ADDIS ABABA UNIVERSITY
SCHOOL OF GRADUATE STUDIES
ADDIS ABABA INSTITUTE OF TECHNOLOGY
DEPARTMENT OF CHEMICAL ENGINEERING

Process Optimization to Reduce Sucrose Loss with Final Molasses: The case of Metahara Sugar Factory

By

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A Thesis Submitted to the School of Graduate Studies of Addis Ababa University in Partial Fulfillment of the Requirements for the Degree of Master of Science in Chemical Engineering
(Food Engineering)

Advisor Eng. Teshome Worku (Ass. Proff.)

July, 2011
Addis Ababa

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Declaration

I, the under signed, declare that this thesis is my original work and has not been presented for a degree in any other university and that all sources of materials used for the thesis have been duly acknowledged.

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Acknowledgements

I want to express my sincere gratitude to my advisor Eng. Teshome Worku for his advice, support, and guidance during the course this thesis work. I also like to forward my great thanks to Eng. Gizachew Shiferaw for his interest on my thesis and unreserved assistance starting from topic selection till the completion of this project.

The Chemical engineering Department ‘Industry, Material and Bio-Chemical’ research group members are greatly acknowledged for giving me fruitful comments for improvement of my project work during progress reports. Here special thanks go to Professor W.Sokol for his great concern and improvement ideas to my project. I also appreciate Dr. Eng. Shimelis Admassu and all staffs of the chemical engineering department for their unreserved assistance and sharing of knowledge during my Post Graduate Study; I learned much from them during my study at the University. All colleagues and friends of the members of chemical engineering department are greatly thanked for the cheerful working atmosphere.

I also would like to thank the management of MSF for giving me the chance to attend my MSc. Program. It is my greatest pleasure to thank Ato Lemma Gurmu, former FLM of MSF. Without his effort, the chance couldn’t have been successful. The Cooperation of MSF Quality control Department, for my laboratory experiment, is gratefully acknowledged. Here my special thanks go to Ato Bekele Mirkena, MSF Quality Control Head, for his assistance in facilitating all necessary conditions for the Laboratory analysis and for his great material and moral support during all the research time.

Table of Contents

Lists of Tables

Tables **Title** **Page**

Lists of Figures

List of Symbols and Acronyms

Acronym	Nomenclature
AAW sugar	A-after worker sugar (2nd cured A-sugar)
AFW sugar	A-Fore worker sugar (1st cured A-sugar)
A-massecuite	First grade boiled massecuite (high grade massecuite)
CC	crystal content
C-massecuite	2nd grade boiled massecute
COMESA	Common Market for Eastern and Southern Africa
DAW sugar	D-after worker sugar 2nd cured D-sugar)
DFW sugar	D-fore worker sugar (1st cured D-sugar)
D-massecuite	Last massecuite (low grade massecuite)
ICUMSA	International Commission for Uniform Method of Sugar Analysis
K_s	Solubility coefficient
KSS	Supersaturation coefficient
Mass.	Massecuite (mixture of sugar crystal and mother liquor
MSF	Metahara Sugar Factory
RC	Crystal growth rate
RSM	Response Surface Methodology
S	Solubility of sucrose (g sucrose/g water)

List of Appendices

Abstract

In this study, process Optimization has been applied to D-massecuite boiling, cooling crystallization, reheating and separation processes in MSF to minimize the high sucrose loss with final molasses. Mother liquor purity and Purity drop were taken as response variables to measure the performance of each process towards minimization of final molasses purity. Design Expert-Response surface methodology was applied to statistically evaluate the data and to solve optimum values factors and response variables. Economical advantages of the process optimizations were compared with the factory's existing working norms. D- massecuite boiling optimization has resulted in nutsch purity reduction of 0.53 units. Similarly, an increase in purity drop of 0.80 (from 4.21 to 5.01) across cooling crystallizers was obtained at optimum cooling time of 17.36h and temperature to 45^{0}C for the existing crystallizer's capacity. The cooling experimental results have indicated that a purity drop of 6.37 units can be achieved if crystallizer capacity of MSF increased so as to give cooling time of 34.5h and temperature of 44^{0}C. For the existing reheaters, the optimum reheating temperature was found to be 52.5^{0}C which gave a purity rise across reheaters of 0.53^0 against the value of 0.97 before optimization and the optimum massecuite flow rate of 19t/h. From the centrifugal separation optimization result, the optimum spray water was found to be 6.3% massecuite giving a purity rise of 2.44 across centrifugal machines and DFW magma purity of 84.0. The overall effect of process optimization was a reduction in final molasses purity by 1.43 units (from 34.86 to 33.43) for the existing capacity of cooling crystallizer which leads to annual saving of 1040.5 tons sugar, equivalent to 13,526,919 Birr. Additional saving of 12,334825birr/year is expected if enough crystallizer capacity is installed for MSF.

Key words: *Final Molasses, Sucrose Loss, Purity, Purity Drop, Exhaustion, Response Surface Methodology*

1. Introduction

1.1 Background

Sugar industry is one of the biggest agro-industries in Ethiopia and one of the most competitive sectors for the national economy. The country has suitable weather conditions at different areas for cane plantation and sugar production. There are three sugar factories with total production of about 3000, 000 tons of plantation white sugar per year. Now days there are a number of projects undertaking in Ethiopia to expand the existing and to install new sugar factories (Metahara Sugar Factory Strategic Plan Manual, 2008).

The main goal of sugar cane factory is to have an efficient and profitable operation with the required sugar quality and maximum sugar recovery. One of the biggest problems in sugar industries is the loss of sucrose in different forms. The monetary value of the losses of sucrose is of extreme importance because of the direct impact on profitability. One of the biggest problems in the sugar industry is the loss of sucrose in different forms. Generally two types of sucrose losses are existing in sugar factories, namely, known or determined and unknown or undetermined losses. The known loss consists of losses of sucrose with by-products: sucrose loss in bagasse, sucrose loss in filter cake, and sucrose loss in molasses. Undetermined sucrose loss includes chemical (inversion), biological and mechanical losses (i.e. sucrose that is unaccounted for when completing a mass balance over the mill). All of these components need to be minimized to maximize sugar recovery. The sucrose loss in molasses is particularly important because it is normally by far the largest of the four components of total loss (RASITC, 2004)

At present in Metahara Sugar Factory (MSF), 15-16% of sucrose incoming with cane is lost; 9.1 – 9.3 % with final molasses, 5.2-5.5% with bagasse, 0.45 -0.65 % with filter cake and 0.4 – 0.45% undetermined loss. The loss with final molasses is the largest and accounts for about 57-62% of all losses. This loss has a direct and significant impact on the recovery and profitability of the factory. During the production year 2009/2010, the loss of sucrose in Metahara Sugar Factory is as shown in figure1.1. Of the total 1,427,053 Qts of sucrose supplied to the factory with cane, 130,718 Qts was lost with final molasses. The overall recovery was only 84% which is low and needs to be improved. By minimizing loss in final molasses only from 9.16% to 9.0%, it is possible to save 0.16x1427053/100 = 2283.3 Qts of sucrose. In monetary terms, it is a saving of more than three million Eth. Birr per annum. According to practice from other sugar factories it may be possible to reduce this loss to about 8.8%.

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. Although sucrose lost in molasses is still available as a saleable product (as part of final molasses) the price of sucrose sold as final molasses is lower than the price of sucrose sold as product sugar (Saska. M S. L. Goudeau and F.M. Beyene, 2010). In Metahara sugar Factory, utilization of molasses for Ethanol production has been started since the December of 2010. One kg of sucrose will be converted into about ½ kg of ethanol and the price of sugar is higher than that of ethanol. Hence it is more beneficial to exhaust maximum possible amount sucrose from final molasses before leaving sugar factory. Only around 45% total fermentable sugar in molasses is sufficed for ethanol production. (Total = sucrose +glucose +fructose).

Final molasses is usually produced at about 3.5 to 4% on cane by weight. In order to minimize the loss of sugar in the final molasses it is essential to consider the factors responsible for the varying degrees of molasses exhaustibility (D.P. Kulkarni, 2005).

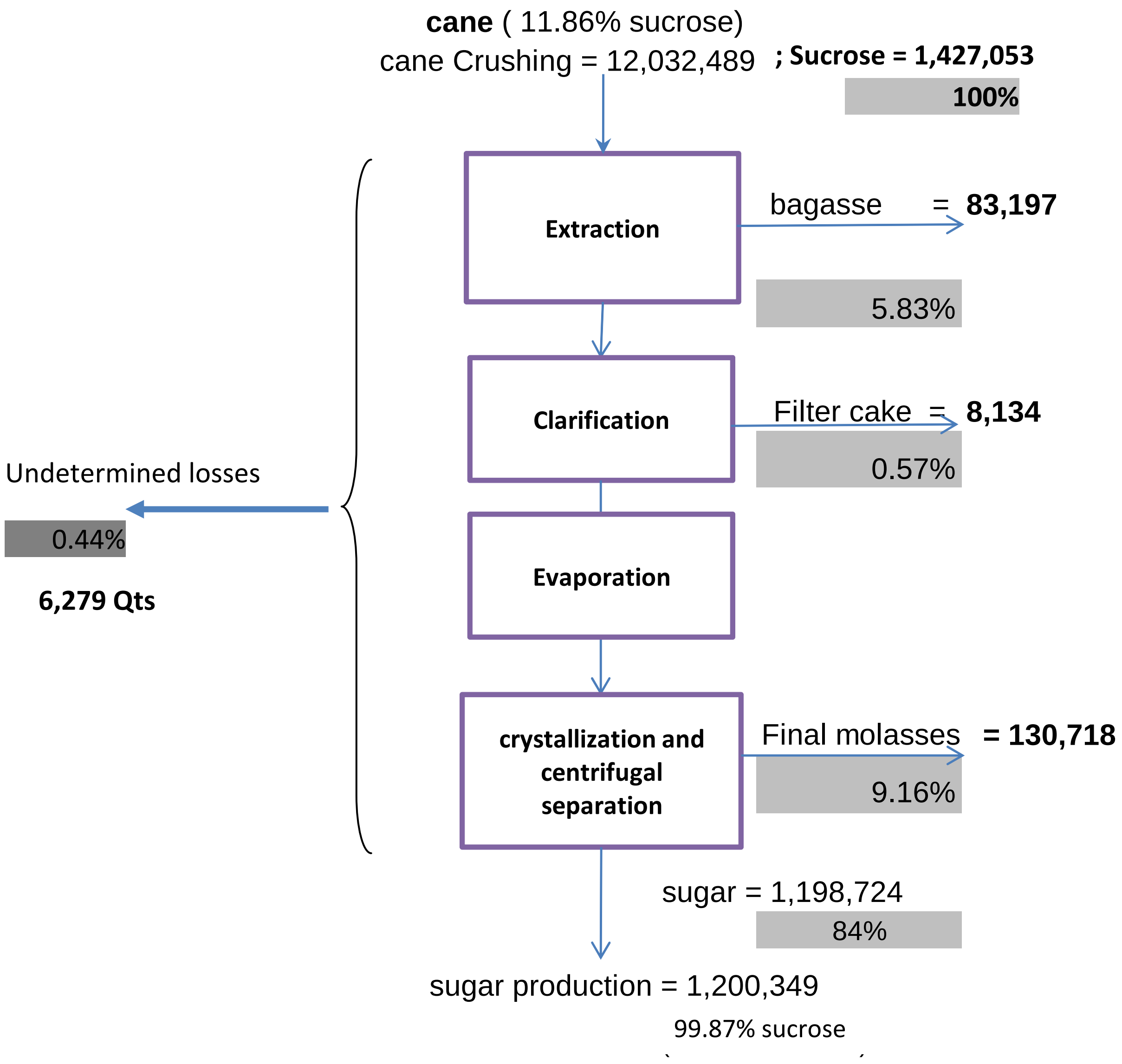

Figure 1.1: Sucrose balance for MSF during 2009/2010 production year (Qts)

The problem of molasses exhaustion and the factors influencing the same can be considered from two angles viz. compositional and operational factors.

The compositional factors depend on the quality of cane and juice clarification efficiency. The non-sugars influence the molasses exhaustion in two ways; one is the retention of sucrose i.e. solubility of sucrose and second is the effect on rate of crystallization. In general the inorganic salts increase the solubility of sucrose while the reducing sugars bring down the retention of sucrose in molasses. By far the most important factor determining the exhaustion potential of molasses is the reducing sugar/ ash ratio. The higher this ratio, lower should be loss of sucrose in molasses. This ratio should be 1.0 or more for good exhaustion of final molasses (D.P. Kulkarni, 2005).

The operational factors, also called controllable factors, affecting exhaustibility of final molasses are the practices and operations mainly at the low grade massecuite processing, i.e, pan boiling, cooling crystallization, reheating and centrifugal separation of the last massecuite.

The purity of molasses (sucrose %total solid in molasses) is a very important process variable, given that it is a measure of sugar content in molasses and therefore of the factories sugar loss; the higher the purity, the higher this loss is.

During 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 massecuite and the purity of the molasses (purity drop) is related to the massecuite exhaustion. In general, the purity drop (PD) can be used to determine how efficiently each process is operating towards molasses exhaustion because the higher the PD, the higher is the efficiency; the more sugar crystallization in the pan and crystallizers and the lower is the purity of the mother liquor throughout the crystallization stages and, consequently, the lower is the final molasses purity. At massecuite boiling, Purity drop (PD) is defined as the purity difference between massecuite and its mother liquor separated from the massecuite. For cooling crystallizer, reheating and centrifugal separation, it is the difference between purity of mother liquor at inlet and outlet of the units. The value will be positive for cooling, and usually negative for reheating and centrifugal separation. The opposite is purity rise (RASITC, 2004).

Although the A- and C- massecuite boiling and processing have their own indirect effect on final molasses exhaustion, the D-massecuite boiling and processing has a direct and immediate effect on molasses exhaustion. In the case of final stage of crystallization (D-massecuite), the entire system of pan boiling, crystallizer operation, reheating and centrifugal working has to be given special attention since the mother liquor from this system will be take out of the process in the form of final molasses and in view of the fact that 8-10% sucrose in cane is lost in the final stage of crystallization (with final molasses) (*D.P. Kulkarni, 2005*); i.e. this final stage of the process determines the degree of exhaustion of the final molasses.

In this thesis the approach to molasses exhaustion for the studied sugar factory will mainly focus on the processes at low-grade (D-massecuite) station which include:

1) Pan boiling 2) continuous cooling crystallizers 3) reheating condition and 4) centrifugal separator.

1.2 Statement of the Problem

For the sugar factory to be competitive and profitable, more attention must be given to overall recovery of sugar. The cost of production of sugar is increasing from time to time due to escalating cost of spare parts and chemicals. The establishment of new sugar factories in Ethiopia and the globalization issue, including the COMESA agreement, may result in high competition among sugar factories. Therefore, the existing Sugar factories need to improve their internal efficiency, particularly the recovery of sucrose, to become competitive, profitable and to assure sustainability.

One of the biggest problems in the sugar industries which challenge their profitability is the loss of sucrose with final molasses. At present, 9.1 to 9.3% of the sugar that enters the factory with the cane is lost with final molasses in Metahara Sugar Factory (MSF). This is a big loss which should be minimized. The company is losing sugar that is expected to be recovered and increase the efficiency of sugar factory.

The causes of the high sucrose loss with final molasses are not clearly identified for MSF. The aim of this thesis is therefore to identify the most significant loss areas and apply optimization techniques to minimize the losses.

1.3 Objectives

1.3.1 General Objective

The main objective of this thesis was to investigate and optimize the process conditions at low grade boiling, crystallization and centrifugal plant, to reduce sucrose loss with final molasses.

1.3.2 Specific objectives

The specific objectives of the thesis were to:

- assess the purity drop profile at boiling, cooling, reheating and centrifugal separation for the last massecuite (D-massecuite)
- optimize D-massecuite boiling parameters (seed amount, final brix and purity) to obtain maximum purity drop between the massecuite and its mother liquor

- study the effect of cooling and reheating temperatures as well as cooling time on purity drop and determine optimum conditions based on viscosity (massecuite flow rate) and purity drop
- study purity rise across centrifugal machines and optimize spray water amount based on final molasses purity and D fore worker sugar purity
- evaluate the effect of the optimization on the techno-economic gain of the factory performance

1.4 Significance of the Project

Since sucrose loss with final molasses is normally the largest component of the total sucrose losses, its minimization is vital to maximizing sucrose recovery and profitability of sugar factory. Based on this fact this thesis is believed to contribute its part in minimizing loss of sucrose with final molasses in Metahara Sugar Factory by identifying the most important causes and optimizing process conditions for the final massecuite processing. Determination of the optimum operational conditions to improve exhaustibility of final molasses should be the first step as it is low cost option compared to the other alternatives like modification of equipments and investment on technology or change of equipment.

1.5 Frame work of the experiment

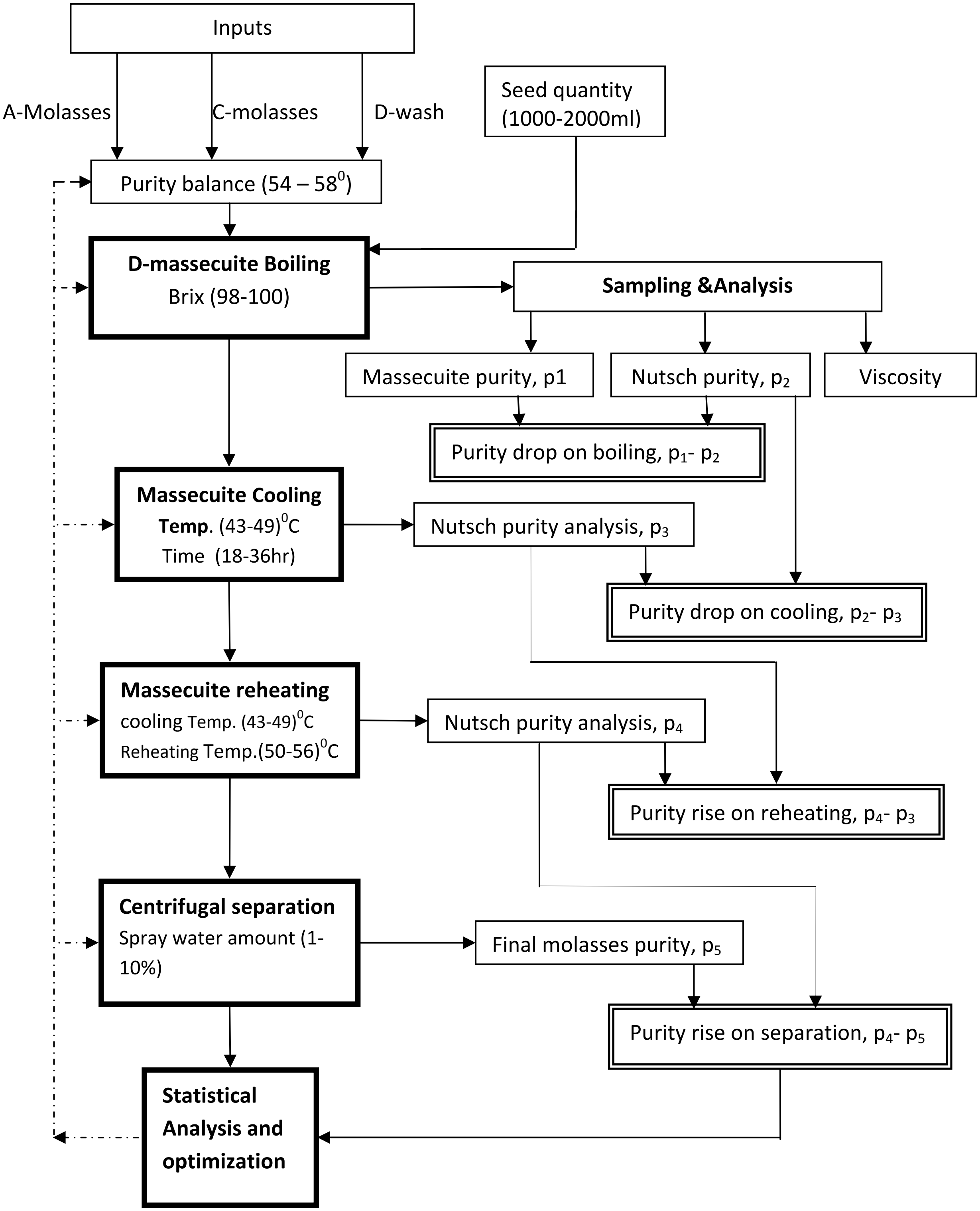

Figure 1.2: Framework of the experiment

2. Literature Review

2.1 Over View of Sugar Manufacturing Process from Sugar Cane

More than 60% of the world's sugar production is from sugar cane; the balance is from sugar beet. Sugarcane contains not only sucrose but also numerous other dissolved substances, as well as cellulose or woody fiber (D.P. Kulkarni, 2005)

Sugarcane is generally about 70% water, 15% fibrous material and the remaining is dissolved solids. The major composition of sugar cane is given below:

- Sugarcane: 100%
- Water: 70 to 7 3%
- Fiber: 12 to 15%
- Sugar (sucrose): 11 to 14%
- Dissolved non-sugar solids: 2 to 3%

In extreme cases there would be even wider variations, depending upon the age and varieties of cane and also the climatic factors (like temperatures, humidity) soil condition agricultural practices followed, etc,

Sugar production from sugarcane uses several processes and unit operations this process is summarized for plantation white sugar production in figure 1. The following are the main unit operations involved in manufacturing sugar from sugarcane:

1. Cane preparation
2. Extracting of juice from sugarcane
3. Clarification of cane juice
4. Evaporation of water
5. Crystallization in vacuum pans
6. Crystallization in motion (by cooling in crystallizers)
7. Centrifuging (separation of mother liquor from crystal sugar)
8. Sugar drying, grading , packing and storing

Cane preparation*:* is cutting and disintegration of cane stack in to small pieces of cane fibers by using cane knives and shredders. Cane preparation is done to facilitate the extraction process by increasing surface area to volume ratio and exposing the juice containing cells for mechanical compression or leaching.

Juice Extraction System*:* The juice is extracted from the cane either by milling, in which the cane is pressed between the heavy rolls, or by diffusion, in which the sugar is leached out with water. The sugar cane bagasse produced at the extraction is delivered to the boiler plant, where it is used as fuel, producing the steam and electricity to the factory. The juice extracted from the cane is treated in the subsequent process to remove the non sugar impurities.

