton cane</td>
<td>ft³/tc</td>
<td>1.0</td>
</tr>
<tr>
<td>Grinding rate (TCH)</td>
<td>tons cane/hour</td>
<td>1,000</td>
</tr>
<tr>
<td>C Mass. total flow</td>
<td>ft³/tc</td>
<td>1,000</td>
</tr>
<tr>
<td>C Mass. Density (97 °Brix)</td>
<td>lb/ft³</td>
<td>98.82</td>
</tr>
<tr>
<td>C Mass. specific heat</td>
<td>Btu/lb/°F</td>
<td>0.47</td>
</tr>
</tbody>
</table>
Table 4.5. Continuous vertical cooling crystallizers operating conditions

<table>
<thead>
<tr>
<th></th>
<th>units</th>
<th>CR1</th>
<th>CR2</th>
<th>CR3</th>
<th>CR4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crystallizer Volume</td>
<td>ft³</td>
<td>7,500</td>
<td>7,500</td>
<td>8,000</td>
<td>8,000</td>
</tr>
<tr>
<td>C Mass. Temp. out of receiver</td>
<td>°F</td>
<td>145</td>
<td>145</td>
<td>145</td>
<td>145</td>
</tr>
<tr>
<td>C Mass. Temp. out of crystallizer</td>
<td>°F</td>
<td>110</td>
<td>110</td>
<td>110</td>
<td>110</td>
</tr>
<tr>
<td>C Mass. Temp. drop in crystallizer</td>
<td>°F</td>
<td>35</td>
<td>35</td>
<td>35</td>
<td>35</td>
</tr>
<tr>
<td>C Mass. flow to crystallizer % total</td>
<td>%</td>
<td>19</td>
<td>23</td>
<td>29</td>
<td>29</td>
</tr>
<tr>
<td>C Mass. flow to crystallizer</td>
<td>ft³/hr</td>
<td>190</td>
<td>230</td>
<td>290</td>
<td>290</td>
</tr>
<tr>
<td>C Mass. flow to crystallizer</td>
<td>lb/hr</td>
<td>18,776</td>
<td>22,729</td>
<td>28,658</td>
<td>28,658</td>
</tr>
<tr>
<td>C Mass. retention time (plug flow)</td>
<td>hour</td>
<td>39</td>
<td>33</td>
<td>26</td>
<td>26</td>
</tr>
<tr>
<td>Required heat loss</td>
<td>Btu/hr</td>
<td>308,862</td>
<td>373,885</td>
<td>471,421</td>
<td>471,421</td>
</tr>
</tbody>
</table>

Figure 4.18 shows the temperature changes for each crystallizer. Cooling temperature measurement was affected by accumulation of massecuite around the probes which reduced the performance of the temperature controllers, mainly for crystallizers 1 and 2. Temperatures recorded at the bottom of the crystallizers were between 140 and 150 °F and at the top were between 115 and 125 °F. Dead zones and channeling reduce the crystallizer capacity. Temperature monitoring should be as close as possible to the temperature of the moving massecuite in order to have a better control of the cooling temperature. Baffles, stirring and heat exchange areas modifications may help with the flow and temperature control (Rein, 2007). These design modifications will help increase crystal content at the exit of the pan (higher density and consistency), lower the cooling temperature and therefore achieve a higher purity drop in both pan and crystallizers.

![Figure 4.18. Registered temperatures in the output of crystallizers and in the input of reheater](image-url)
Table 4.6 summarizes the results of the crystallizer evaluations. Figure 4.19 shows the nutsch molasses purity and purity drop per crystallizer. Considering an average purity drop of 13 points in the pan, the average partial drop for crystallizers 1 and 2 is about 7 (31% of the total drop) compared with the average partial drop for crystallizers 3 and 4 which is about 9 (43% of the total drop). The difference was about 2.4 on purity drop and crystal content.

<table>
<thead>
<tr>
<th>Crystal</th>
<th>Nutsch</th>
<th>Pty Drop</th>
<th>% Crystal</th>
<th>CV(%)</th>
<th>MA(µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRYST 1</td>
<td>33.6</td>
<td>19.6</td>
<td>27.7</td>
<td>53</td>
<td>250</td>
</tr>
<tr>
<td>CRYST 2</td>
<td>33.2</td>
<td>20.0</td>
<td>28.1</td>
<td>51</td>
<td>253</td>
</tr>
<tr>
<td>CRYST 3</td>
<td>31.0</td>
<td>22.2</td>
<td>30.2</td>
<td>51</td>
<td>258</td>
</tr>
<tr>
<td>CRYST 4</td>
<td>30.7</td>
<td>22.5</td>
<td>30.5</td>
<td>46</td>
<td>264</td>
</tr>
</tbody>
</table>

Figure 4.19. Nutsch molasses purity and total purity drop per crystallizer

Figure 4.20 shows the variation of crystal content and crystal size per crystallizer. The crystal size was about 8 µm larger for crystallizers 3 and 4 than the average values obtained for crystallizers 1 and 2.

Figure 4.20. C massecuite % crystal content and crystal size
4.4. Retention Time Distribution (RTD) in Vertical Cooling Crystallizers

Table 4.7 shows the factory conditions and Figure 4.21a-b shows the sampling and injection point for the tracer test (zinc as a tracer). The test was run for 44 hours. During the first 9 hours of the test the massecuite flow distribution into the crystallizers (according to the rpm of the pumps) was ~48% to crystallizers 1 and 2 and, 52% to crystallizers 3 and 4, then it was changed to ~42% to crystallizers 1 and 2 and 58% to crystallizers 3 and 4. Assuming that the flow pattern inside the crystallizer is perfect plug-flow the expected nominal retention time for crystallizers 1 and 2 (42% of the massecuite) would be ~42 hours and for crystallizers 3 and 4 (58% of the massecuite) would be ~33 hours.

Table 4.7. Factory conditions during evaluation of the RTD

<table>
<thead>
<tr>
<th>Factory Conditions</th>
<th>11/29/08</th>
<th>11/30/08</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tons Cane Ground</td>
<td>20,081</td>
<td>22,418</td>
<td>21,250</td>
</tr>
<tr>
<td>Grinding rate, per hour</td>
<td>908.64</td>
<td>934.08</td>
<td>921.89</td>
</tr>
<tr>
<td>C Mass/ton cane, ft³/tc</td>
<td>0.84</td>
<td>0.99</td>
<td>0.92</td>
</tr>
<tr>
<td>C Mass Brix</td>
<td>95.53</td>
<td>95.80</td>
<td>95.70</td>
</tr>
<tr>
<td>C Mass Purity</td>
<td>53.00</td>
<td>53.91</td>
<td>53.49</td>
</tr>
<tr>
<td>C Mass flow, ft³/h</td>
<td>763.30</td>
<td>924.74</td>
<td>847.00</td>
</tr>
<tr>
<td>C Mass flow, ton/h</td>
<td>34.28</td>
<td>41.54</td>
<td>38.10</td>
</tr>
<tr>
<td>Cryst 1 - 2 (42% c-mass), estimated hours</td>
<td>46.79</td>
<td>38.62</td>
<td>42.11</td>
</tr>
<tr>
<td>Cryst 3 - 4 (58% c-mass), estimated hours</td>
<td>36.14</td>
<td>29.83</td>
<td>32.53</td>
</tr>
</tbody>
</table>

Figure 4.21a. Zinc injection and “C” massecuite sampling to evaluate retention time distribution
Figure 4.21b. Zinc injection and “C” massecuite sampling to evaluate retention time distribution

Figure 4.22 shows zinc concentration at the exit of the continuous vacuum pan which is considered the pulse injection for the 4 crystallizers. The tracer injection was considered a pulse since the peak width (time in minutes) is small respect to the peak height. The main objective at the injection point is to have enough tracer and massecuite well mixed, so that after the distribution and passage through the crystallizers, the zinc concentration at the output can be detected and used to determine the retention time and flow behavior of each crystallizer. The expected response for each crystallizer with an ideal plug flow with radial dispersion pattern must be a narrow peak (almost same width and height than the pulse injection) showing after the nominal retention time. Figure 4.23 shows the concentration variation and the area under the curve for each crystallizer.

Figure 4.22. Zinc concentration after pulse injection. Continuous Vacuum Pan – CVP
Table 4.8 presents a summary of the tracer test experiment. The results from the tracer test did not show the flow behavior that was expected from the crystallizers’ performance evaluation. It was expected that the old crystallizers 1 and 2 would show lower retention time and more channeling compared with the new crystallizers 3 and 4. Probably, the reason for this was that more massecuite was being pumped to crystallizers 3 and 4 (~58% compared to ~52%), which, worsened the flow pattern inside the crystallizers (Rein, 2007). The nutsch molasses purity difference between the new and the old was larger than 2 points, and during the tracer test the difference was just 1 point. Since the massecuite proportion was changed 9 hours after the zinc injection, it can be stated that with this change, the flow behavior in crystallizers 3 and 4 tended to have the same flow pattern as that in crystallizers 1 and 2.
Table 4.8. Summary of the tracer experiment results

<table>
<thead>
<tr>
<th></th>
<th>Zinc</th>
<th>CVP</th>
<th>CR 1</th>
<th>CR 2</th>
<th>CR 3</th>
<th>CR 4</th>
<th>Reh.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average Zn, ppm</td>
<td>58</td>
<td>20</td>
<td>9</td>
<td>15</td>
<td>13</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Max Zn, ppm</td>
<td>176</td>
<td>164</td>
<td>18</td>
<td>102</td>
<td>36</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Net Area, ppm^3/hours</td>
<td>100</td>
<td>364</td>
<td>2</td>
<td>100</td>
<td>40</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Estimated flow rate, tons/hour</td>
<td>35</td>
<td>7</td>
<td>7</td>
<td>9</td>
<td>9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Estimated Zn recovered, kg</td>
<td>3.14</td>
<td>3.47</td>
<td>2.38</td>
<td>0.01</td>
<td>0.90</td>
<td>0.36</td>
<td>3.65</td>
</tr>
<tr>
<td>Estimated Nominal Residence Time, hours</td>
<td>42</td>
<td>42</td>
<td>33</td>
<td>33</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Residence Time Distribution - τ, hours</td>
<td>25</td>
<td>22</td>
<td>25</td>
<td>23</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dispersion - σ^2, hours^3</td>
<td>90</td>
<td>113</td>
<td>183</td>
<td>127</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Estimated Capacity Reduction, %</td>
<td>40</td>
<td>48</td>
<td>24</td>
<td>30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C massecuite, Brix</td>
<td>94.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>93.1</td>
</tr>
<tr>
<td>C massecuite, Purity</td>
<td>52.9</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>52.8</td>
</tr>
<tr>
<td>Nutsch molasses, Brix</td>
<td>92.9</td>
<td>91.4</td>
<td>91.0</td>
<td>91.2</td>
<td>91.0</td>
<td>90.0</td>
<td></td>
</tr>
<tr>
<td>Nutsch molasses, Purity</td>
<td>40.6</td>
<td>33.2</td>
<td>33.5</td>
<td>32.3</td>
<td>32.3</td>
<td>33.1</td>
<td></td>
</tr>
<tr>
<td>Crystal Content, %</td>
<td>20.9</td>
<td>29.6</td>
<td>29.2</td>
<td>30.5</td>
<td>30.5</td>
<td>29.4</td>
<td></td>
</tr>
<tr>
<td>Crystal Size MA, µm</td>
<td>243</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>270</td>
</tr>
<tr>
<td>Coefficient of Variation</td>
<td>46</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>43</td>
</tr>
<tr>
<td>CV, %</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nutsch molasses, Consistency (140°F), Pa-s^a</td>
<td>147</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>50</td>
</tr>
<tr>
<td>Nutsch molasses, Consistency (104°F), Pa-s^a</td>
<td>1100</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>331</td>
</tr>
<tr>
<td>Average Temperature, °F</td>
<td>158</td>
<td>104</td>
<td>115</td>
<td>121</td>
<td>126</td>
<td>137</td>
<td></td>
</tr>
</tbody>
</table>

The estimation of zinc recovery for each crystallizer shows that it was a preferential flow mainly to crystallizer 1 and then to crystallizer 3. This can be due to pump performance, to massecuite distribution in the receiver or to massecuite distribution in the pipe probably occurring at the injection time. The test included the flow behavior in the “C” massecuite receiver, in the distribution pipe to the pumps and in the distribution box for crystallizers 3 and 4.