Juice Clarification: Juice extracted from cane by mills or diffusers commonly termed as *raw*-juice or mixed juice contains a complex mixture of the integral components of cane plant. It is in the form of a turbid greenish liquid with a pH about 5.5. The non-sugar impurities in mixed juice are separated by the combination of chemical treatment, heating, flashing and decantation. Chemical reactants such as sulfur dioxide, lime hydroxide and phosphoric acid are used for juice clarification. After chemical treatment and heating, the juice passes through a flash tank, before entering the clarifier (decanter) , to remove air and gasses which unless otherwise hinder the settling of impurities in the clarifier. In the decanters the clear juice and the impurities (mud) are separated by overflow and under flow respectively.

Juice Evaporation: The clear juice with about 83-85% water is concentrated in a multiple effect evaporator under vacuum to yield syrup with about 35-40% water content. Usually quadruple effect evaporators are used for evaporation of juice in sugar factories. Exhaust steam from the mill turbine and power turbine system is used as thermal energy source in the first evaporation effect, which separates an amount of the water presented in the juice, and so producing the heating vapour for the next evaporation effect. The system works with decreasing pressure due to a vacuum imposed in the last effect, to produce the difference of temperature between each effect. The vapor generated in each effect may be used to attend other heat requirements of the process including juice heaters and vacuum pans.

Syrup sulphitation: syrup (concentrated clarified juice) from the evaporation system is sulphited to PH of about 5.5 by addition of the bleaching agent, SO_2 gas, to improve the color of final product.

Sugar Crystallization and Centrifugal Separation: the sulphited syrup is further concentrated in the vacuum pans by boiling until a certain supersaturation is reached. When the required supersaturation is reached, seed crystals are added and the sucrose in the solution starts to deposit on to these seed crystals. When the crystals reach the desired size the boiling process is stopped, resulting in a mixture of crystals and mother liquor known as massecuite. The massecuite from the pan is dropped in crystallizers and subsequently centrifuged in centrifugal machines for separating sugar crystals from mother liquor (molasses). The Crystals are washed, dried, cooled and sent to packaging or to storage.

The mother liquor (molasses) again is sent back to pan for boiling and re-crystallization. Usually three stages of re-crystallization are adopted to ensure maximum recovery of sugar in crystal form. The final mother liquor referred to as final molasses is sent out of the factory as by-product being unsuitable for recovery of sugar under commercial conditions from economic point of view.

The processing of sugarcane obviously should incorporate efficient steps to extract the sugar while eliminating the water, fibre and the non-sugar solids. It is again obvious that at each stage of the process while the other constituents are eliminated, they carry certain amount of sugar also. Hence the process has to be designed to keep at a minimum the sugar losses occurring through the exit of water, fibre and the non-sugar solids.

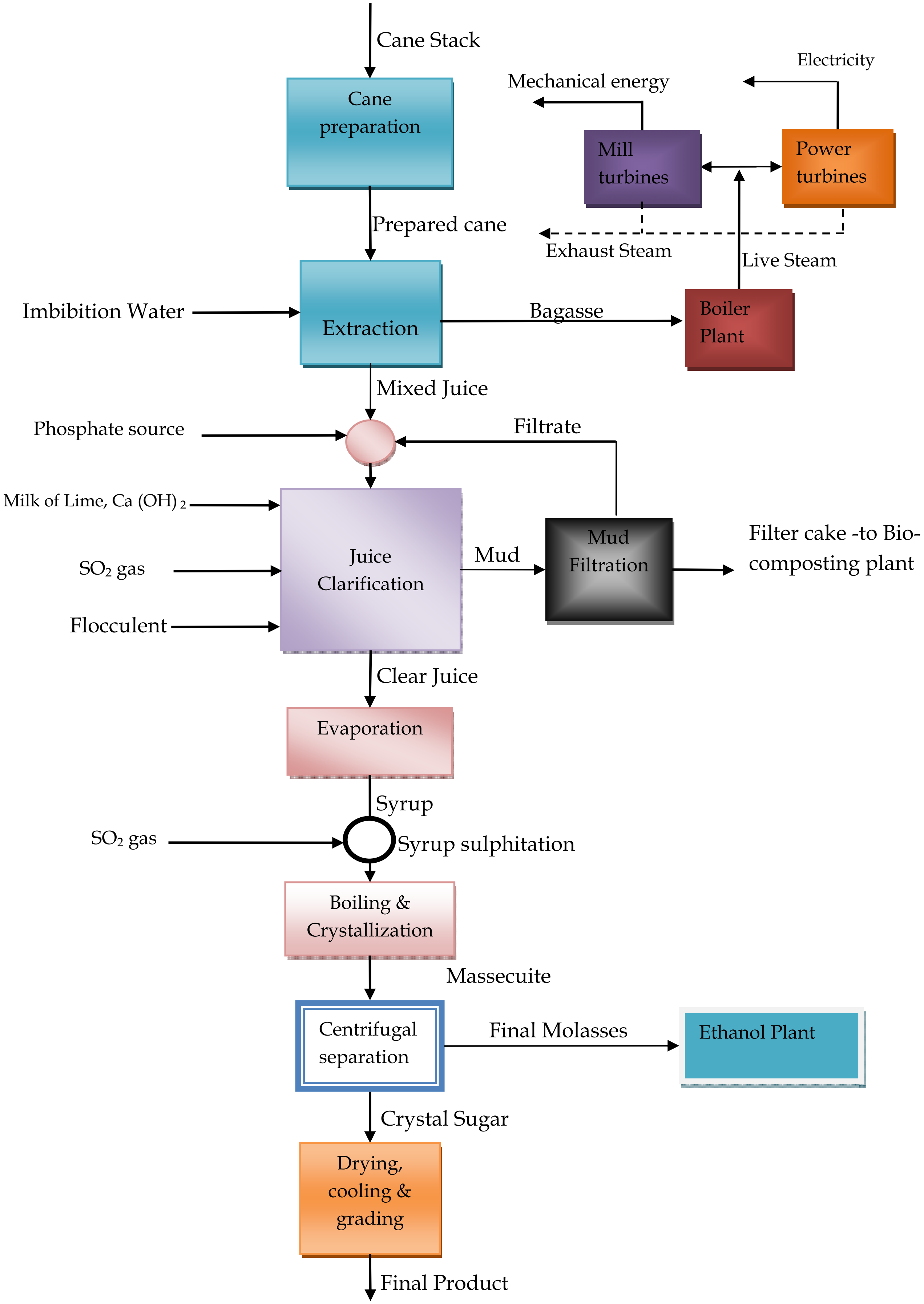

Figure 2.1: Schematic representation of cane plantation white sugar production in MSF

2.2 The Sugar Boiling and Crystallization Process

2.2.1 Crystallization Theory

The central technique to recover sugar in the raw house, in the plantation white sugar as well as in refinery sugar manufacture is the crystallization of sucrose from concentrated sucrose solution. It is the process whereby sugar is recovered by boiling in the vacuum pans and by cooling in crystallizers.

Crystallization is a mass transfer of sugar molecules from the syrup to the solid particles (seeds) to form or grow crystals. Crystallization leaves impurities in the mother liquor known as molasses. It is one of the most effective separation and purification techniques, providing an impurity-elimination effect of up to 99.9%. In other words, impurities are excluded from the crystallization process and pure solute is the only substance that crystallizes (granulated-refined sugar contains above 99.9% sucrose, plantation white sugar about 99.8% and raw sugar 96 to 99%). Crystallization occurs when the syrup is supersaturated and other conditions, like temperature, are met.

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 main purposes of crystallization in the sugar factory are to produce:

- Sugar with the highest possible quality, at high efficiency and low cost
- Molasses with the lowest possible purity (called exhausted or satisfactory molasses).

2.2.2 Sucrose Solubility and Supersaturation

In sugar technology, crystallization is based on the solubility of sucrose in impure solutions with different concentrations and temperatures. Sucrose solubility in pure water is the maximum amount of sucrose (sugar) that can be dissolved in 1g of water (g sucrose/g water) at a given temperature. 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).

Sucrose solubility in pure water depends only on the temperature of solution and increases considerably with rising temperature.

Although pure sucrose solubility in water is dependent only on the temperature, its solubility in impure solutions depends on three factors:

- Temperature
- Non-sucrose content
- Non-sucrose characteristics

At the same temperature, the concentration of sucrose in solution determines the type of solution, which may be unsaturated, saturated, or supersaturated.

In an **unsaturated** (undersaturated) **solution**, the solubility of solute (in our case, sucrose) has not reached its maximum limit yet. For example, a sucrose solution containing 150 g sucrose in 100 g water at room temperature (20ºC) is an unsaturated solution.

In a **saturated solution**, the solubility of solute has reached its maximum limit. If more sucrose is added to an undersaturated solution and stirred to dissolve it, eventually the solubility limit is reached, and more sucrose cannot be dissolved because no free water molecules are left in the solution to interact with sucrose molecules. In a saturated sucrose solution at a given temperature, sucrose will neither crystallize nor dissolve (saturated solution is in equilibrium condition).

In a supersaturated (oversaturated) solution, the solubility of solute has passed its maximum limit. In other words, a supersaturated solution contains more solute than normally can be dissolved in a saturated solution. In a supersaturated sucrose solution, sucrose will crystallize by a disruption such as seeding (a supersaturated solution is not in an equilibrium condition).

To find out the effects of impurities (non-sucrose) on sucrose solubility, the comparison of sucrose solubility in pure water with sucrose solubility in impure solutions can be expressed by three coefficients (a dimensionless ratio that compares the change of a variable with respect to other variables):

- Solubility coefficient
- Saturation coefficient

- Super-saturation coefficient

Solubility Coefficient

Solubility coefficient (K_S) compares sucrose solubility in pure water (part sucrose to each part of water) at different temperatures. In other words, K_S ($_S$ stands for solubility) indicates the **sucrose-to-water ratio** (*Sucrose/Water)* at a given temperature. For example, the maximum amount of sucrose that can be dissolved in 1 g water at 25ºC is about 2.0 g (2 parts sucrose to 1 part of water, or k_S =2.0). This is a 66.7% (by mass) sucrose solution.

$$K_S = \frac{Su}{W} = \frac{Su}{100-Su} \tag{2.1}$$

Where: K_S = solubility coefficient (g sucrose/g water)

S_U = Weight of sucrose (g)

W = weight of water (g)

Over the years, sugar scientists have tried to find a unique expression that correlates the effect of temperature (T) on solubility (S). The solubility of sucrose for a range of temperatures from 0 to 90 °C in pure sucrose solution is well represented by the Charles equation (Rein, 2007):

$$S = 64.397 + 0.07251 * T + 0.002057 * T^2 - 9.035 * 10^{-6} * T^3 \% \tag{2.2}$$

Where: S= g sucrose/100g solution at saturation t

T= temperature in °C

For temperatures between 100 and 125ºC, sucrose solubility can be calculated based on the Smelik equation (the equation is given in Bubnik et al. 1995):

$$S = 71.0615 + 5.3625 \cdot 10^{-2} T + 6.55303 \cdot 10^{-4} T^2 \% \tag{2.3}$$

Saturation Coefficient

Saturation coefficient (K_{IP}) compares sucrose solubility in an actual impure solution with a pure saturated solution, both at the same temperature. In other words, K_{IP} (*I* stands for impure and *P* for pure) indicates the effect of non-sucrose on sucrose solubility in impure sucrose solutions. K_{IP} is given as:

$$K_{IP} = \frac{(\frac{Su}{W})_{ACT}}{(Su/W)_P} = \frac{K_{ACT}}{K_P} \tag{2.4}$$

Where: $(S_u W)_{Act}$:- Sucrose-water ratio in actual solution or in impure solution (g/g)

$(S_u/W)_p$:- Sucrose-water ratio in pure saturated solution (g/g)

Some non-sucrose present in cane juice such as inorganic salts (Calcium and magnesium salts) increase the sucrose solubility and some such as reducing sugars like glucose and fructose decrease it. The net effect of impurities in sugar cane is decrease in solubility of the solution.

The effect of non-sucrose on sucrose solubility depends on the amount of these non-sucrose (the purity of the solution) and their characteristics.

Table 2.1: THIME's table of saturation coefficient at different purity of cane juice/syrup

Purity	100 - 86	86 - 68	68 - 58	58 - 50	50 - 42	42 – 35	35 - 30	30
Saturation Coefficient	1.0– 0.95	0.95–0.9	0.9-0.85	0.85–0.8	0.80– 0.75	0.75 – 0.7	0.7–0.65	0.65

Because of the difference in non-sucrose characteristics, syrups with different K_{IP} can hold different amounts of sucrose, affecting the rate of crystallization. The effect of the purity of a solution on saturation coefficient for cane juice/syrup is well described by Thime's Table (see table1).

In the case of impure solutions for purity above 70%, sucrose solubility can be calculated from the equation (4.5) derived by Vavrinecz (the equation is given in Bubnik et al. 1995):

$$S = [0.178 \times \frac{NS}{W} + 0.820 + 0.180 \times \exp\left(-2.1 \times \frac{NS}{W}\right) \times K_p\% \tag{2.5}$$

$$\frac{NS}{W} = \frac{DS-su}{100-DS} \tag{2.6}$$

Where: NS = Non-sucrose % in the solution

DS = Dissolved solid (brix)% in the solution

S = Sucrose% in the solution

W = water% in the solution

Note: The calculations become complicated when purity is below 70 %.

Super saturation Coefficient

Supersaturation coefficient (KSS) is the degree of supersaturation obtained at determined conditions of temperature and purity. It compares sucrose solubility in an actual impure solution with an impure saturated solution, both at the same temperature. KSS is given as:

$$KSS = \frac{(Su/W)_{ACT}}{(Su/W)_{I}} \tag{2.7}$$

Where: $(Su/W)_{Act}$ = Sucrose-water ratio in impure actual solution (g/g)

$(Su/W)_{I}$ = Sucrose-water ratio in impure saturated solution (g/g)

- KSS <1 – under saturated solution
- KSS = 1 - saturated solution
- KSS > 1 - Supersaturated solution

For crystallization to take place we must have supersaturated solution, that is, a solution containing more sugar than it should.

According to their supersaturation coefficient sugar solutions may be classified into supersaturation zones as follows:

Zones of supersaturation

- 1.0<KSS<1.2 - **Metastable zone**: Crystal grows (on existing crystals) without formation of new nuclei.
- 1.2<KSS<1.3 -**Transition (Intermediate) zone**: Crystal grows and formation of new nuclei in the presence of other crystals.
- KSS>1.3 - **Labile zone**: Spontaneous formation of nuclei (nucleation).

Figure2.2 shows a pure sucrose solubility chart with the most important lines (KSS=1, KSS=1.2, KSS=1.3)

In industrial practice, the presence of intermediate zone is doubtful and more importance is given to the limit between the Metastable and Labile zones. That limit varies with the purity of the solution. Higher supersaturation is needed for the lower purity solutions to reach labile zone.

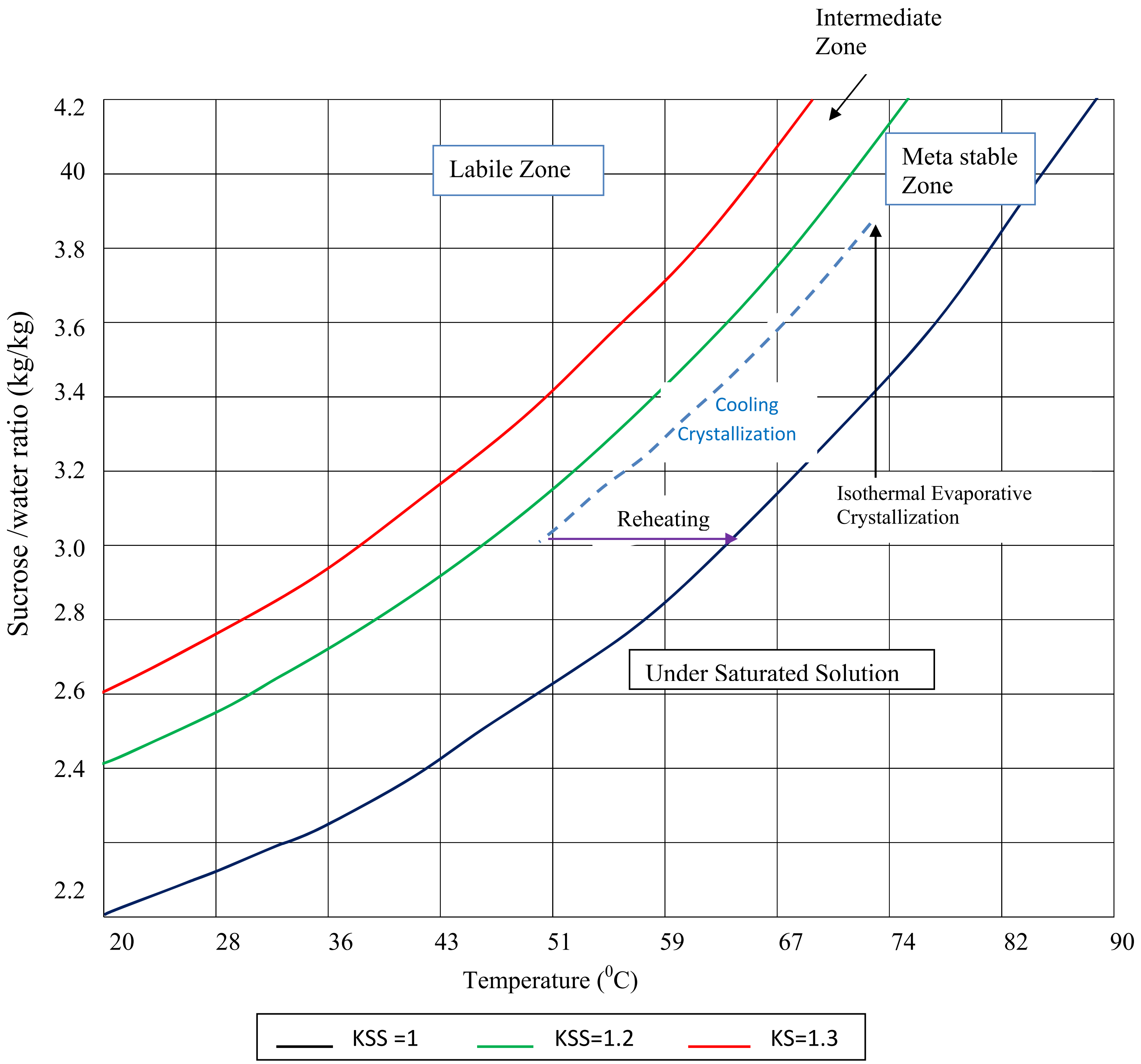

Figure 2.2: Solubility chart and zones of supersaturation for pure sucrose solution

2.2.3 Nucleation and Crystal Growth

Crystallization is a two-step process:

- Nucleation
- Crystal growth

N.B, In sugar factories nucleation is not required and only crystal growth takes place by seeding. This is to obtain the required number and size of crystals in the massecuite boiled. The number of crystals formed is uncontrollable in spontaneous crystal formation (nucleation).

2.2.3.1 Nucleation

Nucleation is the formation of crystals with or without **seeding** at a given supersaturation coefficient (K_{SS})**.** It may occur spontaneously or induced by an external stimulus. There are two types of nucleation, primary and secondary.

Primary Nucleation: This term refers to the nucleation which occurs spontaneously in the labile zone (homogeneous), 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).

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 small 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.

2.2.3.2 Crystal Growth

Basically, crystal growth is a rate process where the supersaturation is the driving force. **It** is expressed as a **crystallization rate** (R_C), which is the amount of the sucrose crystallized in one minute on $1m^2$ of the crystal surface. Crystal growth is a two-step process. The first step, which occurs in the syrup (the mother liquor), is a mass (weight) transfer by the **diffusion process**, in which the sucrose molecules from the solution phase are transferred to the crystal's surface. The second step, which occurs at the crystal's surface, is a position change by the **surface reaction**, in which the sucrose molecules position themselves into the crystal's structure. (Note: There must be enough heat energy to allow individual molecules to move around, position themselves on exiting crystals, and make stable bonds with them).

The driving force of the first step (the diffusion process) is directly proportional to the concentration difference between the diffusion layer (*C*) and the crystal surface (*C*1). The driving force for the second step (the surface reaction) is the concentration difference between the crystal surface and the adsorption layer (*C*0). Note that equilibrium exists at the interface between the liquid and solid (crystal) phases. Refer to figure5. In other words, the syrup concentration on the adsorption layer (*C*0) corresponds to saturated syrup, but at a distance from the crystal surface, the syrup is supersaturated and its concentration is *C*. First, the sucrose molecule diffuses through the distance of *d*1 and when it approaches the crystal, immediately sits on it; therefore, *C*0 remains the same. After eliminating the unknown *C*1 and combining the diffusion equation of both steps, the crystallization rate (*RC*) for a unit base [mass (*m*) per time (*t*)] can be expressed by the following equation:

$$\text{Crystal growth} = R_C = \frac{dm}{dt} = K_D * A(C - C_0) \qquad (2.8)$$

Where: C = Sucrose concentration in the mother liquor

$C0$ = Sucrose concentration on the crystal surface

K_D = Diffusion coefficient (the rate of mass transfer per unit area per unit concentration difference)

A = Overall surface area

The K_D is the diffusion coefficient (the overall mass-transfer coefficient), defined as the rate of mass transfer per unit area per unit concentration difference. The concentrations can be expressed in mass percent (g/100 g water) or mole fractions. According to Einstein (1879–1955), the K_D depends on the temperature of the syrup (T) and its viscosity (μ):

$$K_D = \frac{KT}{\mu} \qquad (2.9)$$

K = Constant, dependent on the molecular mass of solute (in our case, sucrose)

Substituting *KD* from Eq. (4.9) into (4.8) yields:

$$R_C = \frac{KT}{\mu A}(C - C_0) \qquad (2.10)$$

Equation (4.9) means that the sucrose molecule moves (by diffusion) at a rate of R_C from the solution with viscosity of μ to the crystal with surface of A and continues (by surface reaction) at the same rate to establish itself into the crystal structure.