Although the test failures (change in the massecuite flow rate to the crystallizers and the preferential flow to crystallizers 1 and 3), at the specific conditions prevailing during the test, it can be said that (see Figures 4.22 and 4.23 and Table 4.8): crystallizer 1 had channeling and dead zones (40% capacity reduction); crystallizer 2 had dead zones and axial dispersion (48% capacity reduction); crystallizer 3 had channeling, dead zones and axial dispersion (25% capacity reduction); and crystallizer 4 had dead zones and axial dispersion (23% capacity reduction). Causes for the deviation from the desired flow pattern (which is plug flow with radial dispersion) may be:

→ Crystallizers design - baffles, size and design of the cooling coils (eccentric or concentric) and stirrer size.
→ Operation - speed of stirring, weirs position and flow distribution.
Flow direction - flow from the bottom to the top of the crystallizer, higher viscosities and densities of the colder massecuite in the top, favor the formation of stagnant zones and the formation of preferential paths where the hotter massecuite flows straight to the top.

Figure 4.24 shows the range of variation of the crystallizers’ output temperatures and the ambient temperatures during the test. There was a gradual reduction of the ambient temperature (from 66 to 49 °F). The ambient temperature drop contributed to reducing the crystallizers’ output temperature (from 119 to 114 °F), occurring by the heat loss through the crystallizers’ walls.

![Figure 4.24. Cooling and ambient temperatures variation during tracer test](image)

In summary, according to this experience the main difficulties that have to be addressed designing a tracer test in C massecuite cooling crystallizers, are:

- Type of tracer - zinc concentration in the original massecuite may vary from 5 to 10 ppm, affecting the accuracy of the results
- Incorporation of the tracer in the material - amount of massecuite and mixing to give a good representation of the flow behavior inside the crystallizer
- Sampling - the non-newtonian viscous massecuite has a laminar flow pattern inside pipes which affects the sampling (Rouillard and Smith, 1981)
- Viscosity truly affects the flow pattern inside the crystallizer - massecuite viscosity changes with the temperature gradient inside the crystallizer and changes also by time with the quality of the material processed by the factory.
4.5. Nutsch Molasses Consistency

Nutsch molasses consistency from the CVP and from the reheater were determined at 40°C (104°F) and at 60°C (140°F), assuming that these temperatures are close to the temperatures of the crystallizers inputs (140°F crystallizer bottom) and output (104°F ideal cooling temperature) and to the temperature of the reheater output (140°F maximum temperature). Table 4.9 shows the average values found for the CVP and reheater nutsch molasses estimating the consistency of the corresponding massecuites (assuming that the crystal content variation from 20 to 30% did not contribute to the massecuite consistency). Massecuite consistency is a gross estimation from a correlation (equation 4.4 - \( R^2 = 0.76 \)) found in 2005 for nutsch molasses and massecuite samples from several Louisiana mills (van der Poel, 1998, gives a \( \mu_{Ma}/\mu_{Mol} \approx 4.35 \) for 30% crystal content).

The apparent viscosity (\( \mu_{app} \)) for the massecuite can be estimated considering that the shear rate for pipeline work flow is \( \gamma = 1s^{-1} \) (Decloux, 2000); then it can be said that the massecuite apparent viscosity at the crystallizer output (104 °F) was around 2000 Pa•s (2x10\(^6\) centipoises).

\[
K_{Ma} = 5.8 \cdot K_{Mol} - 130.9
\]

(4.4)

Table 4.9. Average nutsch molasses consistency and estimated massecuite consistency in crystallizers and reheater

<table>
<thead>
<tr>
<th>Temp</th>
<th>Molasses</th>
<th>C Massec.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>°F</td>
<td>Brix</td>
</tr>
<tr>
<td>Cryst. Inlet</td>
<td>140</td>
<td>92.3</td>
</tr>
<tr>
<td>Cryst. Output</td>
<td>104</td>
<td>90.2</td>
</tr>
<tr>
<td>Reh. output</td>
<td>140</td>
<td>90.2</td>
</tr>
</tbody>
</table>

Rein (2007) states that the massecuite viscosity limit at the output of the crystallizer is about 2000 Pa•s and it can be as high as 5000 Pa-s. Figure 4.25 shows the variation of the consistency during the season. Weeks 5 and 8 had the highest consistencies at the output of the crystallizers; those weeks also had the lowest TPD’s of the season. It was noticed that the Brix of the massecuite after the reheater was 1 point lower and that the Brix of the molasses was 2 points lower compared to the Brix of the massecuite and molasses from CVP.

Addition of water or diluted molasses after the CVP is not recommended because of the high temperature of the massecuite that can facilitate the dilution of the crystals and waste the work done
at the pan. Addition of surfactants or recycling final molasses at the saturation temperature is recommended (Saska, 1990).

![Figure 4.25. Estimated input and output massecuite consistency and nutsch molasses consistency per week](image)

Table 4.10 shows the summary for the nutsch molasses consistency. The last sample from CVP was not used for the average since it was clearly an outlier (very low Brix and high purity). It can be noticed that the average consistency for the nutsch molasses from the CVP is almost twice the average consistency of the nutsch molasses from the reheater. Lower Brix, lower purity (by sucrose removal) and the addition of surfactants can be the reason for this difference.

**Table 4.10.** Summary for nutsch molasses consistency at 40 °C and 60 °C (CVP and Reheater)

<table>
<thead>
<tr>
<th>Samples</th>
<th>Molasses Consistency</th>
<th>40 °C (104 °F)</th>
<th>60 °C (140 °F)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Brix</td>
<td>Purity</td>
<td>n</td>
</tr>
<tr>
<td>CVP</td>
<td>Average</td>
<td>92.3</td>
<td>40.2</td>
</tr>
<tr>
<td></td>
<td>Std. Dev</td>
<td>0.6</td>
<td>0.8</td>
</tr>
<tr>
<td>Reheater</td>
<td>Average</td>
<td>90.2</td>
<td>32.6</td>
</tr>
<tr>
<td></td>
<td>Std. Dev</td>
<td>0.8</td>
<td>1.2</td>
</tr>
</tbody>
</table>

Rein (2007) gives an equation proposed by Rouillard (1984) relating the consistency \( K \) with the temperature \( T \) and the activation energy \( E_A \).

\[
K = K_0 \cdot \exp \left( \frac{E_A}{R \cdot T} \right) \tag{4.5}
\]

Using the equation (4.5) and recognizing that the solids concentration represents the variation of the consistency a model for nutsch molasses consistency was developed. The model is proposed to predict the consistency based on the consistency at 40 °C, the Brix and the temperature.
\[
\frac{K_2}{K_1} = \exp \left( 664.96 \cdot \text{molBrix} - 51,024 \right) \left( \frac{I}{T_2} - \frac{I}{T_1} \right)
\] (4.6)

Figure 4.26 shows that this model predicts in 91% the consistency of the nutsch molasses at 60 °C. Since the actual temperatures in the crystallizers output were between 46 and 52 °C (115 and 125°F), applying this model it can be estimated that the actual molasses consistency in the crystallizers output ranged between 91 and 316 Pa•s (the estimated massecuite consistency ranged between 400 and 1,700 Pa•s).

![Figure 4.26. Consistency K in Pa•s at 60 °C (140°F), predicted vs. observed](image1)

Figure 4.27 shows the estimated variation of the consistency with Brix and temperature applying the same model.

![Figure 4.27. Consistency K (Pa•s) variation with temperature and Brix](image2)

Rein (2007) states that actually the crystallizers can be designed to handle massecuite consistency up to 10,000 Pa-s and the operational consistency can be raised to 5,000 Pa•s. Some problems with flow ability were witness during the season, but were probably more related to the
flow in pipes from the crystallizer to the reheater. Molasses Brix (directly related to consistency) at the output of the crystallizer should be lower than molasses Brix at the exit of the continuous vacuum pan because of the removal of sucrose (crystallization) from the mother liquor. Optimizing pan and crystallizers operation (crystal content, cooling temperature, stirring and flow) the deposit of sucrose in the crystals will reduce the solids concentration in the mother liquor, reducing at the same time the consistency of the molasses and hence the consistency of the massecuite.

Viscosity is one of the most important parameters in molasses exhaustion at the low-grade station since it is a constraint for: crystallization (the sucrose diffusion is limited, the main process governing the crystallization rate at low temperatures is surface reaction); flowability in pipes and crystallizers (the cold massecuite has more pseudo-plastic flow behavior), centrifugation (the viscosity should be reduced by dilution but better by heat in order to separate the mother liquor from the crystals). According to these measurements and estimations, the viscosity of the massecuite leaving the crystallizer at 40°C (104°F) can be reduced approximately 10-fold (from 2,020 to 260 Pa•s) heating the massecuite in the reheater to 60 °C (140°F). The increase in massecuite production along the season changed the viscosity limit that could be handled by the equipment. The reduction in ambient temperature in the other hand increased the viscosity in pipes conducting the massecuite from the crystallizer to the reheater creating a bottleneck and forcing reduction of the Brix of the massecuite in the continuous vacuum pan (reducing the molasses exhaustion).

4.6. Crystal Size Distribution

The seed or grain to feed the “C” continuous vacuum pan (CVP) is prepared in a 1,300 ft³ batch pan using: sugar powder 10X in alcoholic suspension, a big proportion of “A” molasses and some B molasses to adjust the final purity (64-66). The crystal growth for the grain strike is conducted in 2 stages. After the first growth 2/3 of the pan capacity is transfer to a first development receiver and then 1/3 of the first development is used as a footing for the second development. Final purity and crystal size of the seed is adjusted in the second development.

Seed/massecuite ratio: The flow rate of seed (grain) to the continuous pan has to be regulated in order to obtain a determined crystal size avoiding the formation of false grain (percentages of seed with respect to massecuite range from 22 to 33%). The theoretical amount of seed to grow crystals from approximately 240 to 350 µm can be estimated by the equation (4.7), and Table 4.11 summarizes the results for “C” massecuite (Rein, 2007).
\[ \dot{m}_{\text{Seed}} = \dot{m}_{\text{Ma}} \cdot \frac{w_{\text{Cr, Ma}}}{w_{\text{Cr, Seed}}} \left( \frac{d_{\text{Seed}}}{d_{\text{Ma}}} \right)^3 \]  

(4.7)

**Table 4.11.** Seed/“C” massecuite ratio given by equation (4.7) (Rein, 2007)

<table>
<thead>
<tr>
<th>Pan Boiling</th>
<th>( \dot{m}<em>{\text{Seed}} / \dot{m}</em>{\text{Ma}} )</th>
<th>( w_{\text{Cr, Ma}} )</th>
<th>( w_{\text{Cr, Seed}} )</th>
<th>( d_{\text{Seed}} / d_{\text{Ma}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>C Pan</td>
<td>0.40</td>
<td>28</td>
<td>22</td>
<td>0.68</td>
</tr>
</tbody>
</table>

Figure 4.28 shows an estimation of the volumetric flow of grain (240 µm crystal size, 22% crystal content) that could be required for the volumetric flow (ft³/hr) of “C” massecuite (350 µm crystal size, 28% crystal content). The maximum flow rate of grain required was for the week number 9. This theoretical flow rate (40% seed/massecuite) gives less than 3 hours of strike time considering that the grain pan has a capacity of 1,300 ft³ (the average time for a grain strike is 4.5 hours).