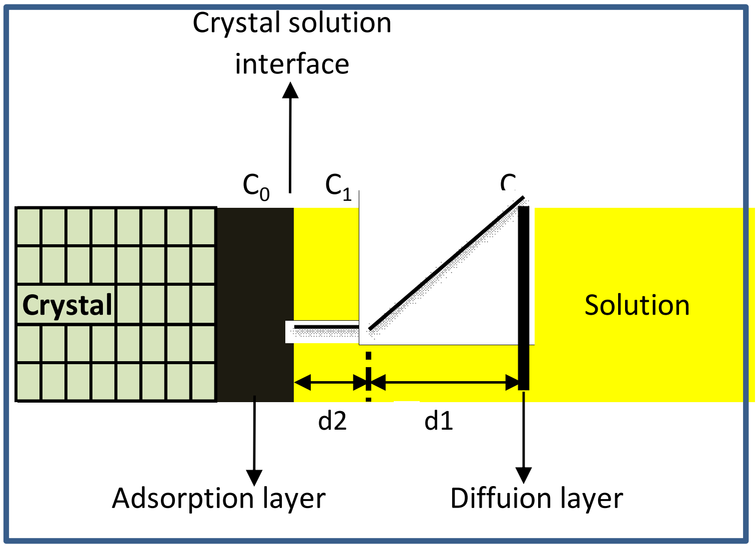

Figure 2.3: Crystal growth (Adsorption layer theory)

If the crystallization were dependent on only the first step (diffusion), all the crystals would have a round shape. The presence of the second step (surface reaction) completes the shaping of the crystal. The factors affecting the crystallization rate (RC) are as follows:

Size of seed grain: RC increases (sugar crystallizes faster) in massecuite with fine crystals because they have a larger total crystal area in a certain volume.

Temperature (T): RC increases with increasing T.

Purity of syrup (P): RC increases with increasing P. In other words, the presence of impurities slows 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). For example, first stage (A boiling) crystallization is faster than the second stage (C boiling) and the third stage (D boiling).

Supersaturation coefficient (KSS): supersaturation is the driving force for crystallization. RC increases with increasing KSS. But in practice supersaturation shouldn't exceed a threshold value to avoid false grain formation and excessive viscosity.

Viscosity: RC *d*ecreases with increasing viscosity because it is more difficult for diffusion of sugar molecules through the liquor. Increasing supersaturation increases the R but increasing viscosity decreases or even stops the growth of crystals, particularly at the end of the final stage of the third crystallization. The role of viscosity in crystal growth is most pronounced when the purity of mother liquor lowers. **Stirring:** *RC* increases with stirring.

In general, crystal growth occurs layer by layer on the crystal face and the growth rate of crystals follows McCabe's rule (McCabe et al. 2001), which states that all crystals grow at the same rate if the KSS stays the same for all crystals. Therefore, the crystals in a massecuite with KSS of 1.10 grow three times faster than that in a massecuite with KSS of 1.03; $\frac{1.1-1.0}{1.03-1.0}=\frac{0.1}{0.03}=3$

2.2.4 Crystal Size and Content

The crystallization rate is higher with greater surface area. The surface area of crystals (Sa) can be calculated from the crystal size and crystal content by the formula

$$Sa=\frac{19X}{L(100-X)} \tag{2.11}$$

Where: X represents crystal content and L is the length of crystal, in mm.

For a given size of sugar crystal, the total crystal area will be determined by the crystal content of massecuite. Thus the rate of deposition of sugar from mother liquor will depend on the total crystal area, for a given set of conditions and for a given size of crystals desugarisation of molasses will be more and faster when the crystal content is high. But as the viscosity of massecuite rises with crystal content and it is essential to maintain the concentration and viscosity as high as possible a practical limit is set to the crystal content of massecuite. It is therefore essential to maintain crystal content at highest level along with high viscosity of mother liquor for maximum exhaustion subject to the workability of massecuite in the crystallisers and centrifugals.

Crystal content (*CC* or crystal yield) is the percentage (by mass) of crystals in the massecuite. *CC* can be calculated from the **purity drop** (purity difference) and from the difference in sucrose content in the massecuite and its mother liquor:

$$CC=\frac{B_M(P_M-P_{ML})}{100-P_{ML}} \tag{2.12}$$

or

$$CC=\frac{S_M-S_{ML}}{100-S_{ML}}\times 100 \tag{2.13}$$

Where: B_M =massecuite of Brix (%)
P_M = Purity of massecuite (%)
P_{ML} = Purity of mother
S_M = Sucrose content of massecuite (%)
S_{ML} = Sucrose content of mother liquor (%)

2.3 Crystallization Techniques for Sugar Production

In general, crystallization can occur from any phase, solid, liquid or gaseous. In sugar production, crystallization 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).

Crystallization occurs when solute (in our case, sucrose) moves from a supersaturated solution (in our case, impure sucrose solution) and attaches to the crystal structure. There are two types of crystallization in sugar factories, namely;

- Evaporating crystallization (crystallization by concentration of the solution)
- Cooling crystallization (crystallization by cooling).

An equilibrium condition is reached when the solution is saturated at a given temperature, which can be shown by the solubility curve as a function of temperature Figure6 helps elucidate this process. For example, let us consider point *A (under saturated solution)*, from which a supersaturated condition can be obtained by evaporation (1), by flashing evaporation (2), or by cooling (3).

From Figure 5, the crystallization zones can be defined as:

Stable (under saturated): In this area, neither nucleation nor growth can happen

Metastable (safe): In this area, nucleation can happen only in the presence of seed.

Unstable (unsafe): In this area, nucleation can happen in the presence or absence of seed

Both the metastable and unstable zones are in supersaturated positions. At low supersaturation (*KSS* = 1.1), which is in the metastable zone, no new crystals are created, and only existing crystals gradually grow. On the other hand, we know that *the* higher the supersaturation, the faster the crystals grow. Therefore, the supersaturation should be increased, but there is a limit to it because supersaturation should be kept below the upper safety zone (*KSS* =1.6) to prevent the creation of new (false) crystals.

Note: the crystallization process in sugar factories takes place in the metastable zone. The syrup is boiled in vacuum pans until the desired supersaturation is obtained and then finely ground sugar suspention in alcohol called seed or slurry is added to the solution. The number and size of the seed crystals is as to obtain the required volume of massecuite with the required crystal grain size at the end. Predetermined number and size of crystals are required. If supersaturation is too high and enters the unsafe zone, new crystals are formed, called false grain.

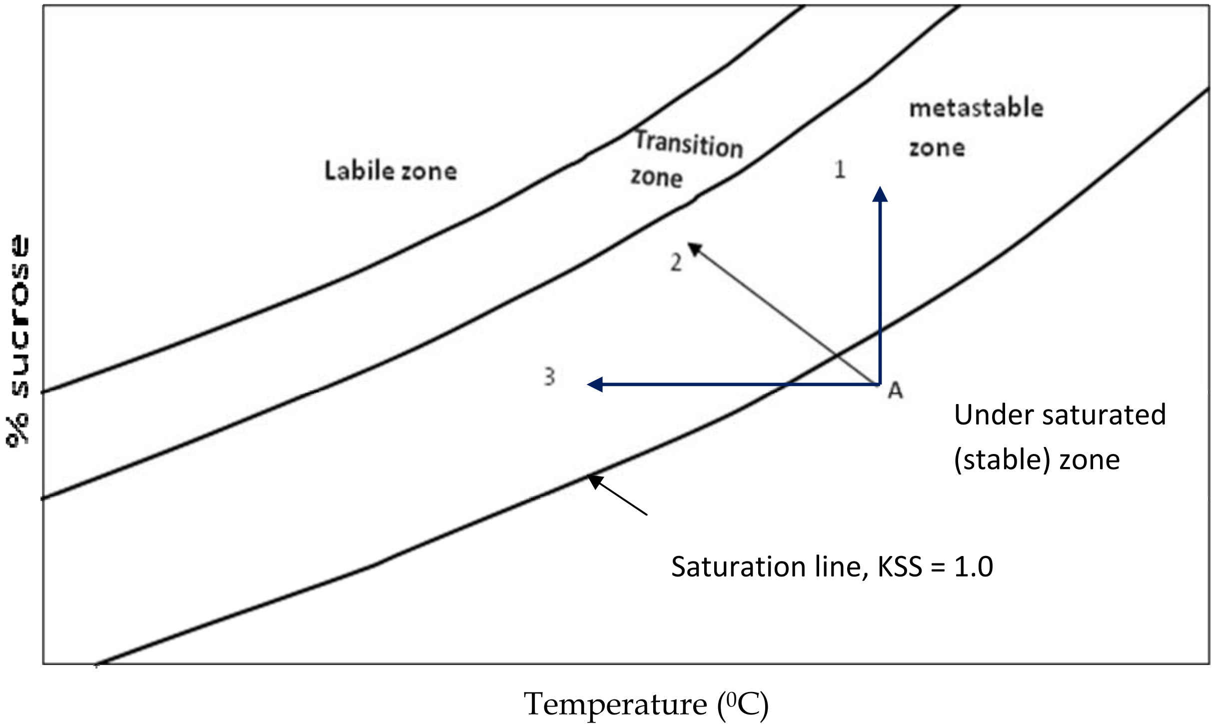

Figure 2.4: Formation of supersaturation solution by evaporation and cooling

2.3.1 Evaporative Crystallization (Crystallization by Boiling)

At low concentrations (undersaturated situation), the syrup contains many free water molecules, so water can easily dissolve the sucrose (sugar) molecules. As evaporation continues, the number of free water molecules and water molecules bonded to each sucrose molecule is gradually reduced until the syrup becomes saturated (*KSS* =1). At saturation, the system is in equilibrium.

Supersaturation in evaporative crystallization is obtained by further removing water from the solution by evaporation under vacuum increasing the brix (soluble solid 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. The crystals absorb more sucrose due the concentration difference between the syrup, in which the crystals are growing, and the saturated syrup, which is on each crystal's surface and is in equilibrium with the crystals. This is the basis of the general statement: the concentration difference in a supersaturated solution is the driving force that causes the crystals to grow. (A positive difference causes sucrose crystals to grow, and a negative difference dissolves them.)

Evaporative crystallization in sugar factories takes place under vacuum (at about 65^0C) in vacuum pans. The presence of a vacuum decreases the boiling point (BP) of the syrup, preventing the thermal degradation and inversion of sucrose, minimizing color formation and allows to use low pressure steam or vapor bleed from evaporation plant for boiling (energy saving).

In sugar terms, the product of the crystallization process is called **massecuite** which is a semisolid mixture consisting of crystals and the surrounding solution around the crystals, called mother liquor or molasses (see Figure 8). Mother liquor is the liquor from which crystals are formed that stays with the crystals until they are separated by centrifuging.

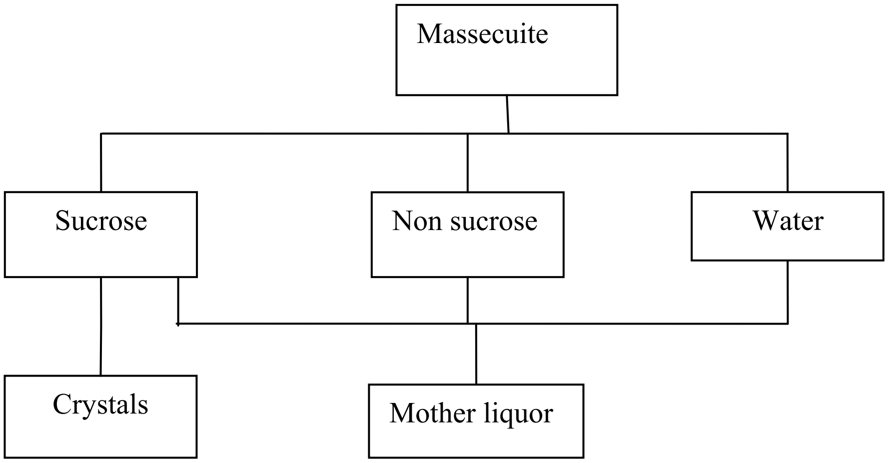

Figure 2.5: Massecuite composition

Evaporative crystallization can be conducted in batch or continuous vacuum pans (van der Poel et al., 1998).

Boiling in Batch Vacuum pans

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).

Charging: The feed (syrup or molasses) is drawn into the pan by vacuum until the level covers the calandria (heating element). 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.

Seeding: Concentration ends when a specific **supersaturatio**n 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. For seeding with magma (C or B sugar mingled with water or juice and used for A -seeding), the level of super saturation can be lower. **Seeding** is one of the most important steps in the crystallization operation because it determines the size and yield of the crystals in the massecuite. The following should be taken into consideration during the seeding process:

- Ensure that the seed is at the right size particles (4 to 10 µm)
- Ensure that the seeding is performed at the right *KSS* (in A- pan 1.1 to 1.2).

Grain Establishment: 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.

Feeding (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 feed into the pan. Liquor (molasses or syrup) is fed in to the pan for growing the crystals in the massecuite. During crystallization, the deposition of sugar on the crystal reduces the concentration of the mother liquour; evaporation of water from the solution tends to concentrate the mother liquor. These two factors must be balanced to maintain constant supersaturation.

Tightening: This phase begins when the maximum working volume of the pan is attained with the desired crystal size of the sugar in the massecuite. At this stage, Feeding is stopped and 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 brix measurement (soluble solids content measured by refractometric index or density) or by indirect measurement of the consistency (stirrer current).

Discharging: When the maximum allowable consistency (Brix) is reached, the vacuum is released by injecting air 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 (by steaming out).

Pan time index (batch time) is used to determine pan capacity, which is the total volumetric (m^3) amount of massecuite divided by the massecuite volumetric flow rate (m^3/h). Depending on the boiling scheme the pan time index is: for “A” massecuite 4 - 5 hours; for “B” massecuite 4.5 - 6 hours and for “C” massecuite 5 - 7 hours (Rein, 2007).

Boiling in Continuous Vacuum Pans

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. For magma graining, the seed flow is approximately 22 to 33% of the final massecuite flow (Rein, 2007).

For example, in a 12 cell pan, the first two cells are used to condition the seed. 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 syrup or molasses feeding rate (factory load requirements). Tightening (brixing up) 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).

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/C” strikes between 3.5 and 4 hours and for “C/D” strikes between 6 and 6.5 hours (Rein, 2007).

2.3.2 Cooling Crystallization

The process of sugar crystallization is started in vacuum pans and completed in cooling crystallizers. Cooling crystallization has its most important application in the final stage of the massecuite processing, to maximize the sugar recovery from the final molasses (molasses exhaustion). The objective of massecuite cooling is to maximize the amount of sucrose that is recovered out of the solution in the massecuite by depositing on the surface of the existing crystals. The solubility of sucrose decreases with cooling.

During the final stage of the evaporative crystallization (tightening), the dry substance content or brix of the massecuite is increased to a maximum value (~97 refactometer brix or 102 hydrometer. 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 the heat transfer in the vacuum pan. After completion of crystallization by boiling, low-grade massecuites, with temperatures between 60 and 65°C and a supersaturation close to 1.2, are sent to the cooling crystallizers. When a crystal forms, heat (energy) is released from the solution, known as the heat of crystallization (crystallization is an exothermic process).
In cooling crystallization, the temperature of the massecuite is lowered to about 40-50˚C, causing crystallization to occur because the water present in the mother liquor holds less sugar at lower temperature(solubility decreases with decreasing temperature). The minimum temperatures that can be reached in the crystallizers vary depending on the workability of the massecuite and should be determined by each factory experimentally (Rein, 2007). 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 40-45°C (95°F). 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 specifically designed heat transfer surfaces.

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, fast cooling is not advised to avoid formation of false grain (unwanted crystals formed spontaneously)(Rein, 2007). The temperature drop is controlled by the flow and temperature of the cooling media (usually cold 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). Generally, cooling rates are between 1.5 and 2°C/hr (Meade and Chen, 1977).

The cooling time (retention time) in crystallizers varies for various types of massecuites and also with viscosity. A significant purity drop is found at a residence time up to 30 hours for low grade (final stage) massecuite. 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 (Meade and Chen, 1977)

Table 2.2: Massecuite cooling time in different sugar factories

Type of massecuite	**Retention time (hrs)**			
	Mauritius	RSA (South Africa)	FSF (Fincha)	MSF (Metahara
"A" massecuite	4-6	10-12	-	
"B" massecuite	6-10	18-24	-	
"C/D" massecuite	18-36	36-48	36-40	16-23

Types of cooling crystallizers;

i. 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).

ii. Continuous cooling crystallizers

With the change from batch to continuous low grade station operation (continuous pans and continuous centrifuges) and to ease automation (fewer valves), it was required to update cooling crystallization from batch to continuous. Two types of continuous crystallizers known in sugar factories; horizontal and vertical. 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 are the oldest and are being used only in few old sugar factories because horizontal they 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 may 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 lower leak problems (Rein, 2007).

Ideal conditions to achieve a good performance in continuous crystallizers is to have 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). 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 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.

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 brix (dissolved solid %), non-sucrose/water ratio, (purity), crystal content and crystal size (Rein, 2007).

2.3.3 Massecuite Reheating

Because the low-grade (D) massecuite is cooled in the cooling crystallizer to about 45ºC, viscosity of the massecuite becomes too high at this low temperature to the point where it is difficult to centrifuge. It is therefore necessary to reheat the massecuite after cooling to a temperature between 50 to 55^{0}C to reduce the viscosity and facilitate curing by centrifugal machines at a reasonable rate and without using excessive wash water or steam(. It is important that the reheating be performed in a way that does not re-dissolve sugar crystals. To avoid re-solution of crystals, reheating must be done rapidly with a minimum retention time of about 30 minutes by a temperature-controlled hot water system and without using any unduly hot surfaces that could cause local overheating and dissolution. The reheater thus must therefore have a high heating surface area to volume ratio and achieve close contact between all the massecuite and heating surfaces. Reheating surface temperatures should be limited to between 60 -65^{0}C. This amount of heating only reduces slightly the mother-liquor supersaturation. In other words, almost no loss of crystals from reheating can occur. Curing (centrifuging) must follow immediately after reheating.

Generally massecuite reheaters should consist the following features:

- Significantly Higher heating surface area to volume ratio
- Very close contact between the heating surface and massecuite
- No 'dead spots',
- No opportunity of massecuite 'channeling',
- Low massecuite- to- water approach temperature to avoid dissolution of crystals.

2.3.4 Massecuite Separation (Centrifugal separation)

It is the separation of massecuite into mother liquor (molasses) and sugar crystal using centrifugal machines. Separation of D-massecuites takes place after crystallization and reheating as the final step of purification to remove molasses from the sugar crystals. This process of separation of sugar crystal from mother liquor is called **centrifugation, fugaling, curing or purging**. Spray water is applied on centrifugal machines to remove molasses adhered to the surface of sugar crystals. The amount of water applied is a compromise between the purity of final molasses and the purity of DFW sugar. The higher the amount of water applied, the higher the purity of final molasses and loss of sucrose. But if the amount of water applied is low, there will be low DFW magma purity and high recirculation of non- sucrose back to the boiling house affecting the capacity of the factory, steam consumption and quality of final product.

2.4 Equipment Setup in MSF at Low Grade Massecuite

For boiling of D-massecuite, there are two batch caldaria type vacuum pans with working volume of each 400HL. One batch takes 5 – 7hr giving the total D-massecuite production rate of 16-20t/h. After completing boiling, the massecuite is discharged by gravity in to a D massecuite receiver of 420HL capacity. The hot massecuite is then pumped from massecuite receiver under vacuum pans to the continuous cooling crystallizers.

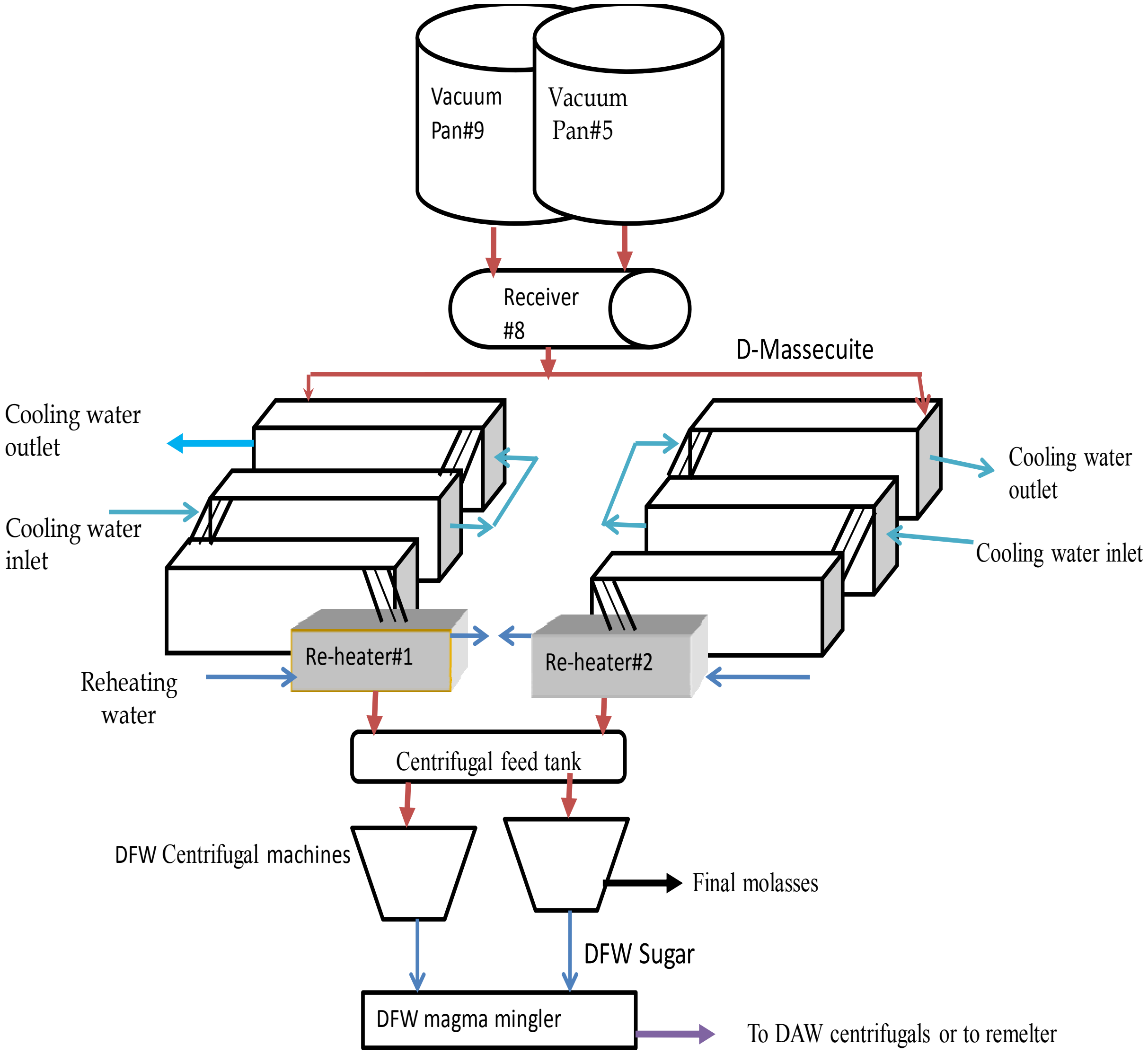

Figure 2.6: Equipment setup for D-Massecuite Processing in MSF

The D-massecuite cooling crystallizer station at MSF consists of two batteries each with three workspoor type U-shaped horizontal crystallizers connected in series. Crystallizers are equipped with agitator shafts fitted with cooling discs. Both shaft and discs are hollow and designed to permit circulation of water. The massecuite is introduced continuously at one end from which the shaft is driven, and flows along the crystalliser by gravity, passing from one space between discs to the next by means of the open sector of the disc, and overflows to the next crystallizer at the opposite end of the crystalliser; the cooling water enters at the latter end, which is thus the outlet end for the massecuile, passes in succession through all the discs, and returns through a pipe or the hollow shaft of the crystallizer to leave at the end at which it entered.

This is therefore a countercurrent circulation. It presents the advantage that the hot massecuite entering comes into contact only with water which has already been heated, and that at any point the temperature of the cooling water is progressively lower as the massecuite becomes cooled (Figure 2.6). These conditions practically eliminate risk of false-grain formation. Only the first two crystallizers of each battery are water cooled while the last crystallizers are air cooled. The total installed crystallizer volume is 220m^3, which gives retention time of 16 – 23hr.

The cooled massecuite is re-heated in two finned tube re-heaters (Heating surface area of 150m^2 and working volume of 3.5m^3 each) and centrifuged in two continuous centrifugal machines.

2.5 Boiling Schemes or Systems

The operation of sugar boiling house is arranged with the aim of maximum sucrose recovery or minimum sugar loss, proper quality of final product and minimum quantity of Total massecuite boiled. Practically it is not possible to exhaust the crystallizable sucrose from syrup in one boiling stage. In order to meet the primary goal of an efficient sugar boiling house, the sugar coming with the evaporator syrup has to be recovered by several crystallization stages or different boiling schemes. The best boiling scheme is the one which gives the required sugar quality, minimum sugar loss with final molasses, and 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).

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 Rein (2007).

The boiling scheme (number of stages and streams distribution) depends on the purity of the syrup from evaporation plant and the desired sugar quality (Birkett, 1978).

- For syrup purities higher than 85%, four boiling stage is recommended.

- For syrup purities between 82 and 85%, three boiling stage may be adopted.
- For syrup purities less than 82% only two boiling stage can do.

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 taken out of the process as by-product. Depending on the scheme, the final product sugar is produced by the first stage or by the two first stages. Sugars from the last stages are recycled back to produce the first stage.

In Metahara Sugar Factory, having 82-84 syrup purity, three boiling system is employed and three types of massecuite are produced. These three grades of massecuite produce sugar of different quality and polarization. The different massecuite grades are designated by letters (“A”, “C”, “D” massecuite) or by numbers (first, second, third massecuite); molasses and sugars (magmas) from the same massecuite take the same designation (“A” molasses, “A” sugar) Etc. the first massecuite is called High grade massecuite and the remaining are called Low grade massecuites.

The first boiling, called A-boiling takes place by using syrup from evaporation plant to produce A-massecuite. This A- massecuite is separated in to A- molasses and A-fore worker (AFW) Sugar by A- fore worker (AFW) batch centrifugal machines. The AFW obtained is mingled with water or A-wash and double cured by A- after worker (AAW) batch centrifugal machines to produce A-wash and A-after worker (AAW) sugar. The AAW is then dried, cooled, graded and packed for commercialization. The A-molasses is rich in sucrose and therefore boiled in C-pans to produce C- massecuite. The C- masscuite is then separated in to C- sugar and C-molasses by C–continuous centrifugal machines. The C- sugar is mingled with hot water to produce C-magma which is pumped to pan floor to be used as seed (footing) for A-boiling. The C-molasses still contains certain amount of sucrose that can economically be recovered by boiling. Hence it taken to pan station for D-mamssecuite boiling. D- massecuite, called final massecuite, is separated by D-fore worker continuous centrifugal machines into D-fore worker (DFW) sugar and D- molasses final molasses) after cooling and reheating. The DFW is either melted (dissolved) to be used as feed for A-massecuite boiling or mingled with water and double cured by DAW continuous centrifugal machines to produce D- after worker (DAW)sugar and D-wash. If D-double curing is adopted, the DAW sugar is melted or dissolved by using water or clear juice and low pressure steam and used as feed for A- boiling. The final molasses, although contains certain amount of sucrose, it is not economical to further boil and recover the remaining sucrose, and hence it is taken out of the system as by-product to be used as raw material for alcohol production, cattle feed or other purposes.

Only the first sugar (A –sugar) is commercialized. The C and D sugar are recycled back for A boiling as a seed(C magma) and remelt liquor respectively.

The three boiling system is described by flow diagram for Metahara Sugar Factory (MSF) in figure 2.7 and 2.8 for double and single D –curing respectively.

1) A massecuite of purity 85 - 87^0 is produced by boiling syrup purity of 82-84^0, A-wash purity of 94-96^0, remelt liquor purity of 88- 92^0 and C– magma (seed) purity of 92-94^0.
2) C massecuite of purity of 70-72^0 is boiled with A- molasses purity of 68–70^0, small amount of syrup. Syrup is used for purity balance.
3) D massecuite of 55–58 is boiled with a small proportion of A-molasses and a large proportion of C- molasses (48-50^0 purity) and-wash (purity of 64-67^0).

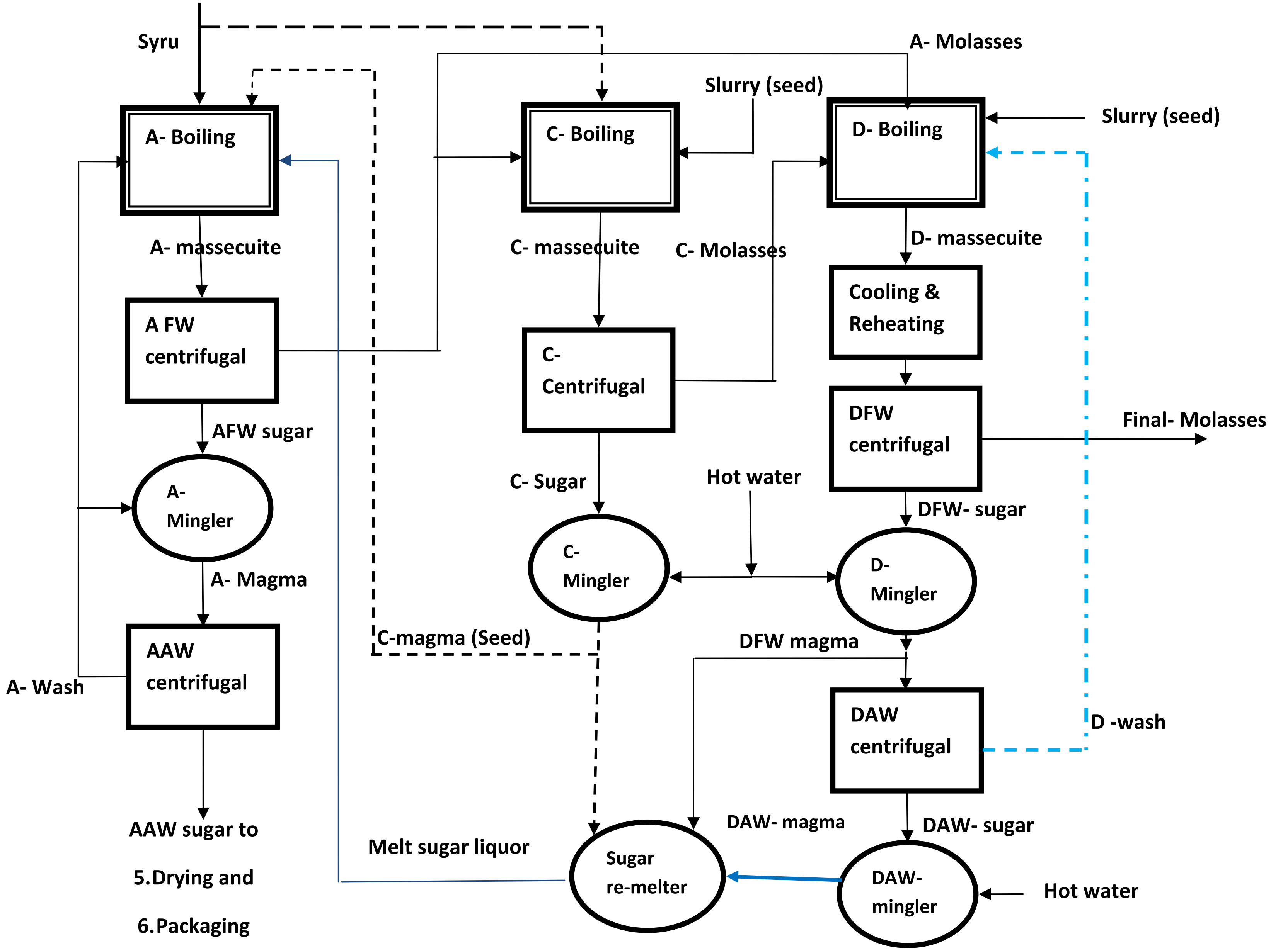

Figure 2.7. Three boiling system with D-double curing in Metahara Sugar Factory

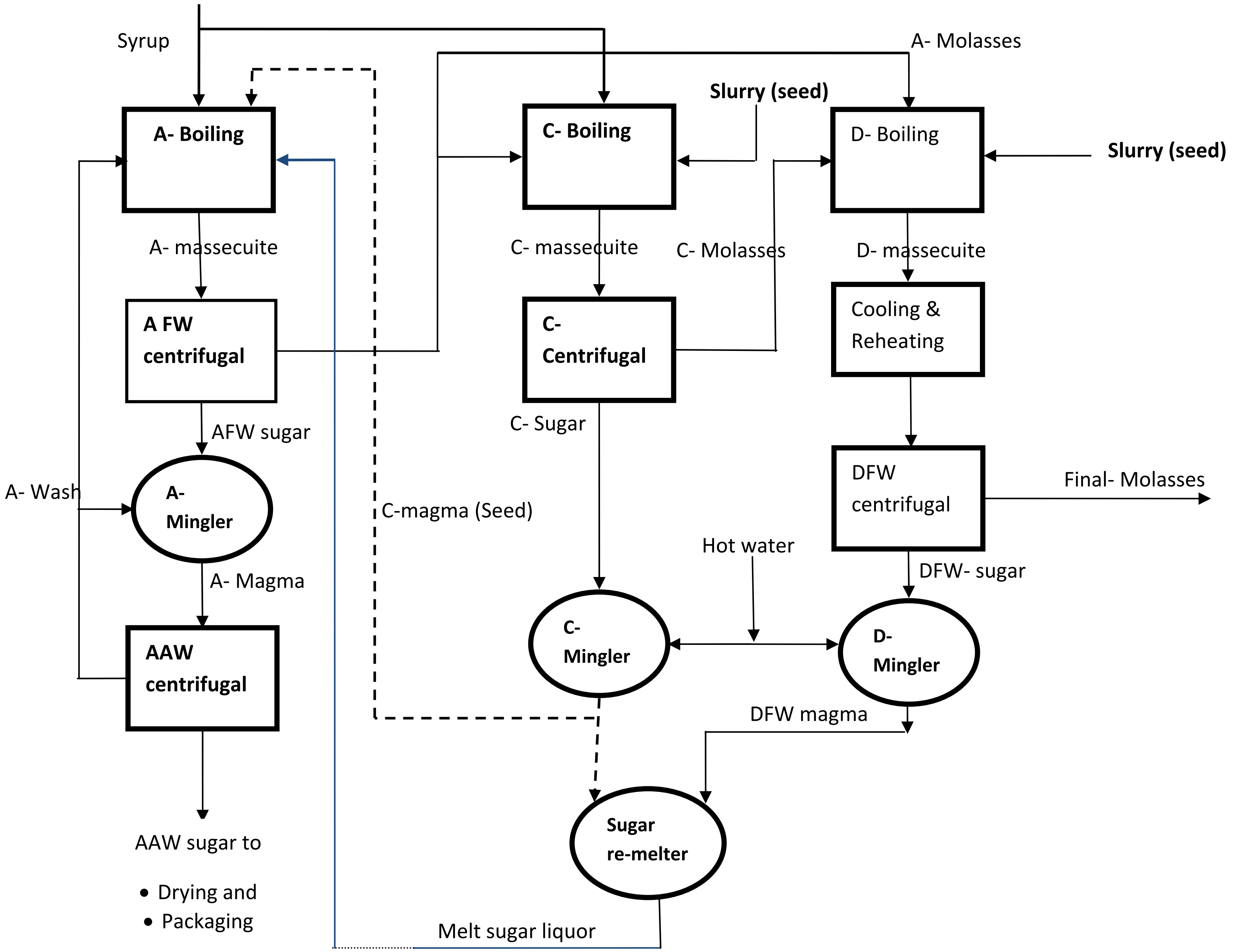

Figure2.8. Three boiling system with D-single curing in Metahara Sugar Factory

2.6 Factors Affecting Molasses Exhaustion and Optimum operation of D Massecuite station

The achievement of low final molasses purity is realized by a combination of the performances of the following processes in the Final massecuite station-station (D-massecuite station): seed preparation, massecuite boiling, cooling crystallisation, reheating and centrifugation. There is a close interaction between these processes and each needs to be optimized in conjunction with the others. In plant operation, the equipment and the technique of operation have both to play important role in reducing the loss of sucrose in molasses. In process operation the conditions which influence exhaustibility are:

Preparation of C-seed

The grain size of the seed for D-boiling is critical and must be sufficiently large to produce a final grain size in the D-massecuite of 0.25-0.3mm. The correct preparation of slurry is essential to achieve this (Ninela and Rajoo, 2006).

Purity drop during C-massecuite boiling

Good operation of D-massecuite boiling not only achieves good crystallization in the pan but sets up the conditions for crystallization in the cooling crystallizers

Massecuite Brix: The high brix massecuites are favored for better exhaustion. There are however practical limits in operation; and attempts have to be made to keep the brix of massecuite consistency (viscosity) with the capacity of equipment to handle the same. The efforts to eliminate water from the massecuite during boiling need to be tempered by the need to ensure that the rate of crystallisation is not too severely retarded. Thus, the massecuite needs to be of a suitable consistency to boil, mix and flow, as well as having sufficient crystal surface area on which the crystallising sucrose can deposit. This led to the recommendation that massecuite brixes should approach 98 refractometric brix or 102 hydrometer brix (Rein,2007).

Purity of the 'D'- massecuite: it should be neither too high nor too low. If too high, sufficient drop in purity will not be obtained in practice and hence final molasses purity will be high. If too low, viscosity of massecuite will necessarily be very high and crystals will be small, thus aggravating the difficulty of massecuite boiling in pans and molasses separation in the centrifugals. Depending on prevailing conditions the right purity of 'D' massecuite may be different. The optimum purity of the massecuite should be determined by specific sugar factory (RASTIC.2004).

Purity Drop on Cooling

For any given massecuite discharged from the pan, good crystallisation is achieved by having crystallisers with sufficient retention time, good flow patterns (i.e. close approach to plug flow) and sufficient cooling. While increasing the residence time up to 48 h generally continues to reduce the molasses purity, the economic optimum is difficult to specify as it depends on the particular details of how the value of the extra sugar recovered is distributed (the division of proceeds system). Rein (2007) reports that, in South Africa, C-crystallisers are often installed to provide a residence time of 45 h. With this installed capacity a molasses purity drop of between 6 and 8 units can usually be achieved. Since there is some evidence that very rapid cooling can promote the formation of very fine crystal that will dissolve out during reheating or washing in the centrifugals, rapid cooling is normally avoided by running the cooling water in a counter flow direction to the D-massecuite. The optimum cooling temperature has also to be determined to obtain good results. Temperature profile, residence time and residence time spectrum are parameters which can be influenced by the design of cooling crystallizers. The massecuite temperature must only drop as quickly as allowed by the crystal growth rate. Otherwise the supersaturation of the mother syrup is liable to increase too much, leading to formation of fine crystals. In actual operation; a cooling rate of approx. 1 ^{0}C/h is a good design figure cooling crystallizers.

Purity change on reheating

A small purity rise or purity rise is normally recorded across the reheater. Reheating temperatures should be optimized and controlled. Purity rise and viscosity should be taken into consideration. Rapid reheating (15^{0}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 m^2/TC for water to massecuite temperature difference of 3°C (Rein, 2007).

Purity Rise on Centrifugal separation

A purity rise is normally expected across curing because some sucrose crystal will always pass through the centrifugal screen holes and some crystal will be dissolved by the wash water. The key to improving final molasses exhaustion here is to minimize the sucrose loss through the screen and minimize the dissolution of sucrose.

The amount of **spray hot water** applied during centrifugation should be optimized which is a compromise between molasses purity and sugar purity. If the spray water amount is excessive, there is a danger of dissolving sugar crystals which will be lost with final molasses; on the other hand if the amount of water is lower than required there will be recirculation of molasses with sugar to boiling house. The purity of the D fore worker sugar should be at least 84-86° otherwise too much molasses is recirculated and crystallization is rendered very difficult because of high mother liquor viscosity at end of process. Double curing may be required to minimize non sugar recirculation.

The ‘D' centrifugals screen should also be examined regularly and damaged screen should be replaced at once to minimize the sugar crystal loss through worn-out screens. Procedures need to be developed for centrifugal screen cleaning, screen maintenance and screen replacement to ensure that the screens are always in the best possible condition. It has been shown that D-massecuite crystal width must be >120 micron on average to minimize passage of crystal through the 60 micron slots or holes in a centrifugal screen (Julienne, 1985). The more regular grain size will produce less small grain that can pass through the screen. Therefore it is critical to pay attention to crystal size and quality in the pan.

In the recent past, with the introduction of continuous centrifugals the trend towards reducing the crystal size to 0.2 mm in final massecuites has been established as against the previous practice of maintaining 0.3 to 0.35 mm which suited the batch machines in respect of separation of sugar from molasses. One limiting factor in size reduction of sugar crystals is the availability of pore space in the crystal bed for passage of molasses during centrifugal separation since as the crystal size diminishes the porosity goes down and resistance to flow of molasses increases tremendously. Thus in practice with high gravity centrifugals and the crystal size of 0.3 to 0.35 mm. good molasses separation is obtained. With modern continuous centrifugals the limit to the size of grain may be considered to be 0.2 mm.

In the context of efficient centrifugal separation uniformity of sugar grains is equally important in as much as the pore volume and the drainage of molasses is adversely affected with sugar crystals of different sizes, since the packed bed of crystals offers resistance to flow of mother-liquor. 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).

Viscosity:

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. “D” 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.

Viscosity is a dominant factor in the final stage of crystallisation and many times becomes a limiting factor in the exhaustion of molasses. Final molasses exhibits slight pseudo plastic flow and massecuites show pseudo plastic tendency to a greater extent than molasses, particularly at high crystal content.

In a massecuite, viscosity is related to temperature (*T*), dry substance (*brix)* and purity of mother liquor. Final molasses is the most *viscous liquid* in the sugar operation. This is because of its low purity (high non-sucrose) and high *dissolved solid or brix* content.

An increase in temperature decreases the viscosity and increases the crystal growth rate (a temperature rise of 10ºC in syrup, doubles the crystal growth rate. Increasing the brix of the mother liquor increases its viscosity. The viscosity of the molasses exponentially increases when its brix increases.

N.B. After nonsucroses, viscosity is the second most important parameter affecting sugar losses to molasses. Viscosity is important not only in sugar crystallization (particularly the low-grade) but also in the transformation of in-process products in pipes, cooling crystallization, molasses exhaustion, and centrifuging of the massecuite.

2.7. Response Surface Experimental Design

An experiment is a series of tests, called runs, in which changes are prepared in the input variables in order to recognize the reasons for changes in the output response (Montgomery & Runger [3]). Design of Experiments (DOE) is a powerful technique used for exploring new processes; gaining increased knowledge of the existing processes and optimizing these processes for achieving world class performance (Jiju Antony, 2003). Often engineering experimenters wish to find the conditions under which a certain process attains the optimal results. That is, by careful design of experiments, they want to determine the levels of the design parameters at which the response reaches its optimum. The optimum could be either a maximum or a minimum of a response (output variable) which is influenced by several independent variables (input variables). One of methodologies for obtaining the optimum results is response surface methodology.

Response Surface Methodology (RSM), invented by Box and Wilson, is defined as a collection of mathematical and statistical tools or techniques useful for modeling, analyzing and simultaneously solving problems in which a response of interest is influenced by several variables and the objectives is to optimize this response (Giovanni, 1983). Response surface methodology also quantifies the relationship between the controllable input parameters and the obtained response surfaces. It is a well known up to date approach for constructing approximation models based on physical experimented observations (Box et al., Montgomery). The main advantage of RSM is the reduced number of experimental runs needed to provide sufficient information for statistically acceptable results (Montgomery 2001).

The design procedure of response surface methodology is as follows:

(i) Designing of a series of experiments for adequate and reliable measurement of the response of interest.

(ii) Developing a mathematical model of the second order response surface with the best fittings.

(iii) Finding the optimal set of experimental parameters that produce a maximum or minimum value of response.

2.8. Summary

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). 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.

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 as by product. Depending on the scheme, the sugar is produced by the first stage or by the two first stages.

The processing of sugarcane obviously should incorporate efficient steps to extract the sugar while eliminating the water, fibre and the non-sugar solids. It is again obvious that at each stage of the process while the other constituents are eliminated, they carry certain amount of sugar also. Hence the process has to be designed to keep at a minimum the sugar losses occurring through the exit of water, fibre and the non-sugar solids.

The achievement of low final molasses purity is realized by a combination of the performances of the processes in the Final massecuite station-station (D-massecuite station): seed preparation, massecuite boiling, cooling crystallisation, reheating and centrifugation. There is a close interaction between these processes and each need to be optimized in conjunction with the others. One of methodologies for obtaining the optimum results is response surface methodology (RSM). RSM will extensively be used in this thesis to optimize the process parameters of D-massecuite boiling, cooling, reheating and centrifugal separation in order to minimize sucrose loss with final molasses.

3 Materials and Methods

3.1 Materials

Samples of D–massecuite, final molasses and D-fore worker magma, Seed or slurry for D-massecuite boiling were taken from different process steps. Cans of more than 1.5litre volume, Sample measuring cylinder, sampling cans, beakers, funnels, flasks for sampling and sample preparation. Infrared temperature sensor and thermometer were used temperature measurement

Horne's Dry Lead acetate, watch glass, and Whattmann No. 91 filter paper were used to clarify the samples for analysis of polarization. Weighing balance, Brix hydrometer, 200mm polarrization tubes, saccharimeter, were used for measurement of the samples. Nutsch bomb was used for pressure filtration (5.5-6bar) of mother liquor from D-massecuite at different steps to study the purity drop. HAAKE 6Plus viscometer was used to measure viscosity of massecuite.