![Graph showing volumetric flow rate over weeks](image)

**Figure 4.28.** “C” massecuite (ft³/hr) and theoretical grain flow rate calculation per week

At Enterprise, one seed strike is used for 3 grain strikes of 1300 ft³ and assuming that the desired final crystal size of the grain is 240 µm and no dilution or false grain formation is affecting the number of crystals, the seed calculations can be done applying the following equations (Meade and Chen, 1977):

\[ m_{\text{Cr,Grain}} = V \cdot \rho \cdot w_{\text{Cr,Grain}} \]  

(4.8)

\[ N_{\text{Cr,Grain}} = \frac{m_{\text{Cr,Grain}}}{1.11 \cdot l_{\text{Cr,Grain}}} \]  

(4.9)
\[ m_{Cr,Seed} = N_{Cr,Grain} \cdot 1.11 \cdot l_{Cr,Seed} \]  

\( m_{Cr,Grain} \) = total crystal weight in lb (*0.454 in kg)  
\( w_{Cr,Grain} \) = grain crystal content in %/100  
\( N_{Cr,Grain} \) = number of crystals in grain  
\( l_{Cr,Grain} \) = grain crystal size in cm (µm/10,000)  
\( l_{Cr,Seed} \) = seed crystal size in cm (µm/10,000)  
\( m_{Cr,Seed} \) = seed weight in g (kg= 10^{-3} g)  

Table 4.12 shows the amount of seed required to obtain a grain with a crystal content of 22% and a crystal size of 240 µm using full seeding. Rein (2007) mentions that any amount of seed used will produce seeding by shock and the proper amount of seed has to be determined by trial and error. The weight of seed at Enterprise during the season ranged from 1.0 to 1.5 kg compared to 14.9 kg from these calculations. Honig (1959) says that during the first 2 minutes after introducing the seed, 17 new nuclei’s crystals are going to be formed per each crystal that is introduced. Taking this into account (using the same sugar powder as seed), the weight of seed to be introduced would be 0.9 kg. However, small crystals can be easily dissolved at the solids concentration kept during the seeding point. The considerations with respect to the amount of seed to be used should lead to a uniform crystal size at the end of the grain strike. Continuous monitoring of grain size and automation can warrant a more homogeneous quality of the crystal in the grain stage.

<table>
<thead>
<tr>
<th>Table 4.12. Theoretical calculations to determine the weight of seed</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>units</strong></td>
</tr>
<tr>
<td>Grain strike volume</td>
</tr>
<tr>
<td>Grain Brix</td>
</tr>
<tr>
<td>Grain Density</td>
</tr>
<tr>
<td>Crystal content</td>
</tr>
<tr>
<td>Grain Crystal size</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Crystal weight in grain</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Number of crystal in grain</td>
</tr>
<tr>
<td>Seed Crystal size</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Required Seed weight</td>
</tr>
</tbody>
</table>
Crystal surface area is a very important parameter for low grade crystallization. The high consistency of “C” massecuite limits the diffusion of the sucrose to the crystals; therefore, it is required to have enough crystal surface area for sucrose deposition in order to avoid spontaneous grain formation. Honig (1959) gives the calculations (Kelly, 1946) to determine the minimum crystal surface area required to prevent spontaneous grain formation.

\[ S \cdot P^{1/4} < K \]  

\[ S = \frac{19 \cdot w_{Cr,Ma}}{l_{Cr,Ma} \cdot (100 - w_{Cr,Ma})} \]

\[ \begin{align*}
S &= \text{surface area in lb/ft}^2 \text{ of mother liquor} \\
w_{Cr,Ma} &= \text{massecuite crystal content in \%} \\
l_{Cr,Ma} &= \text{massecuite crystal size in mm (\(\mu\)m/1,000)} \\
K &= 2,000 (y=1.1); \quad K= 4,000 (y=1.2); \quad K= 8,000 (y=1.3); \quad K= 12,000 (y=1.4). \\
y &= \text{supersaturation} \\
P &= \text{purity pan mother liquor}
\end{align*} \]

Table 4.13 shows the theoretical calculations of the minimum surface area required to prevent spontaneous grain formation in crystallizers. According to these calculations, the surface area for 20% crystal content, 244 \(\mu\)m average crystal size and 40 average purity for molasses after the pan (Enterprise actual values) gives a \(K=3,406\), which is below the minimum \(K=4,000\) for a supersaturation of 1.2. But increasing the crystal content to 28% (Ninela and Rajoo, 2006) \(K=4,042\) above the minimum, despite that the crystal size is larger and the molasses purity is lower (case 3).

**Table 4.13.** Theoretical calculation to determine the minimum surface area

<table>
<thead>
<tr>
<th></th>
<th>units</th>
<th>case 1</th>
<th>case 2</th>
<th>case 3</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>C mass. crystal content</strong></td>
<td>%</td>
<td>20</td>
<td>28</td>
<td>28</td>
</tr>
<tr>
<td><strong>C mass. crystal size</strong></td>
<td>(\mu)m</td>
<td>244</td>
<td>244</td>
<td>290</td>
</tr>
<tr>
<td></td>
<td>mm</td>
<td>0.244</td>
<td>0.244</td>
<td>0.290</td>
</tr>
<tr>
<td><strong>Surface area</strong></td>
<td>ft(^2)/lb</td>
<td>19.5</td>
<td>30.3</td>
<td>25.5</td>
</tr>
<tr>
<td><strong>Purity of mother liq pan</strong></td>
<td></td>
<td>40.0</td>
<td>40.0</td>
<td>37.3</td>
</tr>
<tr>
<td><strong>super-saturation (y)</strong></td>
<td></td>
<td>1.2</td>
<td>1.2</td>
<td>1.2</td>
</tr>
<tr>
<td><strong>K</strong></td>
<td></td>
<td>3,406</td>
<td>5,298</td>
<td>4,042</td>
</tr>
</tbody>
</table>
Table 4.14 summarizes the results of crystal analysis for each stage during the 2008 season - crystal mean size (MA), coefficient of variation (CV), crystal size for the 50% of the distribution, and crystal size for the 10% of the distribution.

**Table 4.14.** Crystal size distribution by low grade station stage

<table>
<thead>
<tr>
<th></th>
<th>MA (µm)</th>
<th>CV (%)</th>
<th>D 50% (µm)</th>
<th>d 10% (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10X Sugar Powder</td>
<td>18</td>
<td>66</td>
<td>15</td>
<td>3</td>
</tr>
<tr>
<td>Grain</td>
<td>227</td>
<td>52</td>
<td>208</td>
<td>87</td>
</tr>
<tr>
<td>C-Massecuite Pan</td>
<td>244</td>
<td>48</td>
<td>225</td>
<td>103</td>
</tr>
<tr>
<td>C-Massecuite Reheater</td>
<td>268</td>
<td>45</td>
<td>259</td>
<td>109</td>
</tr>
</tbody>
</table>

Figure 4.29 shows these results in a bar diagram where the crystal growth and the reduction of the coefficient of variation can be clearly seen. The coefficient of variation (CV) of the grain was around 50. The CV of the “C” massecuite from the pan was normally around 44% and values over 52% were found twice for severe left tail. The “C” massecuite from the reheater had CV around 41%. On average the crystal grew 28 µm in the pan and 24 µm in the crystallizers. The minimum crystal growth rate for “C” massecuite in the pan - evaporative crystallization, is 18 µm/hour (Rein, 2007). In a continuous pan of 12 cells, it is known that the crystal grows rapidly in the first 9 cells and then the rate decreases when the Brix increases (tightening). Considering an average nominal retention time in the CVP of 6.4 hours and assuming that the crystal grew in the first 9 cells (4.8 hours), then the final size of the crystal in the “C” massecuite will be ~313 µm using the same grain with crystal size of 227 µm. Crystal dilution in the first cells of the pan or false grain formation in the last cells affects the distribution and hence affects the mean of the crystal size.

![Figure 4.29](image_url)

**Figure 4.29.** Mean crystal size (µm), coefficient of variation CV (%) and the crystal size for 10% of the distribution (µm) for different stages of the grain station.
Crystal size and distribution are important parameters to optimize since they are linked to the performance of the pan (CVP) and crystallizers (crystal content and purity drop), and ultimately to the performance of the centrifuges (purity rise). Enterprise centrifuge screens have a slot width of 0.04 mm (40 µm), and therefore the minimum crystal size should be 0.08 mm (80 µm). Sugar crystals with high coefficient of variation also affect the drainage of molasses in centrifuges. The improvement of these parameters starts with the implementation of a procedure to prepare sugar slurry, then the automation of the grain batch pan and finally the steady state operation of the continuous vacuum pan (regulated production rate and seed injection).

4.7. Supersaturation

Supersaturation is the driving force of the crystallization process; however, the supersaturation must be limited to the metastable zone in order to avoid the formation of new nuclei. When crystal surface area – which is linked to crystal content and crystal size – is low, the supersaturation obtained to a determined temperature can go to zones beyond the metastable zone. For pure sucrose solution the metastable zone is between 1 and 1.2, but in industrial sugar solutions the impurities concentration and the type of impurities affect the sucrose solubility. Rein (2007) states that at non-sucrose/water ratios (NS/W) between 0 and 3 the sucrose solubility decreases when the purity decreases, giving solubility coefficients (SC) between 0.8 and 0.9; and at NS/W > 3 ~ 4 the sucrose solubility increases when the purity decreases, giving SC>1. This change in sucrose solubility for impure solutions changes the boundaries of the crystallization zones. To achieve the molasses target purity Rein (2007) recommends a NS/W ≈ 4–4.5.

Table 4.15 shows the average solubility and supersaturation values corresponding to the 8 last weeks of sampling (weeks 3 – 8, nutsch molasses analyzed in Audubon Sugar Institute). The solubility coefficient (SC) was calculated using the RS/A ratio given by the molasses survey for the respective week and it was taken into account to calculate the supersaturation. It can be noted that apparently the “C” massecuite is still supersaturated at the reheater conditions, probably a reason for a low or negative purity rise.

Figure 4.30 shows that NS/W ratio decreased from 3.9 to 2.8 for the last 3 weeks in both CVP and reheater.
Table 4.15. Non-sucrose/water ratio, solubility coefficient and supersaturation for the actual conditions at Enterprise mill.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Temperature</th>
<th>NS/W Ratio</th>
<th>Sol. Coef</th>
<th>Supersat</th>
</tr>
</thead>
<tbody>
<tr>
<td>CVP</td>
<td>160°F</td>
<td>4.1</td>
<td>1.23</td>
<td>1.23</td>
</tr>
<tr>
<td>Crystallizers</td>
<td>120°F</td>
<td>3.8</td>
<td>1.18</td>
<td>1.63</td>
</tr>
<tr>
<td>Reheater</td>
<td>137°F</td>
<td>3.8</td>
<td>1.18</td>
<td>1.47</td>
</tr>
</tbody>
</table>

Figure 4.30. Estimated non-sucrose/water ratio for the mother liquor in the massecuite at the exit of the CVP and at the exit of the reheater per week.

Figure 4.31 shows the variation of the supersaturation. The supersaturation increased for the same last 3 weeks and the solubility coefficient decreased, getting close to 1.

Figure 4.31. Estimated supersaturation for the mother liquor in the massecuite at the exit of the CVP and at the exit of the reheater per week.

The most important aspect of this analysis is that the massecuite leaving the pan for the last 3 weeks had poor exhaustion (indicated by the high supersaturation values), and this could not be recovered in the crystallizers. The excuse from the factory point of view was a higher grinding rate and reduced flow ability of the massecuites transported from the crystallizers to the reheater due to
colder ambient temperatures (restricted flow of cold massecuite in pipes causing crystallizers overflow).