The study was conducted on-line using the existing D-vacuum pans, cooling crystallize, massecuite re-heater and D-centrifugal machines at Metahara Sugar Factory.

Figure 3.1: Nutsch Bomb at MSF

Figure 3.2 : Viscometer at MSF

3.2 Study Variables

Cooling Temperature, reheating temperature, brix, polarization (pol), and Purity of final molasses, D massecuite, and liquor extracted from D massecuite at different steps.

3.3 Study Design

Two portions of study were conducted in this thesis. First, assessment of the existing purity drop across each unit of D massecuite processing line (boiling, cooling, reheating and centrifugal separation) was conducted. This was done by intensive nutsch filtration and analysis. After identifying the most significant loss areas, process optimization options were applied for each step as follows;

3.3.1 D -Massecuite Boiling Optimization Design

Response surface methodology (RSM) was adopted in the design of experimental combinations. The main advantage of RSM is the reduced number of experimental runs needed to provide sufficient information for statistically acceptable results (Montgomery 2001). A three-variable (three levels of each variable) Box Behnken experimental design was employed (Montgomery 2001). The parameters and their levels were chosen based on the practical experience and related literature available on molasses exhaustion. The independent variables included massecuite final brix (98-102), purity (54 – 58), and seed volume (1000-2000ml) each at three levels. The higher the brix and seed volume the higher the purity drop, but the viscosity also increases. It was practically observed that discharging from pan is difficult with brix of more than 102^0. With seed volume higher than 2000ml, the grain size of the massecuite becomes very small which can pass through the centrifugal sieves and will cause high sucrose loss with molasses. It is also difficult to handle in crystallizers and pumps due high solidity of the massecuite. Hence seed volume of 2000ml and massecuite brix of 102 are taken as maximum practical values.

Response variables were purity drop between the massecuite and its mother liquor, mother liquor purity and massecuite viscosity.

By inserting minimum and maximum values into Design expert response surface Box Behnken program, the following combinations of the factors have been obtained (Table3.1).

Table 3.1: The Box Behnken experimental design for the three variables with three levels employed for D-Massecuite boiling optimization

Std	Run	Factors		
		Massecuite Purity	Massecuite Brix	Seed Volume (ml)
14	1	56	100	1500
6	2	58	100	1000
9	3	56	98	1000
16	4	56	100	1500
8	5	58	100	2000
12	6	56	102	2000
5	7	54	100	1000
13	8	56	100	1500
15	9	56	100	1500
17	10	56	100	1500
7	11	54	100	2000
3	12	54	102	1500
2	13	58	98	1500
10	14	56	102	1000
1	15	54	98	1500
11	16	56	98	2000
4	17	58	102	1500

3.3.2 Design for Optimization of D-Massecuite Cooling

The effect of cooling temperature (43 – 49^0C) and time (18 - 36hr) on purity drop is studied. Colling of final massecuite is usually done in the temperature range of 40- 50^0C depending on the nature of the massecuite (Rein, 2007). Based on this fact, cooling experiment was aimed to be done in continuous x-lizers at massecuite temperatures of 40- 50 ^{0}C by controlling the cooling water flow rate. But it was found that nutsch filtiration was difficult with the existing compressed air pressure (6.0 bars) for massecuite temperature less than 43^0C. Minimum temperature of 43^0C was considered for the experiment. Maximum and minimum limit of cooling time is taken from Mauritius experience (refer to table 2.2). The experimental design was using design expert response surface with User Defined as in the following table (table 3.2).

Table 3.2: Factors level experimental design for optimization of D- Massecuite cooling

Std	Run	Factor	
		cooling temperature (^{0}C)	cooling time (hr.)
5	1	43.00	27.00
13	2	46.00	27.00
11	3	46.00	18.00
16	4	46.00	27.00
1	5	43.00	18.00
17	6	46.00	27.00
10	7	46.00	36.00
7	8	43.00	27.00
8	9	49.00	27.00
9	10	46.00	18.00
6	11	49.00	27.00
12	12	46.00	36.00
14	13	46.00	27.00
15	14	46.00	27.00
2	15	49.00	18.00
3	16	43.00	36.00
4	17	49.00	36.00

3.3.3 Design for Optimization on Reheater

Two factor, cooling temperature of massecuite (43 – 49^0C) and reheating temperature (50 – 56 ^{0}C) each at three levels and three responses (purity rise across the re-heater, nutsch purity after reheater and massecuite flow rate) were considered. Reheating time is calculated from flow rate and reheater volume. Design Expert-Response surface- 'User Defined' is applied to analyze the data and to find optimum solution. Factors combination is arranged as in the following table by Design Expert software with given maximum and minimum values (table 3.3)

Table 3.3: Factors level experimental for D-Massecuite reheating optimization

Std	Run	Factors	
		Cooling Temp. ^0C	Reheating Temp. ^0C
15	1	46.00	53.00
1	2	43.00	50.00
10	3	46.00	56.00
14	4	46.00	53.00
13	5	46.00	53.00
16	6	46.00	53.00
4	7	49.00	56.00
3	8	43.00	56.00
11	9	46.00	50.00
5	10	43.00	53.00
2	11	49.00	50.00
9	12	46.00	50.00
6	13	49.00	53.00
17	14	46.00	53.00
8	15	49.00	53.00
7	16	43.00	53.00
12	17	46.00	56.00

3.3.4 Design for Optimization on Centrifugal Separation

The amount of water has been optimized based on the final molasses purity and D-fore worker sugar purity. Amount of spray water in flow %Massecuite (1 – 10%) is a factor, and the responses are purity rise across DFW centrifugal machine and DFW magma purity. Design Expert-Response surface- One Factor Design is employed to analyze the data. With given minimum and maximum values of the factor, the following factor levels are designed by the Design Expert program (table 3.4)

Table 3.4: Experimental design for D-massecuite centrifugal separation optimization

Std	Run	**Factor**
		Water% massecuite
20	1	5.5
6	2	8.5
9	3	5.5
7	4	10.0
5	5	7.0
4	6	4.0
3	7	2.5
1	8	1.0
2	9	1.0
8	10	10.0
10	11	5.5
11	12	8.5
12	13	5.5
13	14	10.0
14	15	7.0
15	16	4.0
16	17	2.5
17	18	1.0
18	19	1.0
19	20	10.0

3.4 Experimental Procedure

I. Assessment of Purity Drop for the Existing Conditions

- Sampling of masecuite has been done at pan discharge, crystallizers and re-heaters inlet and outlet with records of temperatures, each centrifugal machines inlet and at the same time final molasses.

- Composite sampling was taken to get representative samples for each process step

- Analysis of each sample of massecuite for pol and brix was done

- Calculation of purity for the massecuite

- Extracting mother liquor from each massecuite sample by nutsch bomb

- Analysis of pol and brix for the extracted mother liquors

- Calculation of purity for the mother liquor (nutsch purity)
- calculation of purity drop; purity drop = masscuite purity – nutsch purity

II. Optimization of Processes

D-Massecuite Boiling Experimental Procedure

- After Preparation of pre-determined volume of slurry (seed) and Conducting purity balance for the feeds liquors, boiling masscuite was done. From the existing two D-pans, experiment was done in one selected pan.
- Representative samples have been taken by composite sampling, sub sampling of massecuite bit by bit until discharging from pan is completed and collecting in one container.
- The massecuite sample was dividing into two; pol, brix and viscosity measurement was done for one sample and mother liquor was extracted from the second sample. Then Analysis of pol and brix was done for the mother liquor.
- Purity and purity drop was calculated for all analysed samples

D-Massecuite Cooling and Re-Heating Optimization Experimental Procedure

- Adjusting of massecuite temperature to the predetermined value
- Sampling of massecuite from inlet and out let of each continuous crystallizers and re-heater.
- Determination of purity, pol and brix of each sample of massecuite
- Extraction of mother liquor from each sample of massecuite using nutch bomb
- Determination of purity for the mother liquor and calculation of purity drop.

D-massecuite Centrifugal Separation Optimization Experimental Procedure

- Setting amount of spray water to the machines as required
- sampling of massecuite at inlet and final molasses at outlet of from centrifugal machine
- extracting of mother liquor from each massecuite sample using nutch bomb

- Purity analysis of mother liquors and final molasses at different spray water.

3.5 Sample Preparation and Analysis

All analyses have been conducted according to ICUMSA (International Commission for Uniform Methods of Sugar Analyses) method for pol and brix and viscosity. Relative Viscosity of massecuite at discharge from pan has been done using the new HAAKE 6Plus viscometer. Purity is obtained from calculation of pol and brix.

3.5.1 Brix Analysis: ICUMSA 4/3-13 (2007)

Brix is the concentration of total dissolved solid in a solution (g/100g solution). Direct determination of brix via brix hydrometer is restricted till about the maximum $30^{0.}$ Above this value, the viscosity become too big and causes the following problems:

- The floating hydrometer reaches very slowly or not at all its equilibrium
- The air will not escape resulting in reading a too low brix hydrometer

Therefore, concentrated solutions will be diluted. Since brix figures are percentage of weights, the dilution there fore to be in a ratio of weights and not of volumes.

The usual dilutions are 1:3, 1:5, 1:6 and 1:10. Therefore, when using brix hydrometer, the samples are diluted as follows:

- 1:3 = 600g sample + 1200g water (dilution factor = 3)
- 1:5 =40g sample + 1600g water (dilution factor = 4)
- 1:6= 400g sample + 2000g water(dilution factor = 6)
- 1:10= 200g sample + 1800 g water(dilution factor = 10)

$$\text{DF} = \text{Dilution factor} = \frac{\text{sample weight} + \text{water weight}}{\text{sample weight}} \qquad (3.1)$$

Determination of Brix of D Massecuite (DF =6)

A) Weight out 400g in 1.5 liter can of well mixed sample of the massecuite and dilute it by 2000g of water and then dissolve it until a sugar crystals (grains) are eliminated.

B) Then add the prepared solution to brix measuring cylinder

C) Immerse the Brix hydrometer spindle dipper in to the solution and wait until the hydrometer stays at rest

D) Allow sufficient time for brix hydrometer spindle to reach the same temperature as the solution.

E) Read uncorrected brix from the brix spindle

F) Read the temp. of the solution from thermometer on the hydrometer

G) Calculate corrected brix using temperature Correction factor from table.

Determination Brix for Final Molasses and D- Massecuite Nutsch (DF=10)

Follow the same procedure as for D- Massecuite except that 200 g sample and 1800g distilled water are used in this case.

Brix Determination for D-Fore worker (DFW) Magma (DF=6)

The same procedure is used as for the brix analysis of D-Massecuite.

3.5.2 Pol (Apparent sucrose) Analysis: ICUMSA 1/2/3/9-1 (2007)

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 (saccharimeter) 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 of a sugar solution is the resultant optical rotation of sucrose and other optically active substances mostly glucose and fructose. The “pol” values normally give an approximation below the real sucrose 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.

A sugar solution has to be clarified before its pol is determined. Horne’s basic lead acetate is a commonly used clarifying agent. Excess lead acetate should not be used for any excess will precipitate some of the fructose out of the solution, resulting in an increase dextrarotatory or high polarization, and will affect the polarization of sucrose.

Procedure for Pol Determination in D Masecuite (DF=6)

a) Take out 100ml diluted D-masecuite solution in a 1000ml flask from the solution prepared for brix analysis

b) Add 6-10g of lead acetate and mix well

c) Filter through whatmann No. 91 filter paper by conning the funnel with watch glass

d) Discard the 1st 2-3ml of the filtrate and collect enough filtrate for pollirization

e) rinse a 200ml pol tube at least three times with the filtrate and then fill the tube with filtrate checking that the liquid column should be free from any air bubbles

f) then polarize it by putting in saccharimeter and take pol reading (uncorrected pol)

g) Correct the pol using Schmitz table.

The Schmitz table is prepared from Schmitz equation (eqn. 3.2).

$$pol = \frac{Saccharimeter\ reading}{3.83545 + .014752xbrix + 0.0000576xbrix^2} \tag{3.2}$$

Pol Determination for final molasses and D Masecuite Nutsch (DF=10)

By taking respective sample from diluted samples prepared for Brix reading, pol analyses of fanal molasses and D –nutsch follow similar procedure as for the pol analysis of D-massecuite except that 4-6g lead acetate is used instead of 6-10g.

Pol Determination for D-Fore worker (DFW) Magma (DF=6)

By taking from prepared sample for brix measurement and using about 5g of lead acetate, similar procedure is followed as for pol analysis of final molasses.

3.5.3 Determination of purity

Purity is the precent ratio of pol to brix. It is obtained by calculation after analysis of pol and brix.

Purity = Pol/Brix*100%

3.5.4 Determination of Viscosity for Massecuite: ICUMSA Method SPS-5 (1994)

The viscosity of the D-massecuite samples was determined at as soon as discharged from pan (at temperature of about 65 °C) using the newly purchased **HAAKE 6Pluss viscometer,** at 10 – 20 rpm with spindle R7. The speed was chosen to give a torque reading between 20 and 80 %.

Slurry preparation

Slurry (seed), a suspension of finely ground sugar in alcohol, is used for starting of C- and D-massecuite boiling. Slurry is prepared by grinding commercial sugar in alcohol by ball mills. In Metahara sugar Factory, 4kg commercial sugar and 9kg alcohol or mehylated spirits are mixed and ground by ball mill for 6-8hrs. Slurry containing about 4µm particle size is obtained.

4 Results and Discussion

4.1 Assessment of Purity Drop for the Existing Conditions (Without Applying Optimization)

Sampling and purity analysis for massecuite and its respective mother liquor at pan discharge, inlet and outlet of each continuous crystallizers, reheaters and centrifugal machine was done. About 15 experiments were executed for each point to get reliable average values. The average values of the figures are as in the following table (tables 4.1-4.3): the detail analysis result data are annexed to this paper at the last pages (appendix-A).

Table 4.1: Average purity drop across 1st battery crystallizers

Unit	Temperature ^0C	Nutsch purity	Pty drop
*Discharge from pan (average massecuite purity =57.35, brix = 101.3)	65.3	36.0	21.35
12 outlet/13 inlet	54.7	33.72	2.28
13 outlet /14 inlet	51.2	32.18	1.54
14 outlet /reheater inlet	48.8	31.66	0.52
Reheater outlet	53.6	32.52	-0.86
Centrifugal machine outlet (final molasses)	52.5	34.09	-1.57

Table 4.2: Average purity drop across 2nd battery crystallizers

Unit	Temperature ^{0} C	Nutsch purity	Pty drop
pan	65.3	36.10	21.25
15 outlet/16 inlet	58.50	33.13	2.97
16 outlet /17 inlet	52.10	32.48	0.65
17outlet /reheater inlet	50.60	32.02	0.46
Reheater outlet	55.82	33.09	-1.07
Centrifugal machine outlet (final molasses)	55.60	35.61	-2.52

Table 4.3:Average purity drop for both battery crystallizers

Unit	Temperature (^{0}C)	Nutch purity	Pty drop
Pan	65.3	36.05	21.30
1st crystallizer	56.6	34.425	2.625
2nd crystallizer	51.65	32.33	1.1
3rd crystallizer	49.7	31.84	0.49
Reheater	54.71	32.81	-0.97
Centrifugal machine	54.05	34.86	-2.05

- Purity drop at boiling = 21.30
- Purity drop across cooling crystallizers = 4.21
- Purity **rise** across reheater =0.97
- Purity rise across centrifugal machines= 2.05
- Overall purity drop =21.3+4.21- 0.97-2.05 = 22.32 units

From the assessment it is seen that the purity drop across crystallizers is lower, only about 4.21 against standard of 6 – 8 in modern vertical crystallizers. The purity rise across centrifugal machine is acceptable (less than 2). The purity rise across the reheaters is also higher than the standard values of less than 0.5 units.

Assessment of the existing operation shows that there was loose control of the operation parameters at all D-massecuite processing steps and resulted in high loss of sucrose with final molasses. The average brix and purity of D-massecuite after boiling were 101.3 and 57.35 respectively, and the purity of mother liquor extracted from this massecuite (nutsch purity at pan discharge) was 36.05. The massecuite was boiled with slurry volume of 2000ml. The nutsch purity obtained was higher than the required and ultimately contributed to the increase in final molasses purity. Therefore, optimization of boiling parameters (massecuite purity, seed volume and massecuite brix) was necessary on boiling to minimize the nutsch purity.

At massecuite cooling, the final cooled temperature of the massecuite was only 49^{0}C which resulted in low purity drop across crystallizers (only 4.21 units against the standard of more than 6 units). Here, the assessment results indicated that there was room for improvement at massecuite cooling to minimize sucrose loss with final molasses by increasing purity drop across cooling crystallizers. This can be achieved by cooling the masecuite to lower possible temperatures. Based on this fact, process Optimization was carried out on massecuite cooling to obtain optimum cooling temperature and time at which high purity drop is achieved.

Considering the assessment study D-massecuite reheating, there was an average mother liquor purity rise of 0.97 across reheaters at reheating temperature was 54.71^0C. The purity rise obtained was higher than the required standard of 0 – 0.50 units. The purity rise should be reduced by controlling and optimizing the necessary parameters (cooling and reheating temperatures).

For D-masecuite centrifugal separation, the average purity rise across centrifugal machines was 2.05 units. Purity rise in the “D” centrifuges should not be higher than 3 and it is better if it is lower than 2 (Rein, 2007). The purity rise obtained from the assessment study was acceptable value when compared with the standard limits recommended on literatures. But during this test, the DFW sugar purity was on the low side (82 – 83), which will cause high recirculation of non sucrose back to the boiling house. Therefore optimization was required to improve the DFW magma (sugar) purity to the standard value of 84 or more without incurring high purity rise the mother liquor across centrifugal machines.

The final molasses purity during the assessment study was also 34.86 which show a high sucrose loss.

4.2 Optimization Results

4.2.1 D-Massecuite Boiling Optimization

Based on the factors combination in table 3.1, triplicate experiment was done and average values of the responses at each factor level combination were taken. Boiling of D- massecuite was done in one pan, the massecuite and its mother liquor was analyzed for purity. The corresponding viscosity was also measured. The average result of mother liquor purity, purity drop and viscosity obtained for each factors combination were given in table 4.4.

Table 4.4: Box Behnken experimental design and the responses for D-Massecuite boiling optimization

Std	Run	Factors			Responses		
		Massecuite purity	Massecuite brix	Seed volume	Purity drop	Nutsch purity	Viscosity (Pa.S)
14	1	56	100	1500	19.52	36.48	215.6
6	2	58	100	1000	20.35	37.63	123.1
9	3	56	98	1000	18.74	37.26	136.5
16	4	56	100	1500	19.54	36.46	215.8
8	5	58	100	2000	21.13	36.87	174.3
12	6	56	102	2000	20.52	35.48	287.9
5	7	54	100	1000	16.79	37.21	232.9
13	8	56	100	1500	19.55	36.45	222.5
15	9	56	100	1500	19.52	36.48	211.1
17	10	56	100	1500	19.54	36.46	212.3
7	11	54	100	2000	17.565	36.435	301.1
3	12	54	102	1500	17.74	36.26	294.0
2	13	58	98	1500	20.36	37.64	123.0
10	14	56	102	1000	19.59	36.41	207.5
1	15	54	98	1500	16.86	37.14	239.5
11	16	56	98	2000	19.43	36.57	220.1
4	17	58	102	1500	21.41	36.59	182.3

4.2.1.1 Data Analysis for D- Massecuite Boiling optimization

Data were modeled by multiple regression analysis and the statistical significance of the terms was examined by analysis of variance for each response. The statistical analysis of the data and three dimensional plotting were performed using Design Expert Software (Stat-Ease 2000). The adequacy of regression model was checked by R^2, Adj R^2, Pred R^2, Adeq Precision and *F*-test (Montgomery 2001). The significance of *F* value was judged at 95% confidence level. The regression coefficients were then used to make statistical calculation to generate three-dimensional plots from the regression model. The degree of relationship between the variables was also checked by using correlation matrix of the factors and response variables.

I. Data Analysis for Response1 - Purity Drop at Boling

Quadratic model is suggested by the design program for this response to test for its adequacy and to describe its variation with independent variables. From ANOVA test in table 3.9, the Model F-value of 15363.10 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Table 4.5: ANOVA test for purity drop at D-Massecuite boiling

Source	Sum of Squares	df	Mean Square	F Value	p-value	
					Prob > F	
Model	29.90043	9	3.32227	15363.1	< 0.0001	Significant
A-pty	25.54338	1	25.54338	118119.7	< 0.0001	
B-bx	1.872113	1	1.872113	8657.168	< 0.0001	
C-seed	1.260078	1	1.260078	5826.951	< 0.0001	
AB	0.007225	1	0.007225	33.4104	0.0007	
BC	0.0144	1	0.0144	66.5896	< 0.0001	
A^2	1.166613	1	1.166613	5394.743	< 0.0001	
B^2	0.030332	1	0.030332	140.2619	< 0.0001	
C^2	0.010058	1	0.010058	46.5108	0.0002	
Residual	0.001514	7	0.000216			
Lack of Fit	0.000794	3	0.000265	1.469907	0.3494	not significant
Pure Error	0.00072	4	0.00018			
Cor Total	29.90195	16				

Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, BC, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The "Lack of Fit F-value" of 1.47 implies the Lack of Fit is not significant relative to the pure error. There is a 34.94% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table 4.6: Post ANOVA statistics for purity drop at D-Massecuite boiling

Std. Dev.	0.014705		R-Squared	0.99994938
Mean	19.30324		Adj R-Squared	0.99988429
C.V. %	0.076181		Pred R-Squared	0.99953766
PRESS	0.013825		Adeq Precision	410.458038

From tabe 3.10, the "Pred R-Squared" of 0.9995 is in reasonable agreement with the "Adj R-Squared" of 0.9999. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 410.458 indicates an adequate signal. This model can be used to navigate the design space (Montgomery 2001).

II. Data Analysis for Response2- Nutsch Purity at Boiling

Quadratic model is suggested by the design program for this response. All statistical analysis including ANOVA test, post ANOVA statistics, lack of fit test, Normal plot of Residuals etc are done for the Nutsch purity data. All the tests indicated that the model is statistically acceptable.

From ANOVA test, the Model F-value was 2125.12 which implies that the model is significant. Values of "Prob > F" was less than 0.0500 indicating that model terms A, B, C, AB, BC, A^2, B^2, $C^2$are significant. The "Lack of Fit F-value" of 1.84 implies the Lack of Fit is not significant relative to the pure error. Non-significant lack of fit is good -- we want the model to fit.

Table 4.7 : Post ANOVA statistics for nutsch purity at D-Massecuite boiling

Std. Dev.	0.015647		R-Squared	0.999634
Mean	36.69559		Adj R-Squared	0.999164
C.V. %	0.042639		Pred R-Squared	0.996365
PRESS	0.017025		Adeq Precision	179.3671

The "Pred R-Squared" of 0.9964 is in reasonable agreement with the "Adj R-Squared" of 0.9992. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. This ratio of 179.367 indicates an adequate signal. This model can be used to navigate the design space.

III. Data Analysis for massecuite viscosity (Response3)

Linear model is suggested by the design program for this response. All statistical analysis including ANOVA test, post ANOVA statistics, lack of fit test, Normal plot of Residuals etc are done for the Nutsch purity data. All the tests indicated that the model is statistically acceptable. All the Data analyses results indicate that the data are statistically valid.

4.2.1.2 Correlation matrix for D-massecuite Boiling Optimization

The correlation matrix is one of the most common and most useful statistics. The correlation coefficient measures the strength of a linear relationship between the variables. The correlation coefficient is always between -1 and +1. The closer the correlation is to +/-1, the closer to a perfect linear relationship.

Table 4.8: Correlation matrix of factors and responses for D- boiling Optimization using SPSS

	A- Massecuite purity	B- Massecuite brix	C-Seed volume	R1- Purity drop	R2- Nutsch purity	R3- Viscosity (Pa.S)
A- Massecuite purity	1.000	0.000	0.000	0.924**	0.275	-0.769**
B- Massecuite brix	0.000	1.000	0.000	0.250	-0.632**	0.418
C- Seed volume	0.000	0.000	1.000	0.205	-0.515*	0.469
F1- Purity drop	0.924**	0.250	0.205	1.000	-0.113	-0.499*
F2- Nutsch purity	0.275	-0.632**	-0.515*	-0.113	1.000	-0.746**
F3- Viscosity (Pa.S)	-0.769**	0.418	0.469	-0.499*	-0.746**	1.000

** Correlation is significant at the 0.01 level (2-tailed)
* Correlation is significant at the 0.05 level (2-tailed)

Table 4.9: Correlation Matrix of Factors [Pearson's r] for D- massecuite boiling optimization using Design Expert-RSM

	A	B	C	AB	AC	BC	A^2	B^2	C^2
A	1.000								
B	0.000	1.000							
C	0.000	0.000	1.000						
AB	0.000	0.000	0.000	1.000					
AC	0.000	0.000	0.000	0.000	1.000				
BC	0.000	0.000	0.000	0.000	0.000	1.000			
A^2	0.000	0.000	0.000	0.000	0.000	0.000	1.000		
B^2	0.000	0.000	0.000	0.000	0.000	0.000	0.056	1.000	
C^2	0.000	0.000	0.000	0.000	0.000	0.000	0.056	0.056	1.000

Where: A, B and C- are factors
Ri - are responses

Almost all the factor- to-factor correlation is zero. The factors- to- responses correlation are different from zero.For the factors- to - factors correlation,Off-diagonal values close to zero are better.

4.2.1.3 Model Eqaution for D-Massecuite Boiling Optimization

Model equations are given in terms of coded factors and actual factors. Coded factors indicate when the minimum and maximum values of the factors are represented by -1 and +1 respectively instead of their actual values.

I. Purity drop at Boiling

Equation in terms of coded factors:

$$\text{purity drop} = 19.53 + 1.79A + 0.48B + 0.40C + 0.04AB + 0.000125AC + 0.060BC - 0.53A^2 + 0.085B^2 - 0.049\,C^2 \qquad (4.1)$$

Equation in terms of actual factors:

$$\text{purity drop} = -188.202 + 14.568Pty - 4.68\,Bx - 4.690x10^{-3}S' + 0.0106PtyBx + 1.250x10^{-6}PtyS' + 6.000\,x10^{-5}Bx\,S' - 0.132Pty^2 + 0.0212Bx^{2} - 1.955x10^{-7}S'^{2} \qquad (4.2)$$

II. Nutsch Purity at Boiling

Equation in terms of coded factors:

$$\text{Nutch purity} = 36.47 + 0.21\,A - 0.48B - 0.39C - 0.043AB + 3.750x10^{-3}AC - 0.060BC + 0.52A^2 - 0.082B^2 + 0.046C^2 \qquad (4.3)$$

Equation in terms of actual factors:

$$\text{Nutch purity} = 192.952 - 13.506Pty + 4.562Bx + 4.444x10^{-3}S' - 0.0106PtyBx + 3.75x10x^{-6}PtyS' - 6x10^{-5}BxS' + 0.13097Pty^{2} - 0.02059Bx^2 + 1.85500x10^{-7}S'^{2} \qquad (4.4)$$

III. D-massecuite Relative viscosity

Equation in terms of coded factors:

$$\text{Relative viscosity} = 211.74 - 58.10A + 31.58B + 35.43C \qquad (4.5)$$

Equation in terms of actual factors:

$$\text{Relative viscosity} = 153.51029 - 29.05000Pty + 15.78750Bx + 0.070850S' \qquad (4.6)$$

where: A = Coded value for purity
B = Coded value for brix
C = Coded value for seed volume

Pty = massecuite purity at pan discharge [54 – 58^0]

Bx= massecuite brix [98 – 102^0]

S’ = Seed volume (ml) [1000 – 2000ml]

Diagnostic Test for the Responses

All diagnostic plots are also tested for all responses for adequacy of the models (normal plot of residuals, residuals vs predicted value, residuals vs factor, box cox plot, studentized residuals, leverage, etc.). Fore example figure 4.1 shows how precisely the purity drop is modeled, because all the points line up nicely and the deviation of points for purity drop from normality is insignificant. Similar results were observed for the remaining responses (nutsch purity and relative viscosity).

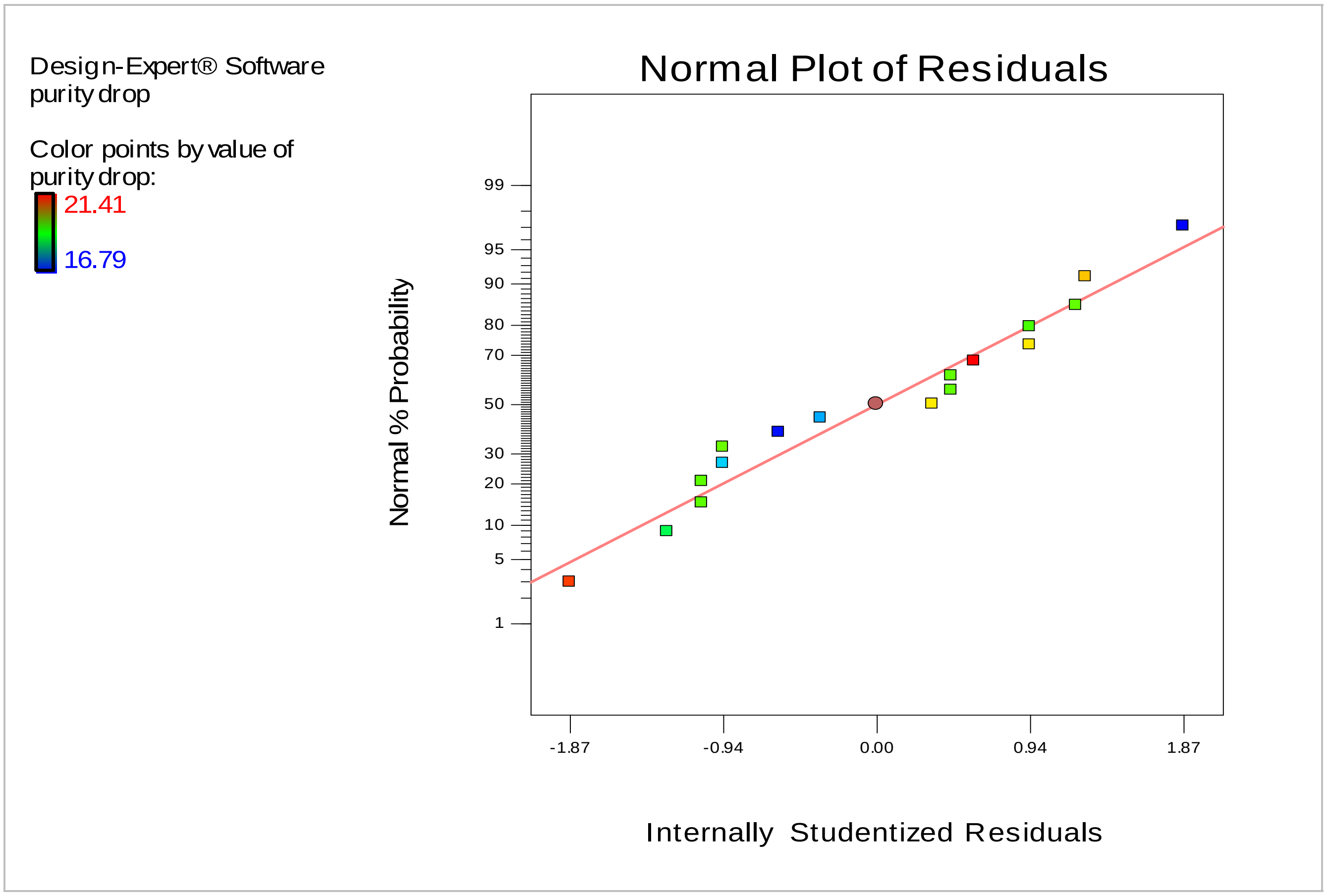

Figure 4.1: Normal plot of residuals for purity drop at D-Massecuite boiling

4.2.1.4 Response Surface Plots Analysis for D-Massecuite Boiling Optimization

The effect of different treatment parameters on purity drop at boiling is given in Table 3.8. To aid visualization of variation in responses with respect to processing variables, series of three-dimensional response surfaces were drawn using Design Expert Software (Stat-Ease 2000). Since the present study involved three variables, it was necessary to fix the value of one variable in order to see the effect of two variables on the response. The non-variant parameter was set at the optimum point and put in Equations (4.1 – 4.6). The relationships developed between dependent variable and independent variables were used to plot response surfaces and representative plots are shown in Figures 4.2–4.4.

I. Response 1- Purity Drop on Boiling

a. The effect of massecuite brix and purity on purity drop at constant seed volume

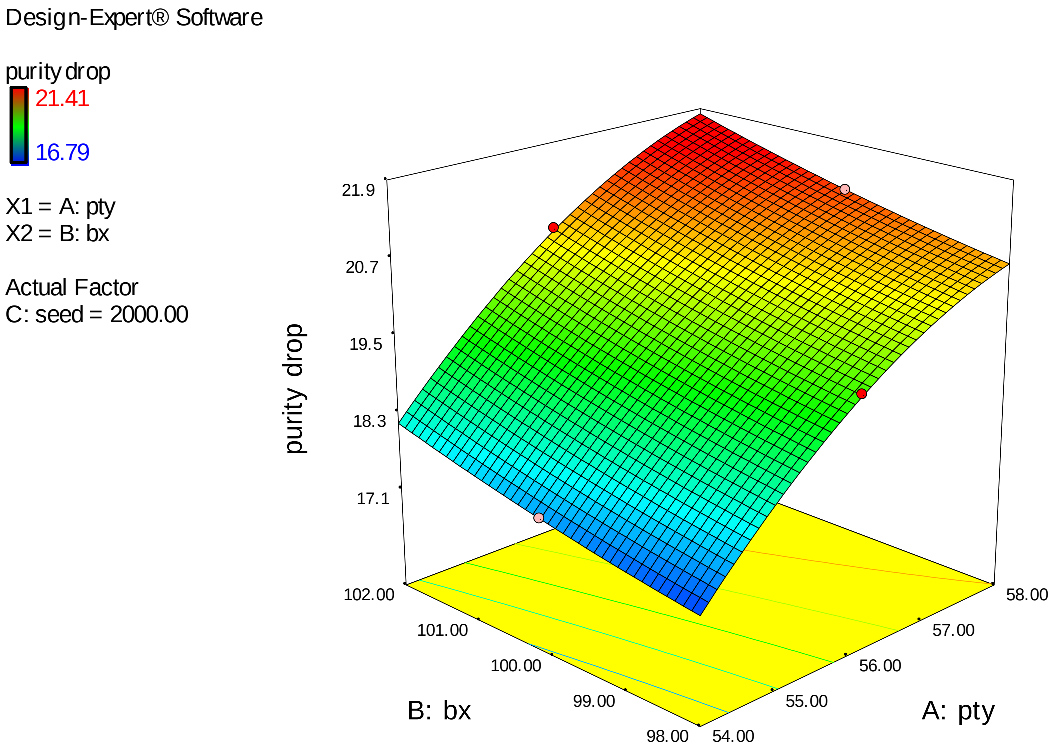

Figure 4.2: Response surface plot for Purity drop at boiling (with constant seed volume)

Purity drop increases with increase in massecuite brix and purity at constant seed volume (optimum seed volume). But there is a limit to the brix and purity of the massecuite. At higher purity of the massecuite, the mother liquor purity (nutsch purity) is also higher and it is difficult to get low final molasses purity. At higher brix the viscosity of the massecuite will be too high and it is difficult to handle the massecuite. Hence there is an optimum point to compromise all the conditions.

b. The effect of purity and seed volume on purity drop at constant brix of massecuite

Purity drop increase with both seed volume and purity at constant massecuite brix. There is sharp increase with purity than seed volume (figure 4.3).

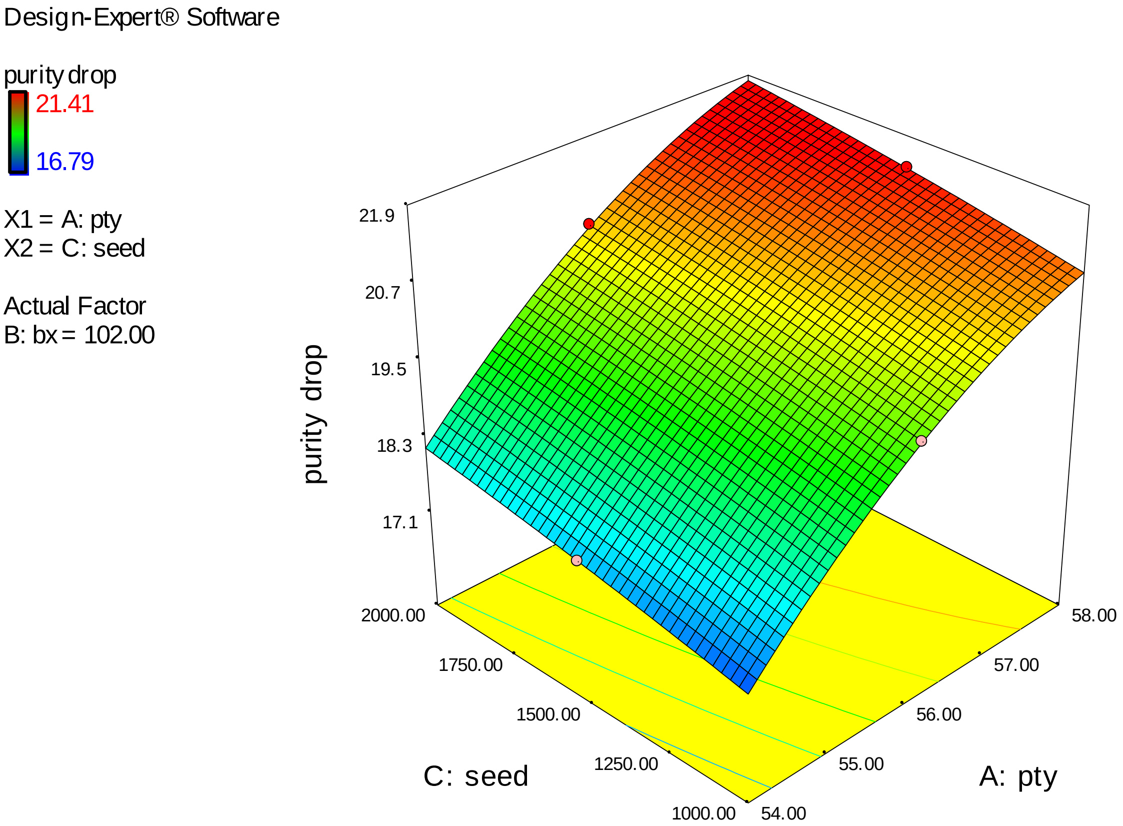

Figure 4.3: Response surface plot for purity drop at boiling (with constant massecuite brix)

c. The effect of brix and seed volume on purity drop at constant purity of d- massecuite

Examination of the response surface plot shown on figure15 indicates that, purity drop increases with increase in seed volume and massecuite brix. At higher brix, more sucrose in the solution will be absorbed to the crystals due to high supersaturation and this will increase purity drop between the massecuite and its mother liquor (nutsch). More seed volume implies that there is large number of sugar crystals available in the solution for the sucrose molecule to be absorbed to. This is why the purity drop increases with seed volume.

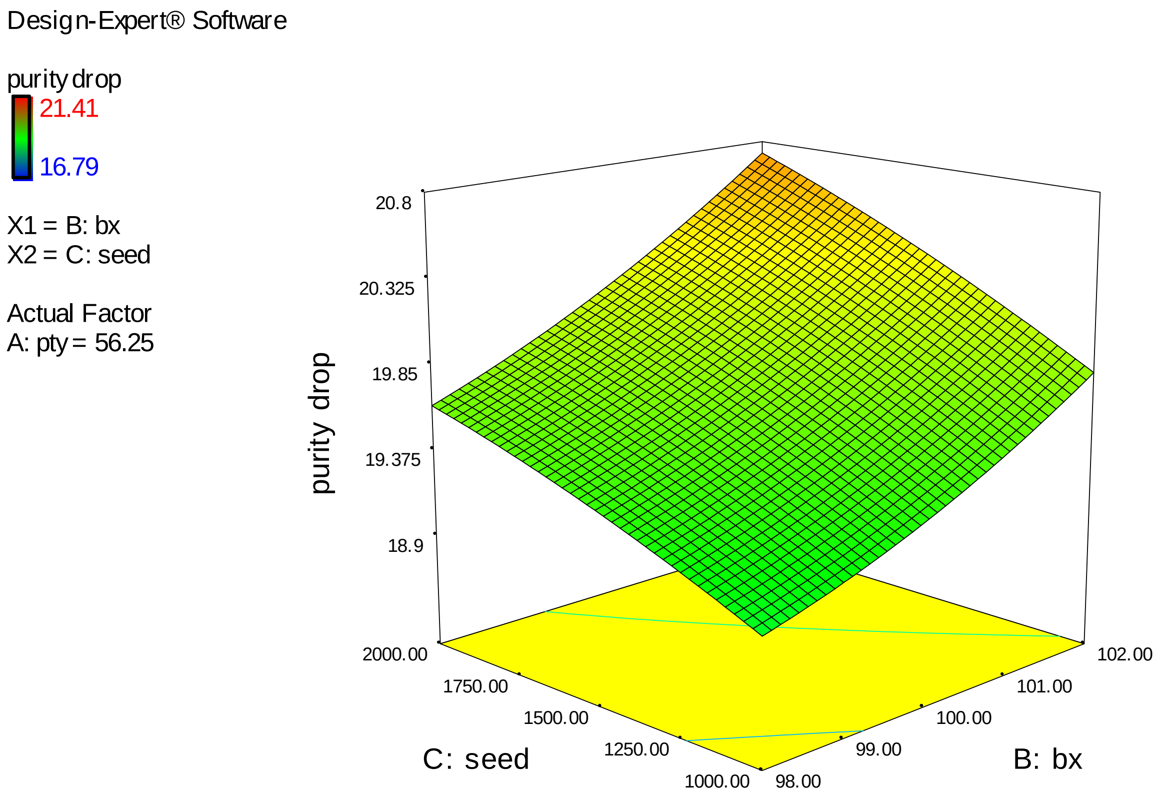

Figure 4.4: Response surface plot for purity drop at boiling with constant D-Massecuite

II. Response 2- Nutsch Purity at Boiling

The effects variation of factors on nutsch purity is also observed. Figure 4.5 is shown as an example on this paper. The plot shows that nutsch purity increases with massecuite brix at constant seed volume and massecuite purity. At constant massecuite brix and seed volume, the nutsch purity increases with increases in massecuirte purity for the massecuite purity of above 56 and then decreases with decreases in massecuite purity for massecuite purity below 56. Minimum nutsch purity is obtained at massecuite purity of about 56. Factors interaction plot also clearly indicates the effect of massecuite purity on nutsch purity at discharge (figure 4.6)

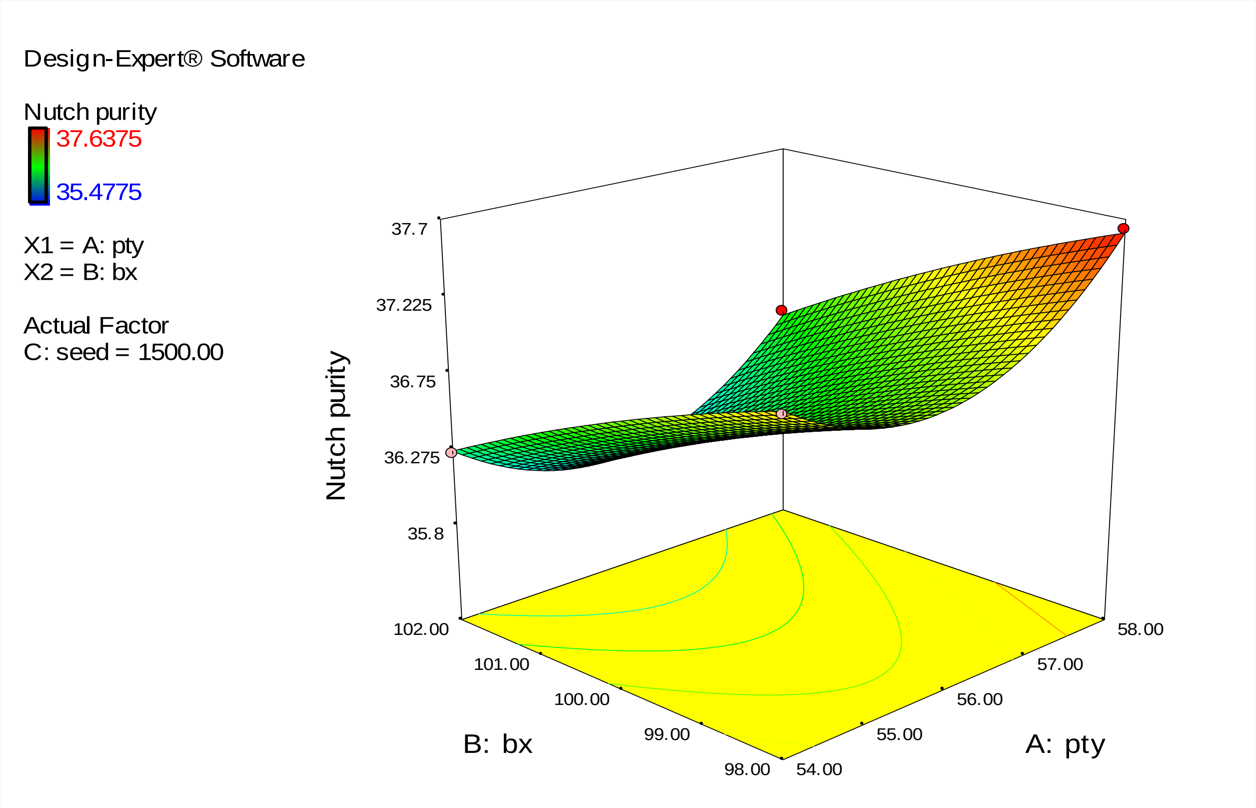

Figure 4.5: Response surface plot for nutsch purity of D-massecuite boiling (constant seed volume)