4.8. Nutsch Analysis for the Low-Grade Station

Sampling and nutsch tests were run to evaluate molasses exhaustion for a period of 10 weeks (from 10/12/2008 to 12/22/2008).

Figure 4.32 and Figure 4.33 show the variation of the molasses nutsch purity and crystal content. Week 9 was affected by the feeding of “A” molasses to the “C” continuous pan (CVP) because of maintenance of the “B” continuous pan during the sampling time. The nutsch molasses purity of the week 10 for the pan was also abnormally high, probably due to dextran presence. It can be seen that the performance of the pan stage (CVP) induces the results of the stage above (cooling crystallization).

![Figure 4.32. Variation of molasses nutsch purity by stage per week](image1)

![Figure 4.33. Variation of “C” massecuite %crystal (on dry substance) by stage per week](image2)
Figure 4.34 shows the variation of the final molasses purity after the centrifuges and the variation of the “C” massecuite purity after the reheater. There appears to be some relation between the “C” massecuite purity and the final molasses purity.

Table 4.16 summarizes the results for the nutsche tests; these averages are represented in Figure 4.35 and Figure 4.36.

The percentage of purity drop in the pan was 64%, compared with 60 - 75% (Meade and Chen, 1977). The crystal content at the exit of the pan was 21%, compared with 25 - 28% (Ninela and Rajoo, 2006). There is an opportunity of improving molasses exhaustion in the pan by increasing the crystal content.

The purity drop in crystallizers was 7.2 points on average but from crystallizers evaluation we can predict that with some design modifications on crystallizers 1 and 2, the purity could be increased to 10 points.

Purity rise after the reheater was 0.3 compared with “0” (Rein, 2007). The heat exchange area per ton of cane along the season fell from 1.5 m²/tc (16 ft²/tc) to 1.2 m²/tc (13 ft²/tc). Rein (2007) recommends a design value of 4.5 m²/tc (48.4 ft²/tc) for a Δt of 3°C. However, capacity and design of the reheater responded well to the process requirements. Reheater performance was little affected by the incrementental increase in the amount of material processed at the end of the season but there were some problems in temperature measurement. The recommended temperature is 55°C (131°F) and the temperature can be increased to values no higher than 60°C (140°F)
Table 4.16. Summary of nutsch analysis by stage

<table>
<thead>
<tr>
<th></th>
<th>Nutsch</th>
<th>% Purity</th>
<th>Crystal</th>
<th>% Purity</th>
<th>% Crystal</th>
<th>Pty Drop</th>
<th>% Purity</th>
<th>Acum. Pty Drop</th>
<th>% Drop</th>
</tr>
</thead>
<tbody>
<tr>
<td>PAN</td>
<td>40.0</td>
<td>20.8</td>
<td>13.1</td>
<td>13.1</td>
<td>64%</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CRYSTALIZERS</td>
<td>32.8</td>
<td>28.3</td>
<td>7.2</td>
<td>20.2</td>
<td>35%</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>REHEATER</td>
<td>32.5</td>
<td>28.9</td>
<td>0.3</td>
<td>20.5</td>
<td>1%</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CENTRIFUGES</td>
<td>34.4</td>
<td>28.5</td>
<td>-1.9</td>
<td>18.6</td>
<td>-9%</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.35. Average nutsch molasses purity and accumulated purity drop

Figure 4.36. Average crystal content and partial purity drop

Figure 4.37 shows the purity rise for each centrifuge. The average purity rise in “C” centrifuges was 1.9 compared with a recommended purity rise of 2 (for a good work) and no more than 3 (Rein, 2007). Enough centrifugation capacity, quality of the screens, regular load to the machines and scheduled maintenance are basic to avoid higher purity rise in the final molasses. With the exception of a few high values, the purity rise in centrifuges was more or less in the value recommended.
An improvement in the purity drop in the pan can have a substantial impact to reach a goal of zero TPD. Controlled Brix profile and final Brix, regulated seed feeding and the right set-point for the seed/massecuite ratio are mentioned as the most important parameters to reduce TPD (Rein, 2007) (Chou, 2000).

4.9. Goals to Achieve Zero Target Purity Difference

The best way to accomplish this objective is to set realistic targets or goals that can be adjusted according to the variation of the actual conditions. During the operation of a factory, particular circumstances may alter the normal conditions. Factory stoppages, cane delivery and cane quality affect the initial plan, and the factory should have a good response capacity to the changes. The response of the factory depends on the adaptability of the equipment and the process knowledge of supervisors and operators.

Process parameters are a conflicting issue during the factory operation. The knowledge, experience and beliefs of managers, supervisors and operators operate to set the process parameters. Target process parameters must be evaluated periodically by a well-designed monitoring program, where speed, reliability and applicability of the results are the key to making opportune decisions and to planning future improvements.

In order to get closer to a zero target purity difference, a plan of tests and a setting of specific goals for “C” massecuite should be purity were implemented. The idea was to quantify how low the purity of “C” massecuite according to the material and process performance to get a final molasses true purity close to the target purity (Ninela and Rajoo, 2006)(Rein, 2007).
Figure 4.38 shows the variation of the “C” massecuite purity, actual value and estimated goal. It can be seen that the estimated purity goal presented a large range of variation, approximately from 50 to 54. This difference can be explained from the variability in the performance of the pan (crystal content), which not only affects the continuous pan but also affects the performance of the crystallizers and centrifuges. Improving the crystal content in the pan, the “C” massecuite purity goal will have a lower variation.

![Figure 4.38. “C” massecuite actual purity and goal purity per week](image)

Figure 4.39 shows the variation of the CVP nutsch molasses purity, where the goal purity ranged approximately from 32 to 36. This high variability is related to the crystal content in the pan. An important concern is the rising of the consistency with higher Brix and higher crystal content. Probably, the lower consistency of the well exhausted molasses will impair the required increment in the crystal content. The transfer of sucrose from the solution to the crystal will reduce the molasses consistency, and the massecuite consistency is highly linked to the molasses consistency. Nevertheless, it is required to solve the equipment problems: flow pattern in crystallizers and in the pipes that connect the crystallizers to the reheater. The recycle of a small portion of final molasses at saturation temperature (~132°F) to the “C” massecuite receiver before the crystallizers is given as a recommendation to lower the consistency of the exhausted massecuite coming from the pan. Addition of water or diluted molasses will dissolve the crystal, and a large recycle of final molasses will increase the processing volume reducing the capacity of crystallizers and centrifuges.
Figure 4.40 shows the variation of the final molasses purity. It can be seen that the goal purities ranged from approximately 27 to 29, presenting a small gradual trend from the beginning to the end of the season due to the reduction in the total reducing sugars. This is the ultimate goal, to reduce approximately 6 points the apparent final molasses purity; of course, increasing the sugar recovery.

Table 4.17 shows the average for the actual values obtained during the sampling period as well as the possible goals that can be implemented to get closer to a zero “0” target purity difference (TPD=0). With some design modifications of the crystallizers 1 and 2, the partial drop during the cooling crystallization process should increase from 7 to 10 points, also that with an equal flow distribution of the massecuite in the crystallizers the consistency of the massecuite can be increased. This consistency increment is equivalent to higher Brix and higher crystal content at the exit of the
pan (better exhaustion in the pan). Figure 4.41 compares graphically the actual apparent purities and crystal content with the goal. According with this graph, the main changes to achieve the final goal of zero target purity difference are: increase the crystal content at the exit of the pan and improve the retention time and cooling temperature in crystallizers to increase the purity drop. Maintenance and operation of centrifuges are also important to reduce the purity rise.

Table 4.17. Low-grade station actual and goal parameters

<table>
<thead>
<tr>
<th></th>
<th>True Pty</th>
<th>TPD</th>
<th>App.Pty</th>
<th>Part Drop</th>
<th>%Crystal</th>
</tr>
</thead>
<tbody>
<tr>
<td>C MASS</td>
<td>53.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nutsch-PAN</td>
<td>40.0</td>
<td>13.1</td>
<td>20.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nutsch-CRYST</td>
<td>32.8</td>
<td>7.2</td>
<td>28.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nutsch-REH</td>
<td>32.5</td>
<td>0.3</td>
<td>28.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CENTR</td>
<td>42.3</td>
<td>6.9</td>
<td>34.4</td>
<td>-1.9</td>
<td>28.5</td>
</tr>
</tbody>
</table>

|                  |          |     |         |           |          |
| GOAL PARAMETERS LOW GRADE STATION |          |     |         |           |          |
| C MASS           | 54.9     |     |         |           |          |
| Nutsch-PAN       | 37.3     | 17.6| 28.0    |           |          |
| Nutsch-CRYST     | 27.3     | 10.0| 37.9    |           |          |
| Nutsch-REH       | 27.3     | 0.0 | 37.9    |           |          |
| CENTR            | 35.4     | 0.0 | 28.3    | -1.0      | 37.0     |

Figure 4.41. Low-grade station actual and goal parameters comparison

It can be said that the targets were very high standards for the given conditions during the 2008 season, but from this evaluation it is known which changes can be adopted to pursue these goals. A strategy has to be implemented to reach the proposed targets. Small steps have to be taken
and continued monitoring is required to avoid abrupt changes in the process. Design improvements of crystallizers 1 and 2 and higher crystal content at the pan exit are mentioned as the main areas to address to achieve the ultimate zero target purity difference goal.

4.10. Manufacturing Costs of the Sugar Lost in Final Molasses

One of the most important performance evaluations of a sugar factory is the sugar account (or pol account). Basically, this evaluation shows to managers and supervisors how much sugar was recovered in the final product (raw sugar in this case) and what are the main sources of sugar lost.

The sugar losses are in: filter cake, bagasse, molasses and undetermined losses. The main by-products, bagasse and molasses are responsible for the largest sugar losses. The challenge for managers and supervisors is to find a profitable way to recover more sugar by reducing the sugar losses.

Physical and economical limitations constrain the amount of sugar that can be recovered from a particular by-product. Therefore, it is important to recognize what are the parameters’ target values that can be pursued and to evaluate the performance against these targets. It has to be mentioned that the chosen target values are high standards that are experimental and empirically determined (molasses target purity, pol extraction and pol % filter cake) (Rein, 2007).

Table 4.18 compares the pol account with the actual and with the target values for the week #7 of the season. It can be seen that when the target values (for bagasse, molasses and filter cake) are achieved, the sugar recovered (%pol in cane) increases more than 3 points (from 84.2 to 87.7). When only the final molasses target is achieved, the recovery of sugar increases 1.7 points and the sugar losses in molasses fall from 7.37 to 5.65 (%pol in cane).

<table>
<thead>
<tr>
<th>POL ACCOUNT</th>
<th>Actual</th>
<th>Target</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% Cane</td>
<td>% Pol Cane</td>
</tr>
<tr>
<td>In bagasse</td>
<td>0.79</td>
<td>6.41</td>
</tr>
<tr>
<td>In Final Molasses</td>
<td>0.90</td>
<td>7.37</td>
</tr>
<tr>
<td>In Filter Cake</td>
<td>0.11</td>
<td>0.93</td>
</tr>
<tr>
<td>Undetermined Losses</td>
<td>0.13</td>
<td>1.09</td>
</tr>
<tr>
<td>Total Losses</td>
<td>1.94</td>
<td>15.80</td>
</tr>
<tr>
<td>Recovered in Sugar</td>
<td>10.31</td>
<td>84.20</td>
</tr>
<tr>
<td>In Juice</td>
<td>11.46</td>
<td>93.59</td>
</tr>
<tr>
<td>In Cane</td>
<td>12.25</td>
<td>100.00</td>
</tr>
</tbody>
</table>

Table 4.18. Pol account for low-grade station actual and goal parameters
The economic performance of any mill is affected mainly by the amount of cane ground and the amount of sugar recovered. Higher sugar cane tonnage gives lower fixed costs and higher net returns. Higher sugar recovery gives higher gross and net returns. The economical performance of a sugar cane mill is dictated by the following parameters (Salassi, 2008):

- Cost of natural gas
- Loss of sugar in filter cake
- Loss of sugar in bagasse
- Loss of sugar in molasses
- Commercially recoverable sugar (CRS)
- Overall sugar recovery

Table 4.19 gives an estimation of the average (10 weeks) **manufacturing cost of the sugar lost** for Enterprise mill, taking as a reference target values. These values may be used to predict the economical return that can be achieved when the targets are reached comparing with the total required investment.

<table>
<thead>
<tr>
<th>unit</th>
<th>Target</th>
<th>Actual</th>
<th>Partial</th>
<th>Acumulated</th>
</tr>
</thead>
<tbody>
<tr>
<td>filter cake</td>
<td>pol</td>
<td>2.0</td>
<td>4.5</td>
<td>$ 0.35</td>
</tr>
<tr>
<td>bagasse</td>
<td>pol</td>
<td>1.8</td>
<td>2.5</td>
<td>$ 1.03</td>
</tr>
<tr>
<td>molasses</td>
<td>purity</td>
<td>28.7</td>
<td>33.9</td>
<td>$ 1.03</td>
</tr>
</tbody>
</table>

**Sugar Price Season:** $20.33/ton (October 2008 – January 2009)

The estimated **net income** for the same period taking into account the fluctuation of the sugar price during the sampling period (USDA report) was $6.29 per ton of cane. Figure 4.42 shows a graphic approximation of how much the net income could be increased reducing the sugar losses to the target values.
The small sucrose recovery, achieving the final molasses purity goal, represents for 1,000,000 tons of cane ground a financial return of $1,030,000. The high financial potential of improving the factory profitability makes valuable whatever effort on reducing sucrose losses in final molasses (molasses exhaustion).

4.11. Results and Discussion Summary

Considering the influence of the syrup purity and the non-sucrose composition, Enterprise Factory had good molasses exhaustion during the 2008 season (target purity difference ~7.1) based on a 6 years comparison. Correlations showed that the molasses production rides on syrup purity; the sugar yield is related to syrup purity, “A” massecuite purity drop, and volumetric flow rates of “A” massecuite; and that the final molasses target purity difference depends on the Brix of the “C” massecuite. There is a high potential to increase sugar yield by increasing the crystal content of the “A” massecuite. “C” massecuite flow volumetric rate hinges on the exhaustion (purity drop) of the “A” and “B” strikes. There was a significant difference between the performance of the old and the new crystallizers for proportional flow distribution (on volume). A tracer test showed that the 4 crystallizers had flow pattern deviations from the ideal plug flow with radial dispersion (channeling, dead zones and axial dispersion). In addition, the measurement of the massecuite temperature inside the old crystallizers was not reliable. The nutsch molasses consistency was close to the recommended value (300 Pa*s^0.5) and had a strong correlation with the Brix and the temperature. Comparing the CVP and the reheater, the nutsch molasses from the reheater had lower Brix, lower purity, and lower consistency and the same temperature (104 °F) because of the exhaustion of the molasses in the crystallizers. The “C” massecuite at the exit of the continuous vacuum pan (CVP)
had crystal content below the recommended values (25 – 28%); and comparing the input (grain) and the output (‘C’ massecuite) at the CVP, it was found that sometimes the mean size of the crystal did not show crystal growth. The supersaturation at the close of the season was above of the safety zone at the exit of the CVP, showing not enough molasses exhaustion; the non-sucrose/water ratio was also below the recommended value (NS/w~4). Purity drops and purity goals analysis showed that the molasses exhaustion can be further improved by increasing the crystal content in the pan (25 – 28%) and modifying the flow pattern of the old crystallizers (retention time > 30 hours). Reheater and centrifuges had an average performance. Massecuite quality (Brix, purity and crystal size distribution) volumetric flow rate, temperature control and scheduled centrifuges maintenance will keep and overpass the actual performance of these stages. Finally, a financial evaluation showed that Enterprise factory may increase its net income in approximately 16% reducing the purity of the final molasses to its target value (zero target purity difference). Enterprise Factory has a high potential of increasing its sugar recovery by means of few changes on capacity and operation for each stage of the boiling house.
5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

This thesis mainly focused on the low-grade station operational parameters that could lead to a zero target purity difference on the final molasses (physical equilibrium purity). Theory was used to define the methods and to analyze the results. The expected outcome is that this information can be used to have a more efficient factory operation, giving achievable parameter goals and defining small design and operational adjustments that will lead to the ultimate goal of zero target purity difference.

5.1.1. Factory and Boiling House

Molasses exhaustion is a challenge for the sugar process that commonly is delegated to the boiling house and more yet to the low-grade station. An integral strategy for sugar recovery (molasses exhaustion) must include: a good cane quality delivered to the extraction process, regular operation of the factory (few stoppages and regulated processing volumes), good clarification, reduced exposition to high temperatures (heaters and evaporators) and a good exhaustion at the boiling house. In terms of molasses exhaustion, the grinding rate should be regulated according to the installed capacity and kept in a smooth pace without abrupt changes (steady state).

The “target purity” formula has been considered as the non-bias parameter to evaluate the factory performance in reference to the molasses exhaustion. Non-bias since it was determined at laboratory level considering the variation of the reducing sugars/ash ratio variation in molasses (as the most important sugar cane melassigenic parameter) and the consistency (as a constraint because of equipment design limitations). However, the target purity does not consider the influence of the particular variation of other non controllable variables such as: syrup purity, salts composition, dextran and other polysaccharides concentration. Concerned about local conditions, researchers from different sugar cane regions have developed their own target purity formula (Saska, 1999). The target purity formula used to evaluate the performance of the sugar cane factories in Louisiana was developed by Rein and Smith (1981) for the given conditions in South Africa and was evaluated and certified by Audubon Sugar Institute (Saska, 1999).
Comparing the molasses target purity difference for Enterprise mill for a period of 6 years, it was observed a small improvement in 2008 (7.1) compared with 2003 (7.2) and 2006 (7.3). The syrup purity for the years with low target purity difference was higher (89.4) than for the other three years (88.6) which means better molasses exhaustion for higher syrup purities.

High-purity materials render high sugar yields and low molasses quantities; furthermore, with low-purity materials the sugar yields decrease and the molasses quantities increase; hence, increasing the sugar losses in molasses. The quantities of “C” massecuites processed in the low-grade station increases with low purity material, reduced the exhaustion capacity of pans and crystallizers.

Molasses production was correlated to the syrup purity, indicating that molasses flow rate increases for low syrup purity. If syrup purity decreases from 86 to 84%, the final molasses flow rate increases 16%. If the “C” massecuite increases in the same proportion as the final molasses, for example, “C” massecuite could increase from 1.00 to 1.16 ft³/tc for a grinding rate of 900 TCH, there would be an additional 145 ft³/hr of “C” massecuite reducing the retention time in the continuous vacuum pan (1 hour) and in the cooling crystallizers (4 hours). Moreover, another correlation showed that the sugar yield was strongly related to the syrup purity, the purity drop in the pan and “A” massecuite/tc ratio. Assuming that the crystal content changes in proportion to the purity (crystal content goal) and that the “A” massecuite/tc ratio does not change, a syrup purity reduction from 86 to 84 could reduce the sugar yield from 11.7 to 11.5 tons sugar (96 pol)/100 tc. The sugar yield reduction for a grinding rate of 900 TCH is equivalent to 1.53 tons of sugar per hour (37 tons of sugar per day).

Deteriorated materials have also a big impact in the boiling house performance. Presence of dextran will increase the consistency of the massecuites reducing the rate of crystallization, modifying the crystals habits and reducing the flow ability of any material, regardless its purity. Analyzing data collected (ASI analytical lab) for Enterprise mill, the final molasses consistency (2004 season, equation 5.1) increases ~48% (13.7 to 20.3 Pa·s) and the final molasses true purity (2007 season, equation 4.2) increases 4 points, for increasing the dextran content from 1 to 2% solids (1000 to 2000 ppm/Brix). Any effort to improve the quality of the sugarcane in the field and to reduce the delay between the field and the factory (deterioration) will give without doubt good results in respect to molasses exhaustion and sugar yield.

\[
\text{Consistency, } K (\text{Pa s}^n) = -181.85 + 1.82 \cdot \text{molTS} + 0.0066 \cdot Dext + 0.91 \cdot \text{molTP} \tag{5.1}
\]
Table 5.1. Enterprise mill 2004. Final molasses consistency $K \text{(Pa} \cdot \text{s}^n\text{)}$ model fit statistics

<table>
<thead>
<tr>
<th></th>
<th>$R^2$</th>
<th>Sign. F</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K$ Model</td>
<td>0.9868</td>
<td>0.0000</td>
<td>2.8</td>
<td>36.2</td>
</tr>
<tr>
<td>df</td>
<td>10</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Coefficients</th>
<th>Sign. F</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intercept</td>
<td>-181.8450</td>
<td>0.0003</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mol. True Sol</td>
<td>1.8174</td>
<td>0.0002</td>
<td>76.5</td>
<td>83.0</td>
</tr>
<tr>
<td>Dextran</td>
<td>0.0066</td>
<td>0.0000</td>
<td>445.0</td>
<td>3772.0</td>
</tr>
<tr>
<td>Mol. True Pty</td>
<td>0.9060</td>
<td>0.0272</td>
<td>40.3</td>
<td>45.3</td>
</tr>
</tbody>
</table>

Figure 5.1. Enterprise mill 2004. Final molasses consistency at 40°C (Pa•s$^n$). Predicted vs. observed

\[ y = 0.99 \Delta x \]
\[ K^n = 0.9866 \]

\[ y = 0.99 \Delta x \]

\[ K^n = 0.9866 \]

\[ \text{Molasses True Purity} = -73.8 + 1.28 \cdot \text{Syrup True Purity} + 0.004 \cdot \text{Dextran} \] \hspace{1cm} (5.2)

Table 5.2. Enterprise mill 2007. Final molasses true purity (true sucrose\% dry substance) model fit statistics

<table>
<thead>
<tr>
<th></th>
<th>$R^2$</th>
<th>Sign. F</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>TrueP Model</td>
<td>0.9363</td>
<td>0.0000</td>
<td>40.7</td>
<td>46.0</td>
</tr>
<tr>
<td>df</td>
<td>10</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Coefficients</th>
<th>Sign. F</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intercept</td>
<td>-73.7924</td>
<td>0.0024</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SyrTruePty</td>
<td>1.2812</td>
<td>0.0001</td>
<td>87.1</td>
<td>89.8</td>
</tr>
<tr>
<td>Dextran</td>
<td>0.0040</td>
<td>0.0000</td>
<td>247.0</td>
<td>1481.0</td>
</tr>
</tbody>
</table>
The boiling house can be considered a cyclic process. One direction of the process is to recover the sugar, and the reverse direction is to grow the crystal. What happens upstream affects downstream and vice versa. A low exhaustion (low purity drop and low sugar yield) in “A” massecuites will increase the purity and the amount of material to be processed by the next steps, reducing their capacity to recover sugar. A low exhaustion of “C” massecuite renders low quality and low quantity of “C” sugar, which is going to reduce the crystal content of the “B” massecuite (double–Einwurf scheme), which in turn will reduce the crystal content of the “A” massecuite, affecting its exhaustion and continuing the cycle. The boiling house has to be well managed in such a way that each stage can do its best. Good quality syrup (high purity, no dextran) solves part of the problem upstream but down-stream the other part of the problem has to be solved on each stage of the low-grade station: seed preparation, evaporative crystallization, cooling crystallization, reheating and centrifugal separation.