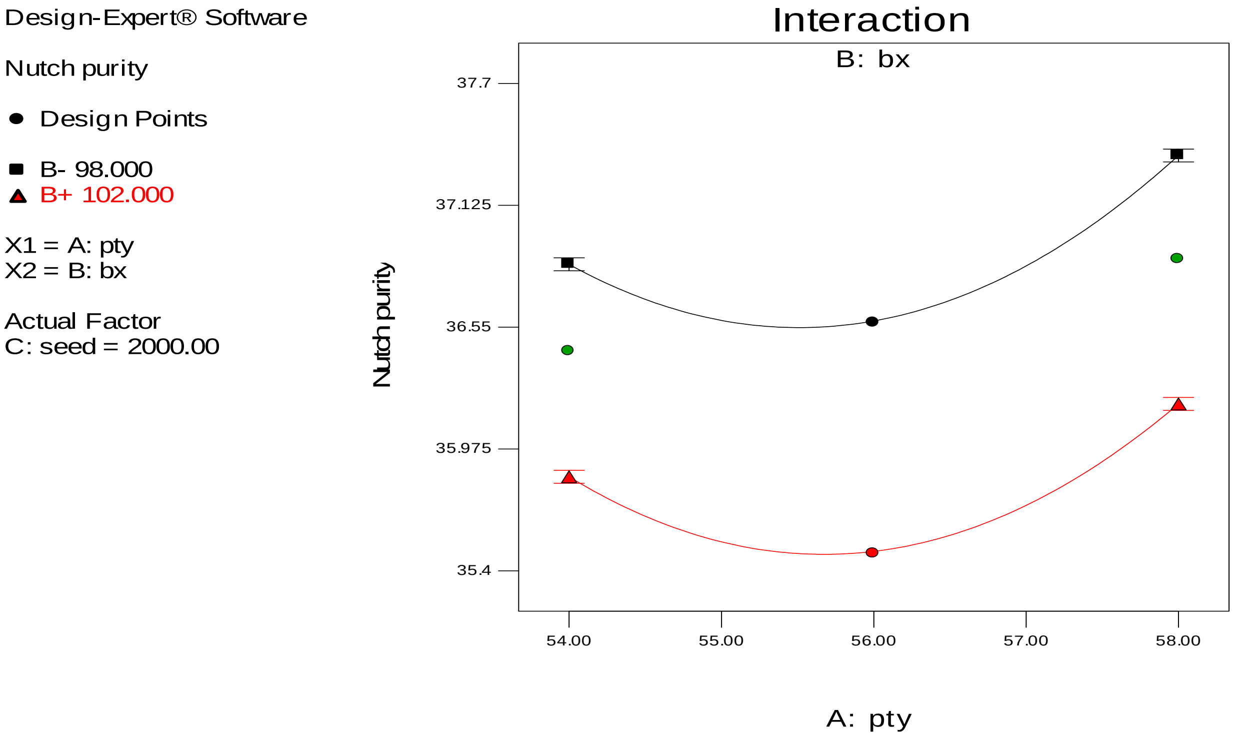

Figure 4.6: Response surface plot for nutsch purity of D-massecuite boiling (constant seed volume)

III. Response 3- Massecuite Viscosity

The effects variation of factors on massecuite viscosity was also observed from the model equations and response surface plots. It is observed that the viscosity of the massecuite has a linear relationship with all the factors. It increases linearly with brix and seed volume and decreases with the purity of massecuite. The following is shown as example on this paper.

The viscosity of D-massecuite increases almost linearly with increase in brix and decrease in purity. At low purity the proportion of non sucrose impurities is high causing increase in the viscosity of the massecuite.

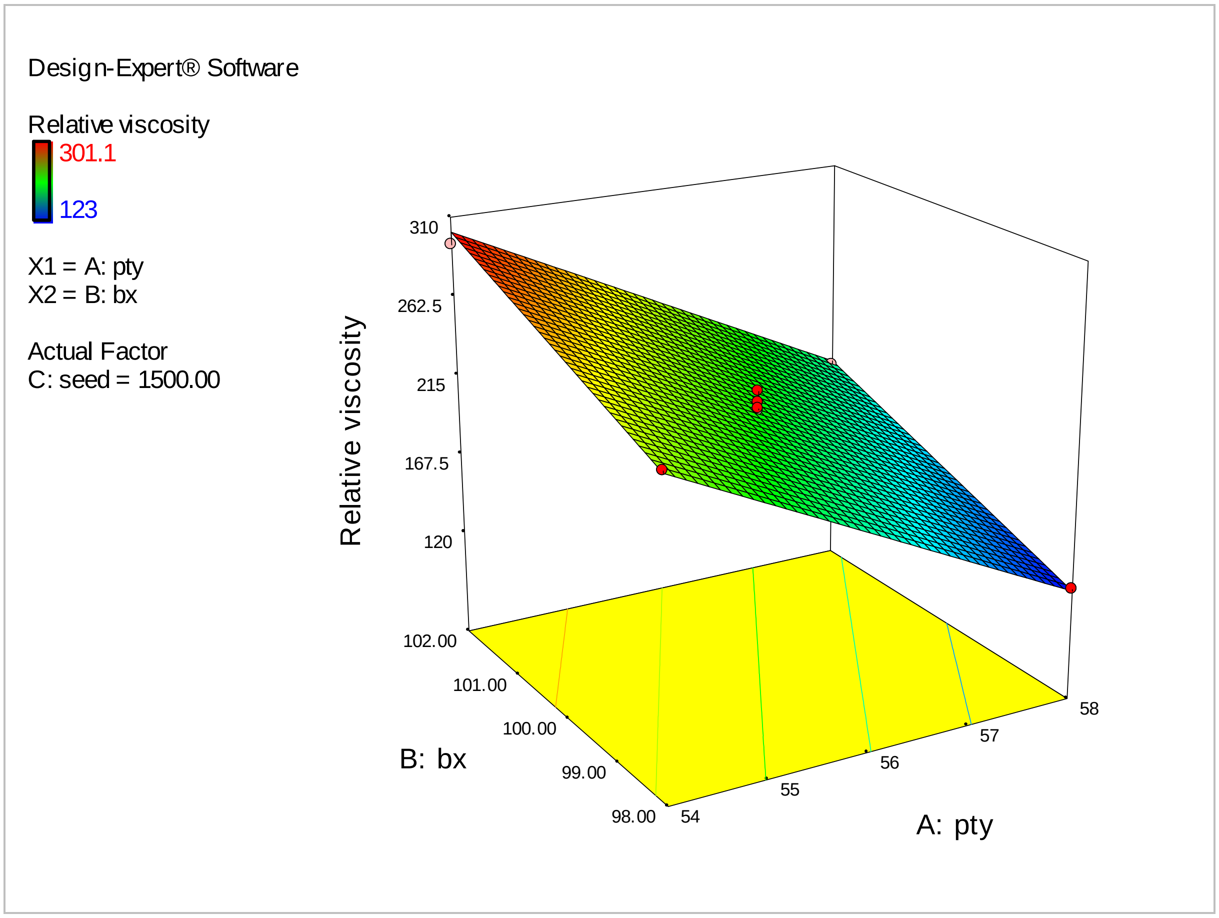

Figure 4.7: Response surface plot for relative viscosity of D-Massecuite at boiling (constant seed volume)

4.2.1.5 Optimization Solution for D-Massecuite Boiling

The optimum parameter values for D-Massecuite at boiling; purity drop, nutsch purity and viscosity were calculated by partially differentiating Equations (4.2), (4.4) and (4.6) with respect to each parameter and equating to zero (Montgomery 2001). The optimization was to get maximum purity drop, minimum nutsch purity and minimum viscosity as much as possible. The Second response (nutsch purity at boiling) was considered as an important response and more weight is given to it since it is the major determining factor of the final molasses purity. 12 solutions are obtained. The solution with maximum desirability is selected as an optimum solution (table 4.10). Massecuite purity of 56.25^0, brix of 102^0 and seed volume of 2000ml are selected as optimum parameters to obtain optimum purity drop of 20.732^0, nutsch purity of 35.52 and massecuite relative viscosity of 271.44Pa.S.

Table 4.10: Optimization solutions for D-Massecuite boling experiment

Number	F1-pty	F2-bx	F3-seed	R1-purity drop	R2-Nutsch purity	R3-Relative viscosity	Desirability	Number
1	56.25	102	2000	20.73198	35.52168	271.4472	0.537518	Selected
2	56.22	102	2000	20.70835	35.51758	272.252	0.537329	
3	56.2	102	1999.94	20.69209	35.51494	272.7964	0.536962	
4	56.25	102	1995.8	20.72762	35.52448	271.1955	0.535371	
5	56.24	101.97	2000	20.71022	35.52867	271.4662	0.528605	
6	56.45	102	2000	20.89961	35.55803	265.5261	0.528104	
7	56.35	101.98	2000	20.80544	35.54578	268.2579	0.527345	
8	56.24	101.94	2000	20.70614	35.54058	270.7603	0.518368	
9	56.18	102	1909.66	20.60595	35.57859	267.0605	0.488794	
10	56.11	101.85	2000	20.56052	35.55569	273.0817	0.485805	
11	56.05	102	1813.69	20.41543	35.63765	264.0833	0.432122	
12	55.73	102	1679.95	20.00834	35.72908	263.7788	0.339007	

Fi- factors, Ri- Responses

4.2.2 D-Massecuite Cooling Optimization

Study of purity drop across continuous crystallizer was conducted by using nutsch analysis at different cooling temperature and time in order to observe the trend of purity drop with temperature and cooling time, and to determine the optimum values. Cooling temperature is varied by changing the flow rate of cooling water to cooling discs and cooling time is varied by changing the massecuite flow rate to the crystallizer under study. Laboratory analyses were done for different factors level combinations as per the design in table 3.2. The laboratory analyses results of the responses data are depicted in table 4.11.

Approach to this experiment

There are two batteries of crystallizers in MSF. One set of crystallizer (1st battery) was used for the study while the 2nd battery was working under accustomed condition. Before starting of the experiment, massecuite and cooling water flow were adjusted at different rates by manipulating the valves on the discharge lines in order to get the desired massecuite cooling temperature at desired cooling time. After several trials, the predetermined values were obtained at different valves openings. The valve opening positions and the water pressure on the cooling water discharge line were noted corresponding to the massecuite cooling temperature and cooling time. The cooling time was calculated from the known volume of the crystallizers and flow capacity of centrifugal machine in terms of current load.

Table 4.11: Response surface User Defined experimental design and the responses for D-Massecuite cooling experiment

		Factor		Response
Std	Run	cooling temperature (^{0}C)	cooling time (hr.)	Purity drop
5	1	43.00	27.00	6.53
13	2	46.00	27.00	5.41
11	3	46.00	18.00	3.98
16	4	46.00	27.00	4.11
1	5	43.00	18.00	5.7
17	6	46.00	27.00	4.87
10	7	46.00	36.00	6.24
7	8	43.00	27.00	5.24
8	9	49.00	27.00	4.14
9	10	46.00	18.00	5.61
6	11	49.00	27.00	4.02
12	12	46.00	36.00	6.17
14	13	46.00	27.00	5.52
15	14	46.00	27.00	4.27
2	15	49.00	18.00	2.79
3	16	43.00	36.00	6.87
4	17	49.00	36.00	5.17

4.2.2.1 Data Analysis for Massecuite Cooling optimization

Data were modeled by multiple regression analysis and the statistical significance of the terms was examined by analysis of variance for each response. The statistical analysis of the data and three dimensional plotting were performed using Design Expert Software (Stat-Ease 2000). The adequacy of regression model was checked by R^2, Adj R^2, Pred R^2, Adeq Precision and F-test (Montgomery 2001). The significance of F value was judged at 95% confidence level. The regression coefficients were then used to make statistical calculation to generate three-dimensional plots from the regression model.

Response1: Purity Drop across Cooling Crystallizers

Linear model is suggested for this response**.** From the Analysis of variance (ANOVA) test in table 3.14, the Model F-value of 17.45 implies the model is significant.

The "Lack of Fit F-value" of 0.56 implies the Lack of Fit is not significant relative to the pure error. There is a 75.15% chance that a "Lack of Fit F-value" this large could occur dueto noise. Non-significant lack of fit is good -- we want the model to fit.

Table 4.12: ANOVA table of purity drop for D-Massecuite cooling optimization test

Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob > F	
Model	13.51816	2	6.759081	17.45429	0.0002	significant
A-Cooling Temperature	8.44605	1	8.44605	21.81063	0.0004	
B-Cooling time	5.072113	1	5.072113	13.09795	0.0028	
Residual	5.421426	14	0.387245			
Lack of Fit	1.605356	6	0.267559	0.560911	0.7515	not significant
Pure Error	3.81607	8	0.477009			
Cor Total	18.93959	16				

From the post ANOVA statistics, Table 4.13, the"Pred R-Squared" of 0.5947 is in reasonable agreement with the "Adj R-Squared" of 0.6729. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. This ratio of 13.953 indicates an adequate signal. This model can be used to navigate the design space.

Table 4.13: Post ANOVA statistics for purity drop on D-Massecuite cooling experiment

Std. Dev.	0.62229
Mean	5.096471
C.V. %	12.21021
PRESS	7.676022

R-Squared	0.713752
Adj R-Squared	0.672859
Pred R-Squared	0.59471
Adeq Precision	13.95296

4.2.2.2 Correlation Matrix for D-Massecuite Cooling Optimization

Table 4.14: Correlation matrix of factors and responses using SPSS

	A- Cooling temp.	B- Cooling time	R- Purity drop
A- Cooling temp.	1.000	0.000	-0.668**
B- Cooling time	0.000	1.000	0.517*
R- Purity drop	-0.668**	0.517*	1.000

** Correlation is significant at the 0.01 level (2-tailed).
* Correlation is significant at the 0.05 level (2-tailed)

Table 4.15: Correlation Matrix of Factors [Pearson's r] using Design Expert-RSM

	A	B	AB	A^2	B^2
A	1.00				
B	0.000	1.00			
AB	0.000	0.000	1.00		
A^2	0.000	0.000	0.000	1.00	
B^2	0.000	0.000	0.000	0.056	1.00

Where: A, and B - are factors (independent variables)
R - is response (dependent variable)

Almost all the factor- to-factor correlation is zero. The factors- to- responses correlation are different from zero.For the factors- to - factors correlation,Off-diagonal values close to zero are better.

4.2.2.3 Model Equations for Purity Drop on Massecuite Cooling

Equation in terms of coded factors:

$$\text{purity drop} = +5.10 - 1.03\,A + 0.80\,B \tag{4.7}$$

Equation in terms of actual factors:

$$\text{purity drop} = +18.46272 - 0.34250T_C + 0.088472t_C \tag{4.8}$$

Where: A = coded value of cooling temperature
B = coded value of cooling time
T_C = cooling temperature
T_C = cooling time

Diagnostic Test

All diagnostic plots were also tested for adequacy of the model (normal plot of residuals, residuals vs predicted value, residuals vs factor, box cox plot, studentized residuals, leverage, etc.). All the plots indicated that the model statistically valid to predict the data. The following plot is described as an example for the purity drop across cooling crystallizers.

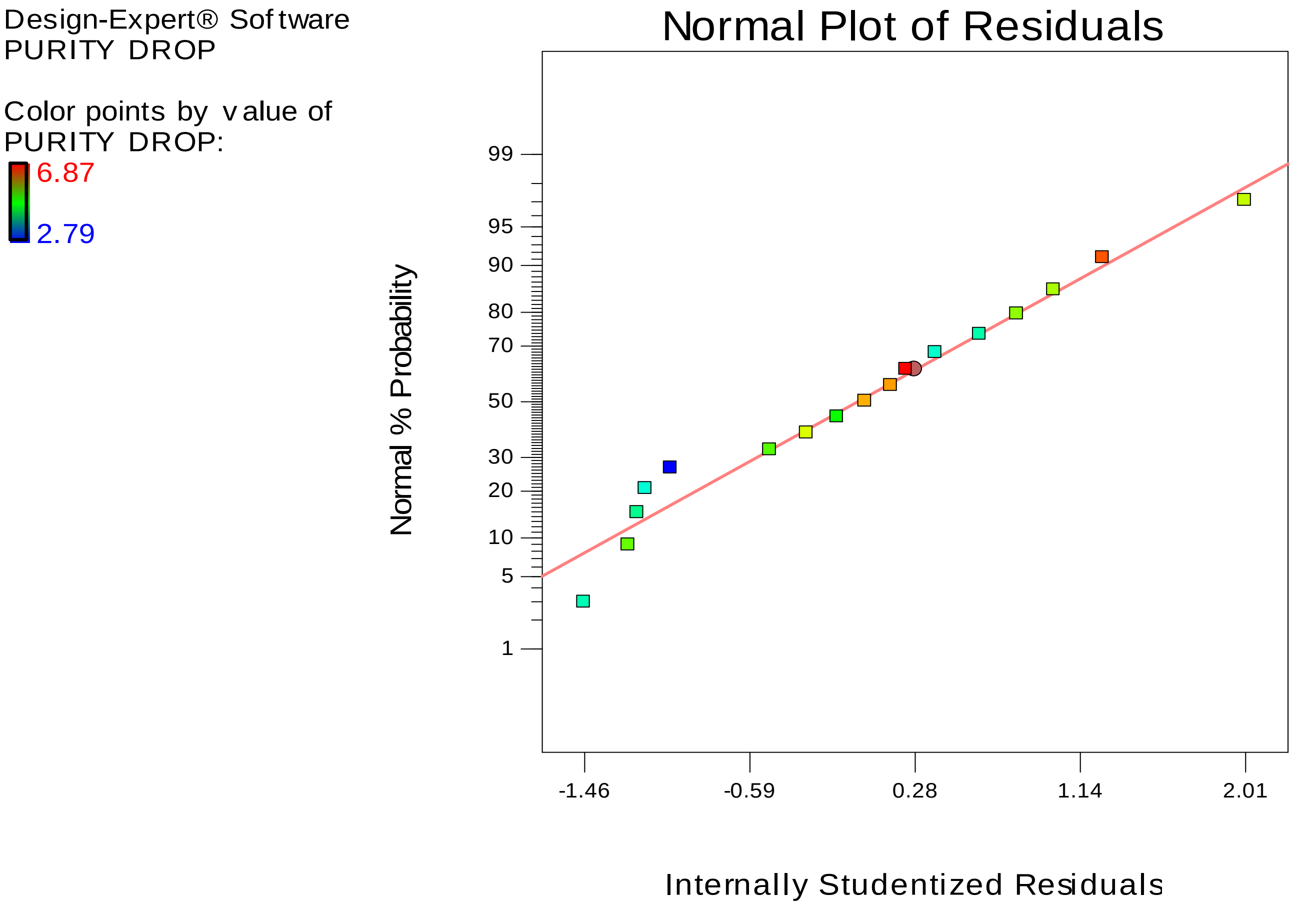

Figure 4.8: Normal plot of residuals for purity drop on massecuite cooling

4.2.2.4 Response Surface Plots Analysis of Purity Drop on Massecuite Cooling

As shown by graph on figure 4.9, the purity drop increases with decrease in cooling temperature and increase in cooling time. The lower the temperature, the lower the solubility of sucrose in the solution and hence less sucrose remain in solution, more sucrose from the solution deposited to the crystal surface.