Through the 2008 season at Enterprise mill, the grinding rate increased from the beginning to the end. The materials processed in the boiling house increased at the same pace, and the exhaustion capacity of the boiling house became limited at the same time. This affected primary the exhaustion of the “A” massecuite (sugar yield) and promoted a higher volumetric flow rate for the posterior stages (reducing retention time). The factory must look for a good exhaustion of the “A” massecuite which can directly benefit the sugar yield and will reduce the quantity and purity of the “C” massecuite. Amount and quality of the “B” magma are very important to keep the proper crystal content of the “A” massecuite (good exhaustion).
The purity drop in the CVP is responsible for 60-75% of the total purity drop (Meade and Chen, 1977). Any effort to increase this purity drop (higher crystal content about 25-28 %,) keeping the C massecuite purity as low as possible, will significantly increase the purity drop in crystallizers. Water is a non-sucrose component highly responsible for molasses formation. A Brix of 97 is required to achieve the proper crystal content in the “C” massecuite leaving the pan, and will lead to a molasses with a target purity difference close to zero (Rein, 2007).

Water evaporation during analysis (dilution with hot water in open container) may give a false high Brix of “C” massecuites at Enterprise mill. “C” massecuite samples reprocessed in Audubon (dilution in close container) gave an average Brix of 94 while the average Brix reported for the same period was 96. In addition, in comparing the “C” massecuite leaving the CVP with the “C” massecuite from the reheater a reduction in Brix of 1 point was found. Water or diluted molasses addition to the hot “C” massecuite leaving the pan will dissolve the crystals that were developed in the pan (Saska, 1999). It has to be evaluated what massecuite consistency can be handled by the crystallizers without reducing the Brix of the massecuite in the receiver. Design modifications of crystallizers 1 and 2 to increase crystallizers’ capacity will help to increase the massecuite consistency at the exit of the pan (crystallizers’ tracer test results estimated that the capacity of crystallizers 1 and 2 is reduced to about 60% of the nominal capacity).

5.1.2. Continuous Cooling Vertical Crystallizers

Nutsch and crystal size analysis for the individual crystallizers showed that the capacity of crystallizers 1 and 2, and also 3 and 4 are reduced. Significant differences in purity drop (2.6 points), crystal content (2.5 points) and crystal size (9 µm) between the old (1 and 2) and the new (3 and 4) crystallizers are a true proof of this problem. It is important to have a good temperature measurement to improve the cooling temperature control. More temperature probes with enough length located in places where the massecuite has movement or with a system that removes the massecuite collected around the probe have to be implemented for crystallizers.

The tracer test accomplished the goal of giving a glimpse of the flow pattern in crystallizers. Flow pattern problems because of channeling, axial dispersion and dead zones clearly appeared in the plots for zinc concentration versus time. On average, crystallizers 1 and 2 were working at 60% and the crystallizers 3 and 4 at 70% of their capacity. The flow distribution applied during the test apparently affected the retention time distribution for each crystallizer. Design and size of the
cooling coils, baffles position, stirring speed, flow direction, material consistency and flow rate are some of the most important factors that determine the flow pattern inside the crystallizer.

The retention time distribution curves showed by Figure 4.23 revealed also problems in the test design; for instance, the input of the massecuite mixed with the tracer to each crystallizer was not proportional to the flow distribution. It will be ideal to have more complete information about the variation with time of material quality, crystal content, crystal size distribution, consistency at the input and output of the crystallizer, more reliable temperature readings for different sections of the crystallizers, and temperatures and flow rate of cooling water. All this information could be used for a simulation of the performance of this crystallizer design predicting flow patterns, temperature profiles, crystal growth and crystal content (exhaustion) for different flow rates and massecuite consistencies.

The reduced capacity of the crystallizers and flow behavior in the pipes that transport the cold “C” massecuite to the reheater create a bottleneck for molasses exhaustion. Hence, the Brix of the massecuite had to be set in a lower set-point to avoid high consistencies that cannot be handled. The main point is to force the massecuite to flow through the crystallizers in plug flow giving a rapid cooling to an obtainable minimum temperature and giving enough retention time. The movement of the massecuite and the crystal surface area are fundamental to help the growth rate, since it makes a closer path for the sucrose in solution to attach to a crystal site.

5.1.3. Nutsch Molasses Consistency

The nutsch molasses consistency analysis at two different temperatures showed that the consistency of the nutsch molasses from the CVP is higher (777 Pa•s) than the consistency of the nutsch molasses from the reheater (371 Pa•s) at the same temperature (104°F). Indeed, the molasses from the reheater had lower Brix and lower purity (90.2 Brix, 32.6 purity) than the molasses from the CVP (92.3 Brix, 40.2 purity) as a result of molasses exhaustion in crystallizers. The highest predicted massecuite consistency was approximately 1,700 Pa•s at the average lower registered cooling temperature (115°F) and 3,150 Pa•s at the recommended temperature (104°F). The target purity formula used was developed limiting the molasses consistency to 300 Pa•s at 40°C (104°F) with an estimated massecuite consistency of 2,000 Pa•s. Rein (2007) states that the massecuite consistency can be as high as 5,000 Pa•s. But this limit will depend on the low-grade station’s equipment arrangement, design and size. It can be estimated that for the given conditions (Brix and
temperatures) during the 2008 season, the massecuite consistency did not reach values of 2,000 Pa•s (equivalent to 2,000,000 centipoises viscosity for a shear rate of 1s\(^{-1}\)). Consistency is a true constraint for molasses exhaustion and the best way to approach it is the quality of the material delivered to the factory. Other actions that may help to deal with high consistency at the low-grade station are: lower quantities and lower purities of the material sent to the low-grade station (improve exhaustions of the “A” and the “B” massecuites). Finally, a well-exhausted molasses in the “C” continuous vacuum pan will reduce the consistency of the mother liquor, so the massecuite consistency will be reduced.

### 5.1.4. Crystal Size Distribution

The grain fed to the “C” continuous vacuum pan (CVP) had variation of purity (63 to 67%) and crystal size (200 to 260 µm). A regulated proportion of grain with respect to the massecuite with a specified and homogeneous crystal size is the condition to obtain a “C” massecuite with the desired final crystal size distribution. A procedure for seed preparation, a larger grain pan and automation of the grain strike will have a significant effect on crystal size distribution and on a regulated supply to the continuous vacuum pan.

The Brix profile (supersaturation) in each compartment of the continuous vacuum pan has to be carefully controlled to avoid dilution or spontaneous nucleation. A compartment’s sampling of the continuous vacuum pan (CVP) performed by (Audubon Sugar Institute, 10/27/09) showed that the mean crystal size dropped from 255 µm in compartment 6 to 225 µm in compartment 9, and ended at 219 µm in the final compartment (12). Low crystal content favors spontaneous nucleation when the supersaturation is high (worst in the final compartments, 9 -12); consequently, the formation of small crystals decreased the mean crystal size. The recommendation at that time was to increase the seed/massecuite ratio to the continuous vacuum pan. Figure 5.3 and Figure 5.4 show the crystal size distribution analysis before and after increasing the seed/massecuite ratio. Crystal size analysis and nutsch analysis in steady state will give a better idea of the crystal content and crystal size profile along the continuous vacuum pan (CVP).
5.1.5. Supersaturation

Supersaturation is the driving force of the crystallization process but has a limit to avoid spontaneous nucleation. The evaluation of the non-sucrose/water ratio gave an average of 4.1 for the
massecuite from the continuous vacuum pan, but it was below 4 for the last 3 weeks of the sampling. The recommended value for this parameter is approximately 4 to 4.5. The supersaturation coefficient for the CVP nutsch molasses for the same last 3 weeks surpassed the safe boundary of 1.2. This high supersaturation also indicated that the molasses exhaustion at the pan was not enough (low crystal content) and the crystallization was outside the safe metastable zone. Nutsch molasses for the crystallizers and reheater massecuites show the same trend (gradual rise of the supersaturation) at the corresponding temperatures.

5.1.6. Nutsch Analysis and Goals for the Low-Grade Station

The biggest contribution from the nutsch analysis was recognizing the performance of each stage in terms of the purity drop and crystal content. The nutsch analysis, combined with the establishment of goals, helps to focus on the most representative problems that need to be solved for instance, molasses exhaustion at the pan and at the crystallizers. The most important goals are: increase crystal content in the pan up to 25 and 28% and increase the purity drop in crystallizers to 10 points.

The reheater operation was very stable. During the first 7 weeks of the season the “purity drop” was negative compared with the nutsch purity for crystallizers. For the last 3 weeks there was a rise ~0.5 certainly related to a higher grinding rate and higher “C” massecuite/tc ratio. In general, the performance of the reheater was good, but as with the crystallizers the temperature control is also an issue. The goal for the reheater is to keep the purity rise below or as high as zero.

Centrifuges performance was in general between the parameters. Purity rise was most of the time between 3 and 2. Higher purity rises were detected once for screen damage and once for low load. High purity rise was found once due to massecuite characteristics, crystal size distribution with left tail. In general, it can be said that the centrifugal capacity satisfied the volumetric load of massecuite during the 2008 season. The goal is to keep the purity rise around 1%.

Final molasses purity in centrifuges was slightly related to the “C” massecuite purity at the reheater, highlighting the importance on keeping the “C” massecuite purity in the lower workable level with crystal content around 25-28% (Ninela and Rajoo, 2006).

In summary, a good molasses exhaustion and hence a target purity difference close to zero can be achieved with the knowledge of the process obtained through periodic evaluation of several target parameters at the boiling house. As a reference, parameters obtained in other countries have
been used, but continued analysis can give the practical parameters that should be used for a particular location. The continuous evaluation also can show what practical changes have to be implemented to increase the sugar recovery in a profitable way.

### 5.2. Recommendations

According to the results, the practical recommendations to be implemented at Enterprise Mill or any other mill interested in molasses exhaustion are:

- Implement a periodic (one week frequency) sampling for nutsch test and crystal size distribution, comparing the results with achievable goals given by trusted sources or by history from successful past tests.
- Optimize molasses exhaustion in the continuous vacuum pan (CVP) by improving: seed/massecuite ratio and Brix profile - crystal content.
- Supply the proper amount and quality of grain to the CVP. Establish procedures for seed preparation and automation of the grain batch pan.
- Improve the internal design of crystallizers as well as the input to the crystallizers, to achieve the goal flow pattern – plug flow with radial dispersion.
- Increase the pipe size or reduce the turns on the pipe that conducts the cold massecuite from the crystallizer to the reheater.
- Locate more than three (3) places to install the temperature probes in crystallizers, considering the length and the wide of the probe and avoiding possible stagnant zones.
- Perform a tracer test to verify flow pattern and an estimated retention time for each crystallizer.
- Optimize the exhaustion of the “B” massecuite but most important of the “A” massecuite. It is required to increase crystal content.
- Reduce the delay time between cane harvesting and juice extraction.
REFERENCES


APPENDIX A. GENERAL FORMULATIONS

A.1. Mass Balance

SJM Formula

$\text{J} \rightarrow \text{M} \rightarrow \text{S}$

$w_{\text{sol},\text{J}} = w_{\text{sol},\text{S}} + w_{\text{sol},\text{M}}$  \hspace{1cm} (A-2)

$w_{\text{sol},\text{J}} = \text{Solids or Brix on juice} = 100 \text{ units}$

$w_{\text{sol},\text{S}} = \text{Solids or Brix on sugar} = x \text{ units}$

$w_{\text{sol},\text{M}} = \text{Solids or Brix on molasses} = (100 - x) \text{ units}$

A.1.1 Solids or Brix Balance:

Solids or Brix on juice = Solids or Brix on sugar + Solids or Brix on molasses

$w_{\text{sol},\text{J}} = w_{\text{sol},\text{S}} + w_{\text{sol},\text{M}}$  \hspace{1cm} (A-2)

\begin{align*}
\text{Recovered Sucrose (or Pol) on Sugar} &= R_s = \frac{P_s \cdot (P_J - P_M)}{P_J \cdot (P_s - P_M)} \cdot 100 = \text{SJM} \\
\end{align*}

\begin{align*}
P_J &= \text{Juice or Syrup Purity} \\
P_S &= \text{Sugar} \\
P_M &= \text{Molasses}
\end{align*}

A.1.2 Sucrose or Pol Balance:

Sucrose or Pol on juice = Sucrose or Pol on sugar + Sucrose or Pol on molasses.
\[ w_{\text{sol},J} \cdot P_J = w_{\text{sol},S} \cdot P_S + w_{\text{sol},M} \cdot P_M \] (A-3)

\[
100 \cdot P_J = x \cdot P_S + (100 - x) \cdot P_M
\]

\[
\text{Recovered Solids (or Brix) on Sugar} = x = \frac{(P_J - P_M)}{(P_S - P_M)} \cdot 100
\] (A-4)

### A.1.3 The Cobenze diagram:

The Cobenze diagram is mainly used to calculate the proportion or ratio between molasses and magma to produce a specified purity of massecuite.

For example, in the case of a 2nd strike, where “A” molasses (purity \( P_M = 68 \)) and magma (purity \( P_S = 80 \)), are going to be used to prepared 100 units of solids of “B” massecuite (purity \( P_J = 72 \)), the ratio of magma, molasses and massecuite is given by:

\[
P_S = 80 \quad P_J - P_M = 72 - 68 = 4 \rightarrow \text{Magma/B mass} = (4/12) \times 100 = 33
\]

\[
P_J = 72
\]

\[
P_M = 68 \quad P_S - P_J = 80 - 72 = 8 \rightarrow \text{Molasse/B mass} = (8/12) \times 100 = 67
\]

\[
\text{-----}
\]

\[
P_S - P_M = 80 - 68 = 12 \rightarrow \text{B mass. solids} = 100
\]
A.1.4 Boiling House Mass Balance:

These calculations assume that there are not losses or formation of sucrose and also that there are not losses or formation of non-sucrose through the crystallization stages. Birkett (1978), van der Poel et. al (1998) and Bubnik (1995) have described how the mass balance calculations are conducted (Rein, 2007).

Table A.1, shows a material balance for a three boiling scheme. According to Birkett (1978) the information highlighted has to be known or crystal yields for each strike can be established. The SJM formula and the Cobenze diagram are used to calculate the amounts of each stream.

For a basis amount of solids in syrups the recovered sugar and final molasses formation is calculated. The mass balance starts from the end of the process with the calculation of the “C” massecuite and then it continues with the other materials (From the final stages to the first stages).

Table A.1. Material balance for a three boiling scheme.

<table>
<thead>
<tr>
<th></th>
<th>Pol Units/hr</th>
<th>Solids Units/hr</th>
<th>Purity</th>
<th>Brix</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Syrup</strong></td>
<td>85</td>
<td>100</td>
<td>85.00</td>
<td>60.00</td>
</tr>
<tr>
<td><strong>“A” Strike</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C Magma</td>
<td>10.1</td>
<td>11.9</td>
<td>85.00</td>
<td>60.00</td>
</tr>
<tr>
<td>Syrup</td>
<td>77.4</td>
<td>91.0</td>
<td>85.00</td>
<td>60.00</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>87.5</td>
<td>103.0</td>
<td>85.00</td>
<td>62.41</td>
</tr>
<tr>
<td>A Massecuite</td>
<td>87.5</td>
<td>103.0</td>
<td>85.00</td>
<td>62.41</td>
</tr>
<tr>
<td>A Sugar</td>
<td>53.4</td>
<td>54.2</td>
<td>98.49</td>
<td>99.50</td>
</tr>
<tr>
<td>A Molasses</td>
<td>34.1</td>
<td>48.7</td>
<td>70.00</td>
<td>84.89</td>
</tr>
<tr>
<td><strong>“B” Strike</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C Magma</td>
<td>4.9</td>
<td>5.7</td>
<td>85.00</td>
<td>60.00</td>
</tr>
<tr>
<td>Syrup</td>
<td>7.6</td>
<td>8.9</td>
<td>85.00</td>
<td>60.00</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>40.8</td>
<td>55.2</td>
<td>74.00</td>
<td>66.01</td>
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<tr>
<td>B Massecuite</td>
<td>40.8</td>
<td>55.2</td>
<td>74.00</td>
<td>93.00</td>
</tr>
<tr>
<td>B Sugar</td>
<td>25.7</td>
<td>26.1</td>
<td>98.49</td>
<td>99.50</td>
</tr>
<tr>
<td>B Molasses</td>
<td>15.1</td>
<td>29.1</td>
<td>52.00</td>
<td>87.84</td>
</tr>
<tr>
<td><strong>“C” Strike</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grain</td>
<td>7.5</td>
<td>11.5</td>
<td>65.00</td>
<td>90.00</td>
</tr>
<tr>
<td>B Mol (dil)</td>
<td>13.4</td>
<td>25.9</td>
<td>52.00</td>
<td>65.00</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>20.9</td>
<td>37.4</td>
<td>56.00</td>
<td>71.07</td>
</tr>
<tr>
<td>C Massecuite</td>
<td>20.9</td>
<td>37.4</td>
<td>56.00</td>
<td>95.00</td>
</tr>
<tr>
<td>C Sugar</td>
<td>15.0</td>
<td>17.7</td>
<td>85.00</td>
<td>98.00</td>
</tr>
<tr>
<td>Final Molasses</td>
<td>5.9</td>
<td>19.7</td>
<td>80.00</td>
<td>92.46</td>
</tr>
<tr>
<td><strong>Grain Strike</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A Mol (dil)</td>
<td>5.8</td>
<td>8.3</td>
<td>70.00</td>
<td>65.00</td>
</tr>
<tr>
<td>B Mol (dil)</td>
<td>1.7</td>
<td>3.2</td>
<td>52.00</td>
<td>65.00</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>7.5</td>
<td>11.5</td>
<td>65.00</td>
<td>65.00</td>
</tr>
<tr>
<td>Grain</td>
<td>7.5</td>
<td>11.5</td>
<td>65.00</td>
<td>90.00</td>
</tr>
</tbody>
</table>
A.2. Boiling House Performance

Crystal Content: It is a limiting factor since a massecuete with very high crystal content become at solid; therefore, the crystallization is conducted to the point where the crystal content gives the maximum allowable viscosity to guarantee flow ability (Rein, 2007).

\[
\text{Crystal content ~} % \text{ massecuete} = w_{Cr,Ma} = \frac{w_{DS,Ma} \cdot (P_{Ma} - P_{Mol})}{(100 - P_{Mol})} \cdot 100
\]  

(A-5)

\[
\text{Crystal content ~} % \text{ solids (or Brix)} = w_{Cr,DS} = \frac{(P_{Ma} - P_{Mol})}{(100 - P_{Mol})} \cdot 100
\]  

(A-6)

In South Africa, the goal crystal content ~% solids for A and B strikes is estimated with the following equation (Rein, 2007).

\[
\text{Crystal content ~} % \text{ solids (A ~& ~ B strikes)} = w_{Cr,DS} = 0.78 \cdot P_{Ma} - 10
\]  

(A-7)

Exhaustion: It expresses how much of the sucrose in the massecuete become crystal with 100% purity (Rein, 2007).

\[
\text{Exhaustion} = \frac{(P_{Ma} - P_{Mol})}{P_{Ma} \cdot (100 - P_{Mol})} \cdot 100
\]  

(A-8)

High exhaustion (high crystal content) in the “A” strikes is one of the most important goals to achieve in the boiling house since it reduces massecuete quantities (increases pan and crystallizers capacity) and reduces recirculation avoiding also sucrose losses due to degradation. Again in South Africa the goal exhaustion for “A” massecuete is estimated by the following equation (Rein, 2007).

\[
\text{Exhaustion (A massecuete goal)} = 0.775 \cdot P_{Ma}
\]  

(A-9)
**Purity Drop:** The purity drop for massecuites has been used as a rapid and easy way to evaluate how well exhausted is the massecuite.

\[ PD = P_{Ma} - P_{Mol} \]  

(A-10)

**A.3. Goals for the Low Grade Station**

**Goal – Final Molasses Apparent Purity** \( P_{App, fm, Goal} \): Determined from Target Purity and Pol/Sucrose ratio.

\[ P_{App, fm, Goal} = \left( \frac{Pol}{Sucrose} \right) \cdot T_{arg \ et \ Purity} \]  

(A-11)

**Goal – Nutsch Pan Apparent Purity** \( P_{App, NP, Goal} \): Calculated from the \( P_{App, fm, Goal} \) and the nutsch molasses purity drop from pan to centrifuges

\[ P_{App, NP, Goal} = P_{App, fm, Goal} + ( P_{NP} - P_{App, fm, Goal} ) \]  

(A-12)

**Goal – C Massecuite Apparent Purity** \( P_{App, CM, Goal} \): Calculated from the \( P_{App, NP, Goal} \) and a target crystal content of 28 \% for C-massecuite leaving the pan.

\[ P_{App, CM, Goal} = 28 + 0.72 \cdot P_{App, NP, Goal} \]  

(A-13)

**A.4. Molasses Survey Equations:**

**True solids (TS):** The Total of solids concentration (dry substance) in final molasses is determined applying a statistical model developed at Audubon Sugar Institute.
True Solids = \( w_{DS} = 0.5345 + 0.9519 \cdot Brix + 0.1461 \cdot Cond.Ash + 0.0347 \cdot (Fruct + Gluc) \)  \( (A-14) \)

**Pol/Sucrose ratio:** The pol/sucrose ratio expresses how close the pol reading is to the actual sucrose concentration (True Sucrose).

**True Purity and Apparent Purity:** Expresses how much sucrose (Pol) is contained in the total dissolved solids (Brix or RDS).

\[
True \ Purity = P_{True} = \frac{True \ Sucrose}{True \ Solids} \cdot 100
\]  \( (A-15) \)

\[
Apparent \ Purity = P_{App} = \frac{Pol}{Brix} \cdot 100
\]  \( (A-16) \)

**Target Purity:** It is the minimum final molasses purity (equilibrium purity -saturation) that can be obtained a laboratory level, taking into account a workable maximum molasses consistency of 300 Pa-s at 40 °C (Rein, 2007).

\[
Target \ Purity = P_{target} = 33.9 - 13.4 \cdot \log \left( \frac{G + F}{Ash} \right)
\]  \( (A-17) \)

\( G + F = RS = \text{Glucose plus Fructose concentration by HPLC} \)

\( Ash = A = \text{Conductivity Ash} \)

**Target Purity Difference (TPD):** Difference between True Purity and Target purity.

\[
TPD = True \ Purity - Target \ Purity
\]  \( (A-18) \)

A.5. **Solubility and Supersaturation Equations:**

Charles (1960) equation to represent the solubility of sucrose for a range of temperatures from 0 to 90 °C in pure sucrose solution is: (Rein, 2007)
\[ w_{S,Sat} = 64.397 + 0.07251 * t + 0.002057 * t^2 - 9.035 * 10^{-8} * t^3 \]

\[ t \text{ : temperature } ^\circ C \]

Saturation Coefficient \( q_{Sat,p} = \left( \frac{w_S}{w_W} \right)_{Sat,p} = \frac{w_{S,Sat}}{100 - w_{S,Sat}} \) \hspace{1cm} (A-20)

\[ q_{Sat,p} = g \text{ sucrose} / g \text{ water at Saturation in pure solution} \]

The Solubility Coefficient (SC) to express the ratio of sucrose concentration at saturation between the impure and the pure sucrose solutions is calculated by:

\[ So\ lub\ ility\ Coefficient = SC = \frac{q_{Sat,i}}{q_{Sat,p}} \] \hspace{1cm} (A-21)

\[ q_{Sat,i} = g \text{ sucrose} / g \text{ water at Saturation in impure solution} \]

Lionnet and Rein (1980) give the following correlation \( r^2=0.85 \):

\[ SC = \frac{q_{Sat,i}}{q_{Sat,p}} = 0.742 + 0.182 \cdot \frac{NS}{W} - 0.346 \cdot \frac{RS}{A} \] \hspace{1cm} (A-22)

\[ \frac{NS}{W} = non - sucrose \text{ / water ratio} \]

\[ \frac{RS}{A} = Reducing \ Sugars \text{ / Ash ratio} \]

Supersaturation Coefficient \( y \) is the degree of supersaturation obtained at determined conditions of temperature and purity and is determined by the ratio of the sucrose/water ratio at supersaturation and the sucrose/water ratio at saturation.

\[ \text{Supersaturation Coefficient} = y = \frac{q_{Supersat}}{q_{Sat}} \] \hspace{1cm} (A-23)
Since all these calculations have to be done with true values, when only Brix (RDS) and pol are known, the following conversions have to be applied (Rein, 2007):

\[
\text{True Solids} = w_{DS} = \text{Brix} \cdot [1 - 0.00066 (\text{Brix} - \text{pol})] \tag{A-24}
\]

\[
\text{True Sucrose} = w_S = \text{Brix} \cdot 0.874 \cdot (\text{Brix} - \text{pol}) \tag{A-25}
\]

\[
\text{Non-sucrose/water ratio} = \frac{NS}{W} = \frac{w_{DS,\text{Ma}} \cdot (1 - P_{\text{True,\text{Ma}}} / 100)}{(100 - w_{DS,\text{Ma}})} \tag{A-26}
\]

**Seed/Massecuite ratio**: For batch pans the proportion between the seed and the final volume of the massecuite is approximately 33 %, obtained rising the level of the seed in the pan 1” (inch) above the calandria. The flow rate of seed to the continuous pan has to be regulated in order to obtain a determined crystal size (normally the percentage of seed respect to the final massecuite range from 22 to 33% and it can be estimated with the following equation (Rein, 2007).

\[
\dot{m}_{\text{Seed}} = \dot{m}_{\text{Ma}} \cdot \frac{w_{\text{Cr,\text{Ma}}}}{w_{\text{Cr,\text{Seed}}}} \left(\frac{d_{\text{Seed}}}{d_{\text{Ma}}}\right)^3 \tag{A-27}
\]

<table>
<thead>
<tr>
<th>Pan Boiling</th>
<th>$\dot{m}<em>{\text{Seed}}/\dot{m}</em>{\text{Ma}}$</th>
<th>$w_{\text{Cr,\text{Ma}}}$</th>
<th>$w_{\text{Cr,\text{Seed}}}$</th>
<th>$d_{\text{Seed}}/d_{\text{Ma}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A Pan</td>
<td>0.25</td>
<td>50</td>
<td>40</td>
<td>0.58</td>
</tr>
<tr>
<td>B Pan</td>
<td>0.33</td>
<td>40</td>
<td>35</td>
<td>0.66</td>
</tr>
<tr>
<td>C Pan</td>
<td>0.40</td>
<td>28</td>
<td>22</td>
<td>0.68</td>
</tr>
</tbody>
</table>

**Table A.2.** Seed/Massecuite ratio with equation (A-26) (Rein, 2007)
## APPENDIX B. TOTAL COLLECTED DATA

### Table B.1. Data collected from Enterprise weekly report and Audubon Sugar Institute molasses survey

<table>
<thead>
<tr>
<th>Material</th>
<th>Parameter</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>To 12/20/08</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cane Sugar</td>
<td>TC/hour</td>
<td>745.23</td>
<td>820.63</td>
<td>812.19</td>
<td>824.93</td>
<td>841.68</td>
<td>841.54</td>
<td>873.87</td>
<td>928.29</td>
<td>987.63</td>
<td>977.57</td>
<td>866.96</td>
</tr>
<tr>
<td>Cane Sugar</td>
<td>Yield, 96°Sg%</td>
<td>10.02</td>
<td>10.71</td>
<td>11.51</td>
<td>11.07</td>
<td>10.79</td>
<td>11.18</td>
<td>10.74</td>
<td>10.64</td>
<td>10.52</td>
<td>10.31</td>
<td>10.70</td>
</tr>
<tr>
<td>F.Molass</td>
<td>EP App.Pty</td>
<td>36.1</td>
<td>33.7</td>
<td>32.9</td>
<td>32.9</td>
<td>33.3</td>
<td>34.4</td>
<td>34.7</td>
<td>34.1</td>
<td>33.0</td>
<td>34.3</td>
<td>34.1</td>
</tr>
<tr>
<td>F.Molass</td>
<td>ASI App.Pty</td>
<td>34.5</td>
<td>32.0</td>
<td>32.6</td>
<td>33.9</td>
<td>33.7</td>
<td>34.3</td>
<td>34.5</td>
<td>33.8</td>
<td>33.8</td>
<td>35.0</td>
<td>33.8</td>
</tr>
<tr>
<td>F.Molass</td>
<td>ASI True.Pty</td>
<td>43.2</td>
<td>40.6</td>
<td>41.2</td>
<td>41.9</td>
<td>41.0</td>
<td>42.3</td>
<td>43.4</td>
<td>42.3</td>
<td>42.4</td>
<td>44.3</td>
<td>42.3</td>
</tr>
<tr>
<td>F.Molass</td>
<td>ASI Pol/Suc</td>
<td>0.81</td>
<td>0.80</td>
<td>0.80</td>
<td>0.82</td>
<td>0.83</td>
<td>0.82</td>
<td>0.80</td>
<td>0.81</td>
<td>0.81</td>
<td>0.80</td>
<td>0.81</td>
</tr>
<tr>
<td>F.Molass</td>
<td>ASI (F+G)/Ash</td>
<td>1.02</td>
<td>0.92</td>
<td>0.84</td>
<td>0.83</td>
<td>0.75</td>
<td>0.71</td>
<td>0.72</td>
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<td>0.65</td>
<td>0.67</td>
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<td>F.Molass</td>
<td>ASI Target Pty</td>
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<td>34.4</td>
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<td>ASI TPD</td>
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<td>6.3</td>
<td>6.9</td>
<td>5.4</td>
<td>6.4</td>
<td>7.6</td>
<td>6.2</td>
<td>6.0</td>
<td>8.1</td>
<td>6.9</td>
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<td>F.Molass</td>
<td>gln/TC @79.5%</td>
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<td>6.7</td>
<td>6.7</td>
<td>6.3</td>
<td>5.9</td>
<td>5.7</td>
<td>5.7</td>
<td>5.5</td>
<td>5.4</td>
<td>5.7</td>
<td>6.0</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>EP App.Pty</td>
<td>84.0</td>
<td>83.6</td>
<td>84.4</td>
<td>84.8</td>
<td>85.4</td>
<td>86.2</td>
<td>86.2</td>
<td>86.1</td>
<td>85.7</td>
<td>85.8</td>
<td>85.8</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>ASI App.Pty</td>
<td>80.7</td>
<td>84.1</td>
<td>84.8</td>
<td>84.8</td>
<td>85.4</td>
<td>86.9</td>
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<td>86.0</td>
<td>85.1</td>
<td>86.6</td>
<td>85.0</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>ASI True.Pty</td>
<td>84.2</td>
<td>88.8</td>
<td>88.7</td>
<td>87.5</td>
<td>88.9</td>
<td>90.3</td>
<td>90.3</td>
<td>91.2</td>
<td>89.5</td>
<td>91.1</td>
<td>89.1</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>ASI (F+G)/Ash</td>
<td>0.97</td>
<td>0.98</td>
<td>0.97</td>
<td>1.24</td>
<td>0.95</td>
<td>0.74</td>
<td>0.82</td>
<td>0.74</td>
<td>0.79</td>
<td>0.78</td>
<td>0.90</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>A Mass App.Pty</td>
<td>84.3</td>
<td>83.5</td>
<td>84.4</td>
<td>85.0</td>
<td>85.6</td>
<td>86.5</td>
<td>86.6</td>
<td>86.5</td>
<td>86.2</td>
<td>86.4</td>
<td>85.5</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>Pty Drop</td>
<td>-17.2</td>
<td>-17.7</td>
<td>-18.3</td>
<td>-17.8</td>
<td>-17.8</td>
<td>-17.3</td>
<td>-16.3</td>
<td>-17.0</td>
<td>-16.0</td>
<td>-15.6</td>
<td>-17.1</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>%Crystal (DS)</td>
<td>52.2</td>
<td>51.8</td>
<td>53.8</td>
<td>54.3</td>
<td>55.1</td>
<td>56.2</td>
<td>54.9</td>
<td>55.7</td>
<td>53.7</td>
<td>53.4</td>
<td>54.2</td>
</tr>
<tr>
<td>Sugar Syrup</td>
<td>ft³/°C</td>
<td>4.1</td>
<td>4.6</td>
<td>4.7</td>
<td>4.5</td>
<td>4.3</td>
<td>4.3</td>
<td>4.3</td>
<td>4.2</td>
<td>4.3</td>
<td>4.2</td>
<td>4.3</td>
</tr>
<tr>
<td>B Mass App.Pty</td>
<td>%Crystal (DS)</td>
<td>39.3</td>
<td>37.4</td>
<td>37.1</td>
<td>38.4</td>
<td>40.5</td>
<td>42.0</td>
<td>44.4</td>
<td>41.3</td>
<td>41.7</td>
<td>42.5</td>
<td>40.6</td>
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Table B.2. Continuous cooling vertical crystallizers. Tracer experiment results (Zinc)

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<th>Crystall. #3</th>
<th>Crystall. #4</th>
<th>Output</th>
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Table B.3. Crystal Size and distribution by stage

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<th>C-Mass Reheater</th>
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<td>MA CV d 50%</td>
<td>d 10% MA CV d 50%</td>
<td>d 10%</td>
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<td>258 49%</td>
<td>260 44%</td>
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<td>240 52%</td>
<td>258 49%</td>
<td>260 44%</td>
<td>231</td>
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<td>11/17/2008</td>
<td>234 49%</td>
<td>249 49%</td>
<td>243 44%</td>
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<td>225 51%</td>
<td>231 45%</td>
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<td>225 50%</td>
<td>231 45%</td>
<td>244</td>
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<td>201 59%</td>
<td>225 59%</td>
<td>231 45%</td>
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<td>250 49%</td>
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<td>208 52%</td>
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<td>244</td>
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<td>Average</td>
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<td>208 52%</td>
<td>231 45%</td>
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### Table B.4. Nutsch molasses consistency at 104 °F and at 140 °F (continuous vacuum pan – CVP and reheater)

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<th>Continuous Vacuum Pan - CVP</th>
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<td>40 °C (104 °F)</td>
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<td>Consistency</td>
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<td>0.78 612</td>
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<td>11/16/2008</td>
<td>94.7 54.3 92.6 40.6 23.0</td>
<td>0.68 1201</td>
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<td>11/23/2008</td>
<td>94.1 53.4 91.9 40.3 21.9</td>
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<td>0.74 1100</td>
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### Table B.5. Nutsch molasses non-sucrose/water ratio, solubility coefficient and supersaturation at 160 °F (CVP), at 120 °F (reheater inlet) and, at 137 °F (reheater output)

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<td>Saturation</td>
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<td>4.2 0.75 4.79 1.24</td>
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### Table B.6. “C” massecuite and nutsch molasses results

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VITA

Luz Stella Polanco is from Cali, Colombia. Luz Stella has been working at Audubon Sugar Institute – LSU AgCenter as a Research Associate since 2003, and previously she worked eight years for a Colombian sugarcane factory as a chief chemist. She has a bachelor’s degree in chemical engineering from “Universidad del Valle” (Cali-Colombia). Luz Stella began her studies at Louisiana State University as a part-time student in August 2005. Her technical interest is to continue researching on sugar manufacturing or on the manufacturing of sugar related products.