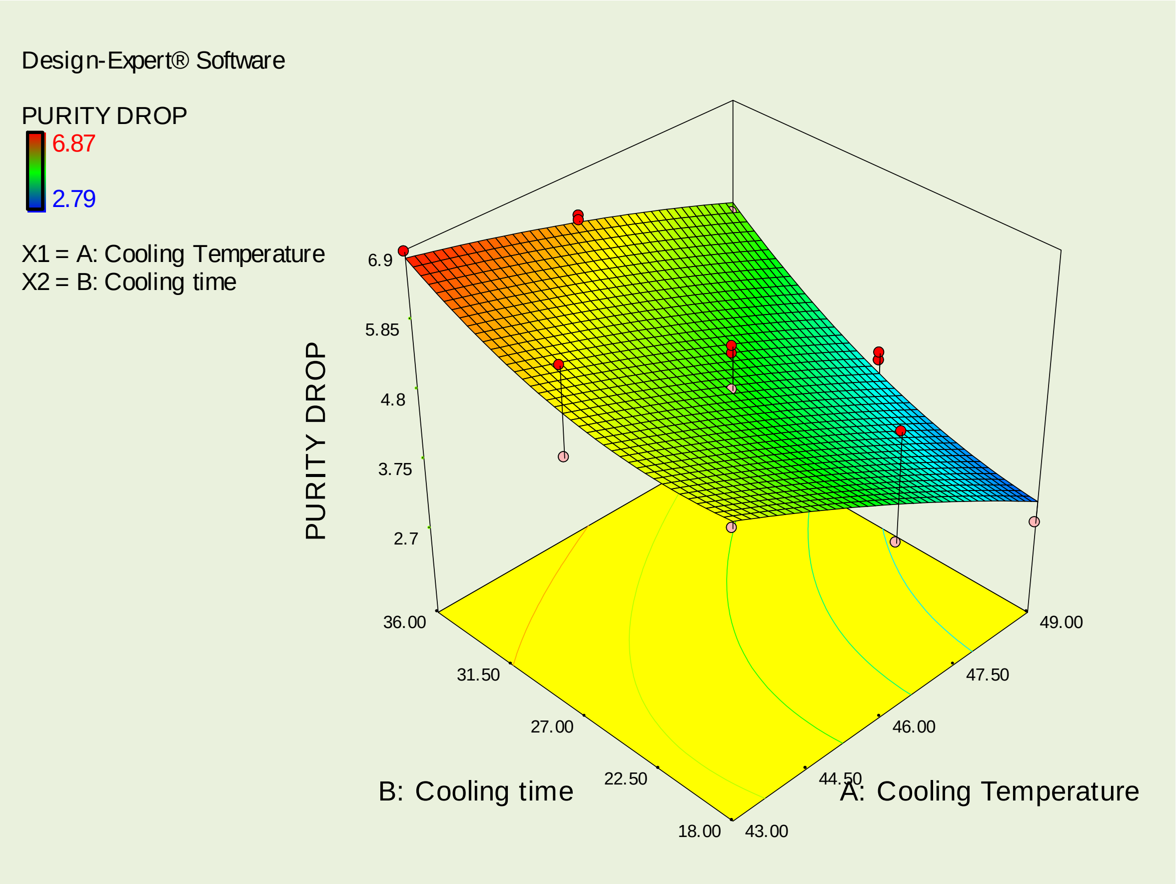

Figure 4.9: Response surface plot for purity drop across cooling crystallizers

4.2.2.5 Optimization Solution for D-Massecuite Cooling

The optimum purity for D-Massecuite Cooling was calculated by partially differentiating Equation (4.8) with respect to each parameter and equating to zero (Montgomery 2001). The optimization was to get maximum purity drop at minimum cooling time and maximum cooling temperature as much as possible. 1 solution is obtained as in (table 4.16) at Cooling temperature of 44^0C and cooling time of 36. At these optimum parameters the purity drop is 6.37 units.

Table 4.16: Optimization solution for D-Massecuite cooling experiment

Number	Cooling Temperature	Cooling time	purity drop	Desirability	
1	44	34.5	6.37469	0.186	Selected

4.2.3 D-Massecuite Reheating Optimization

Reheating and cooling temperatures were changed by varying the flow of cooling and reheating temperatures. Massecuite flow rate calculated from the load on the centrifugal machine. The test was done by one of the two reheatrs (reheater#1) and the massecuite flow rate in this table is multiplied by two to get total massecuite flow rate.

Table 4.17: Factors level design and the response for D-Massecuite reheating optimization experiment

Std	Run	Factors		Responses		
		Cooling Temp. ^{0}C	Reheating Temp. ^{0}C	Purity rise on reheater (%)	Nutsch Purity After reheater	Massecuite flow rate (T/H)
15	1	46.00	53.00	0.52	31.09	20.7
1	2	43.00	50.00	0.53	29.32	16.1
10	3	46.00	56.00	1.15	33.21	19.9
14	4	46.00	53.00	0.42	30.28	19.7
13	5	46.00	53.00	0.63	30.84	18.2
16	6	46.00	53.00	0.31	31.01	19.7
4	7	49.00	56.00	0.98	34.85	22.1
3	8	43.00	56.00	0.65	32.98	17.7
11	9	46.00	50.00	0.31	30.25	16.4
5	10	43.00	53.00	0.25	29.51	19.5
2	11	49.00	50.00	0.09	34.02	16.5
9	12	46.00	50.00	0.35	30.41	17.9
6	13	49.00	53.00	0.44	33.48	21.2
17	14	46.00	53.00	0.75	32.02	18.9
8	15	49.00	53.00	0.69	33.98	20.7
7	16	43.00	53.00	0.25	29.74	18.1
12	17	46.00	56.00	0.93	32.89	21.2

4.2.3.1 Data Analysis for Optimization Experiment on D-Massecuite Reheating

I. Response1.- Purity Rise Across Reheater

2nd order model is suggested for this response. All the necessary statistical data analyses were done for this response. From ANOVA test, the "Lack of Fit F-value" obtained was 2.25 which implies the Lack of Fit is not significant. Non-significant lack of fit is good. we want the model to fit.

Table 4.18: Post ANOVA statistics for purity rise on D-masseciute reheating

Std. Dev	0.18	R-Squared	0.6857
Mean	0.54	Adj R-Squared	0.6132
C.V. %	33.10	Pred R-Squared	0.4368
PRESS	0.76	Adeq Precision	11.361

The "Pred R-Squared" of 0.4368 is in reasonable agreement with the "Adj R-Squared" of 0.6132. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. This ratio of 11.361 indicates an adequate signal. This model can be used to navigate the design space.

II. Response 2-Nutsch Purity after Reheater

"Lack of Fit F-value" of 1.02 was obtained which implies the Lack of Fit is not significant relative to the pure error. Non-significant lack of fit is good -- we want the model to fit.

Table 4.19: Post ANOVA statistics D-nutsch purity after reheater

Std. Dev.	0.474921	R-Squared	0.951088
Mean	31.75765	Adj R-Squared	0.928856
C.V. %	1.495453	Pred R-Squared	0.874737
PRESS	6.353975	Adeq Precision	21.89477

The "Pred R-Squared" of 0.8747 is in reasonable agreement with the "Adj R-Squared" of 0.9289.

III. Response 3- Massecuite Flow Rate across Reheater

The Model F-value of 12.03 implies the model is significant. There is only a 0.04% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, AB, B^2 are significant model terms. The "Lack of Fit F-value" of 0.62 implies the Lack of Fit is not significant relative to the pure error. There is a 62.41% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

The "Pred R-Squared" of 0.6639 is in reasonable agreement with the "Adj R-Squared" of 0.7751. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 11.289 indicates an adequate signal. This model can be used to navigate the design space.

4.2.3.2 Correlation Matrix for D-Massecuite Reheating Optimization

Table 4.20: Correlation matrix of factors and responses of D- Massecuite reheating using SPSS

	A- Cooling temp.	B-Reheating temp.	C- Purity rise on reheater	R1-Nutsch purity After reheater	R2-Massecuite flow rate
Cooling temp.	1.000	0.000	0.159	0.734**	0.443*
Reheating temp.	0.000	1.000	0.742**	0.493*	0.681**
Purity rise on reheater	0.159	0.742**	1.000	0.505*	0.518*
Nutsch Purity After reheater	0.734**	0.493*	0.505*	1.000	0.481*
Massecuite flow rate	0.443*	0.681**	0.518*	0.481*	1.000

Table 4.21: Correlation Matrix of Factors [Pearson's r] for D- Massecuite reheating using Design Expert-RSM

	A	B	AB	A^2	B^2
A	1.00				
B	0.000	1.00			
AB	0.000	0.000	1.00		
A^2	0.000	0.000	0.000	1.00	
B^2	0.000	0.000	0.000	0.056	1.00

Where: A, and B - are factors (independent variables)
R - is response (dependent variable)

Almost all the factor- to-factor correlation is zero. The factors- to- responses correlation are different from zero.For the factors- to - factors correlation,Off-diagonal values close to zero are better.

4.2.3.3 Model Equation for D-massecuite Reheating Optimization

I. Purity rise across reheater

Equation in terms of coded factors:

$$\text{purity rise} = 0.54 + 0.065A + 0.30B + 0.19AB \quad (4.9)$$

Equation in terms of actual factors:

$$\text{purity rise} = + 46.32731 - 1.11194T_C - 0.88264T_R + 0.021389T_CT_R \quad (4.10)$$

II. Nutsch purity after reheater

Equation in terms of coded factors:

$$\text{Nutsch purity} = 30.95 + 1.85A + 1.24B - 0.71AB + 0.85A^2 + 0.87B^2 \tag{4.11}$$

Equation in terms of actual factors:

$$\text{Nutsch purity} = 260.02181 - 3.94298T_C - 6.17031T_R - 0.078611T_CT_R + 0.094839T_C^2 + 0.096228T_R^2 \tag{4.12}$$

III. Massecuite flow rate

Equation in Terms of Coded Factors:

$$\text{Massecuite flow rate} = 19.69 + 1.14A + 1.75B + 1.00AB - 0.13A^2 - 1.15B^2 \tag{4.13}$$

Equation in Terms of Actual Factors:

$$\text{Massecuite flow Rate} = -146.81667 - 4.21849\,T_C + 9.03216\,T_R + 0.111T_CT_R - 0.014035T_C^2 - 0.12792T_R^2 \tag{4.14}$$

Where : A= Coded value of cooling temperature

B= Coded value of reheating temperature

T_R = reheating temperature (^{0}C)

T_C = Cooling temperature (^{O}C)

4.2.3.4 Response Surface Plots Analysis for D- Massecuite Reheating Optimization

I. Purity Rise on Reheating

Purity rise across the reheater increases with increase in cooling as well as reheating temperatures. The higher the gap between the cooling and reheating temperature, the higher is the purity rise. This is due to the relatively longer time taken to bring the reheated temperature to the required value.

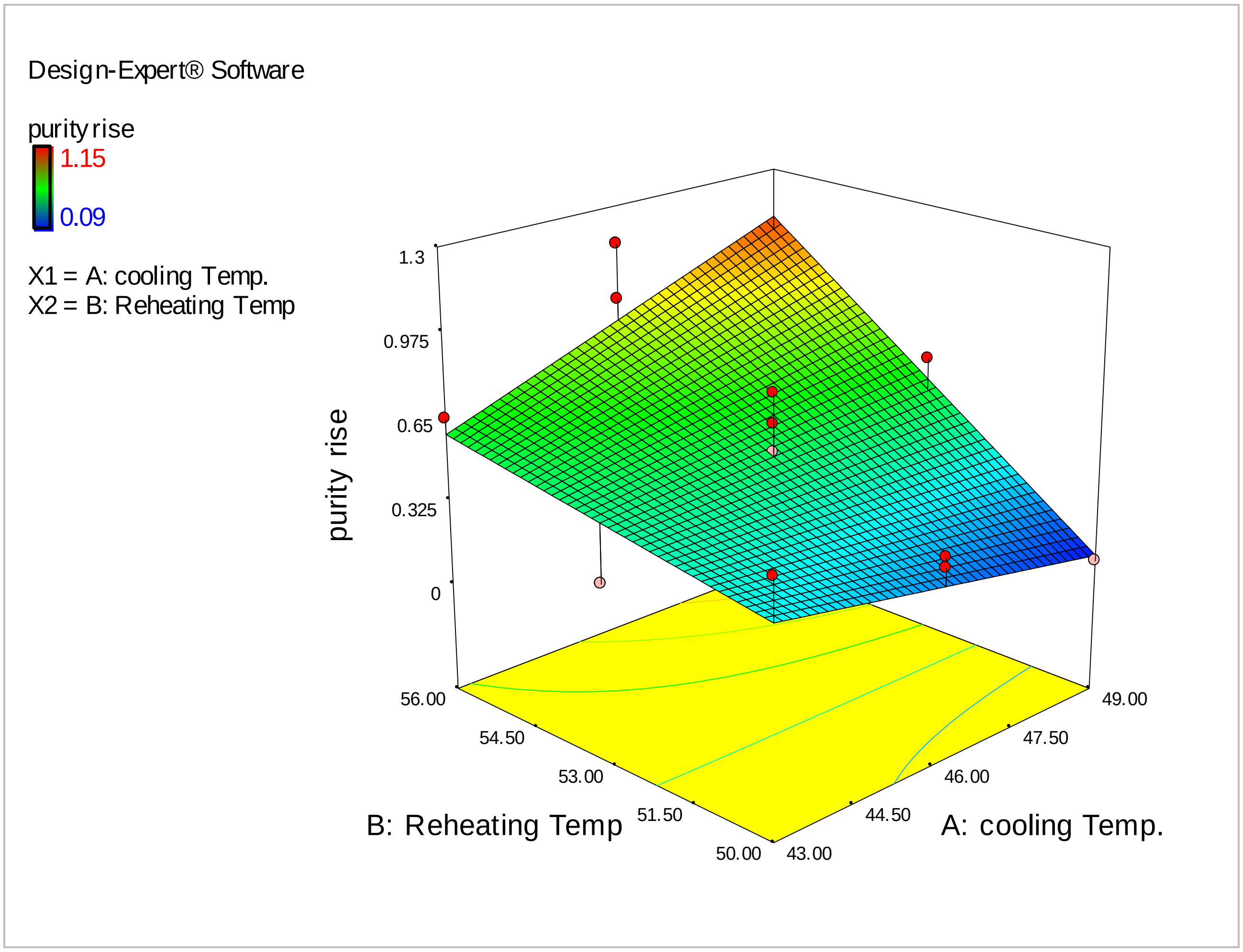

Figure 4.10: Response surface plot for purity rise across reheater

II. Nutsch purity after reheating

Nutsch purity after reheating also increases with increase in both tepprатures. Increase in temperature leads to dissolution of crystals in tne massecuite and hence the purity of mother liquor.

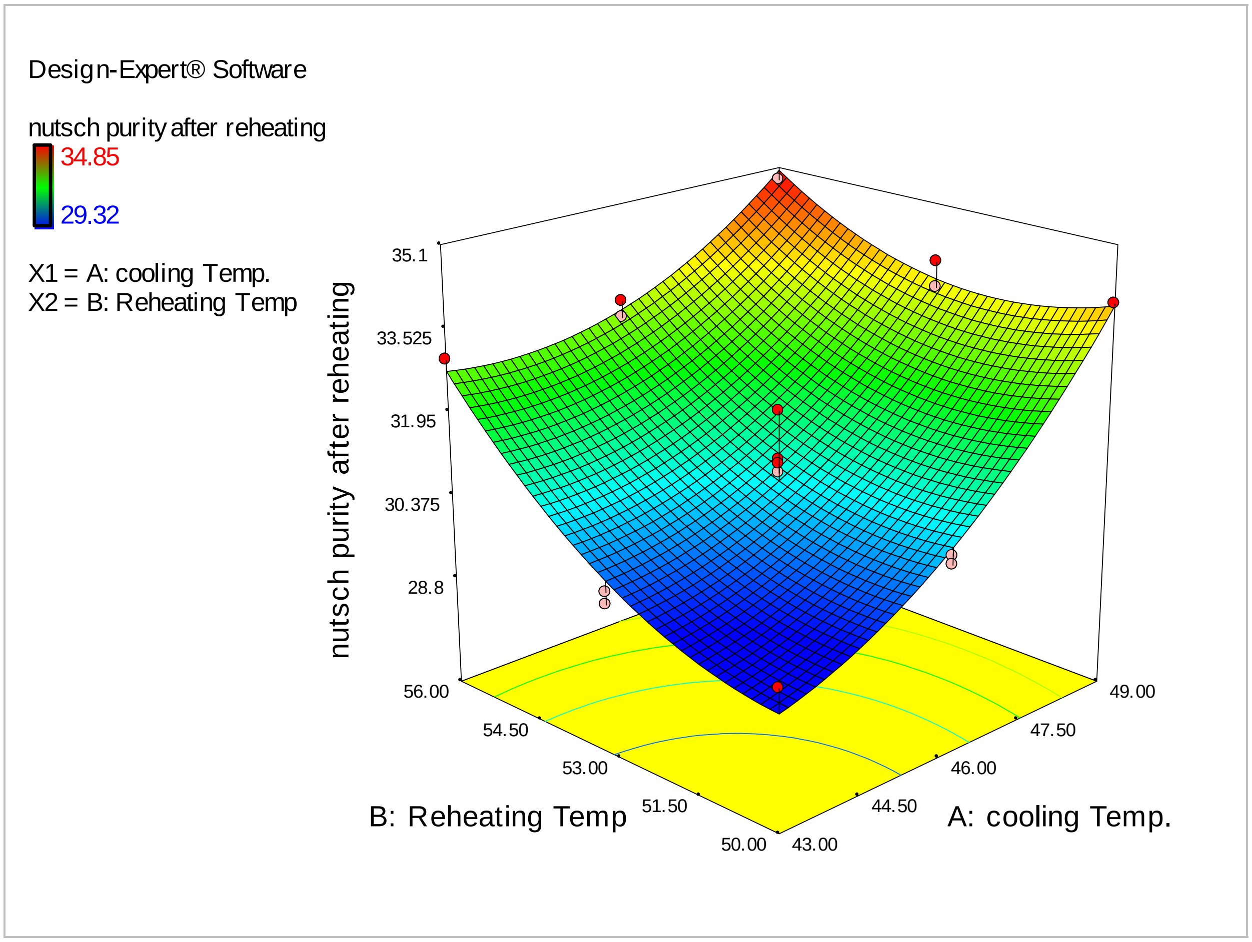

Figure 4.11: Response surface plot for mother liquor purity of D-massecuite across reheater

III. Massecuite flow rate

Obiviously the massecuite flow rate increases with increase in tempertaure due to low viscosity at high temperatures. Figure 4.12 reveals this fact and shows that the effect of reheating temperature is more siginificant on the massecuite flow rate than cooling temperature. Flow rate sharply increases with increase in reheating temperature up to about 53^{0}C and then slow increase is observed above 53^{0}C.

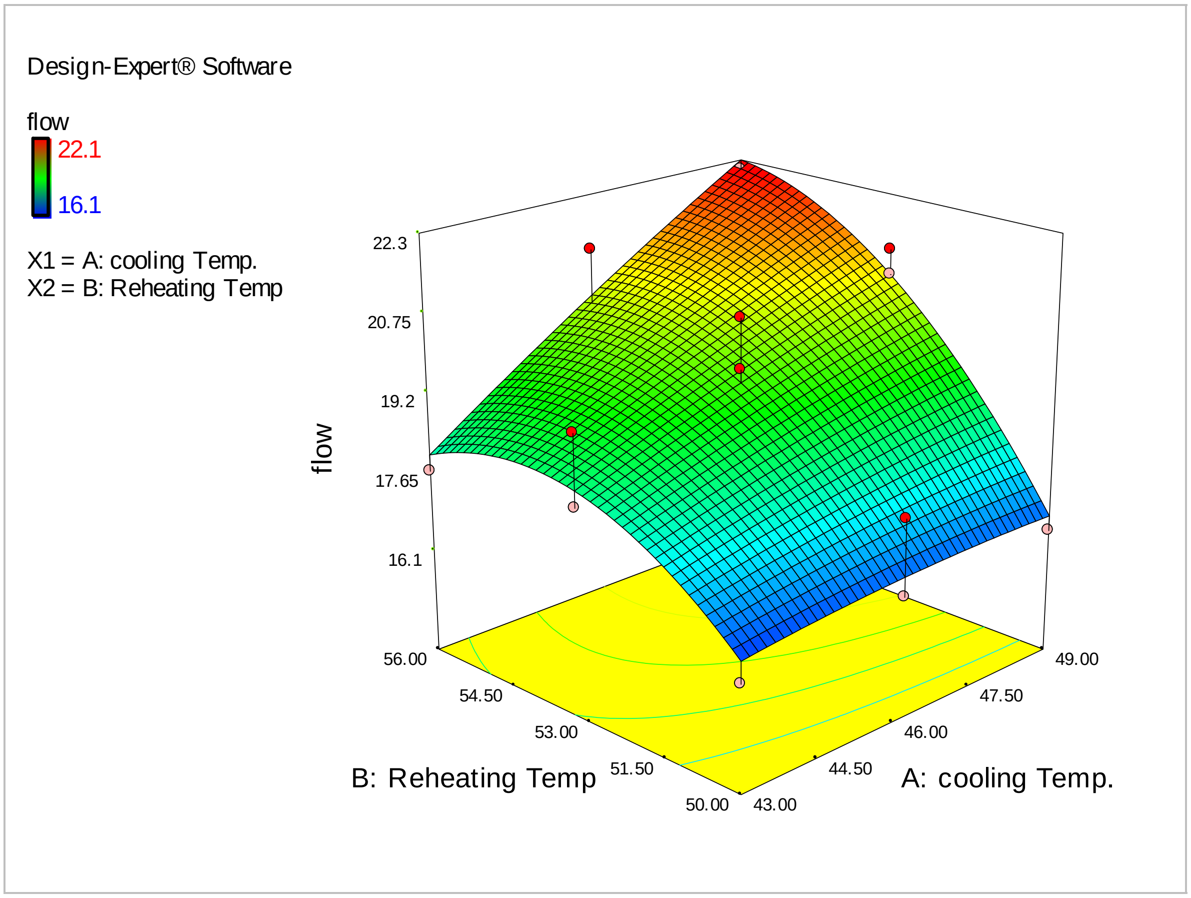

Figure 4.12: Response surface plot for flow rate of D-Massecuite across reheater

4.2.3.5 Optimization solution for Massecuite Reheating Experiment

Table 4.22: Optimization solution for Massecuite Reheating Experiment

Number	Cooling Temp.	Reheating Temp	purity rise	Nutsch purity after reheating	Flow	Desirability	
1	45.04	52.5	0.482653	30.23	19.04127	0.63672	Selected

One solution is obtained at desirability of 63.7%. Minimum purity rise of 0.483 units is obtained at optimum cooling temperature of 45.04^{0}C and reheating temperature of 52.5^{0}C.

4.2.4 D-Massecuite Centrifugal Separation Optimization

The amount of water was measured by online flow meter on the hot water line and the massecuite flow rate was calculated from the centrifugal machine load current. The load current has a direct relation with flow rate. The following data are generated for optimization across centrifugal machines.

Table 4.23: Factor level design for D-Massecuite centrifugal separation optimization

		Factor	Responses	
Std	Run	Water% massecuite	Purity rise	DFW magma purity
20	1	5.5	2.67	83.00
6	2	8.5	3.89	86.45
9	3	5.5	2.15	82.50
7	4	10.0	5.30	87.30
5	5	7.0	2.19	83.80
4	6	4.0	1.45	82.00
3	7	2.5	1.28	79.80
1	8	1.0	0.99	77.45
2	9	1.0	1.74	76.71
8	10	10.0	4.72	90.20
10	11	5.5	1.99	83.20
11	12	8.5	2.89	85.70
12	13	5.5	2.13	82.02
13	14	10.0	3.91	87.20
14	15	7.0	3.57	84.70
15	16	4.0	1.08	79.98
16	17	2.5	0.87	81.27
17	18	1.0	0.51	78.26
18	19	1.0	0.69	79.50
19	20	10.0	4.17	90.78

4.2.4.1 Data Analysis for Optimization Experiment on Massecuite Separation

I. Response1- Purity Rise on Centrifugal Machine

Table 4.24: Analysis of variance [ANOVA] table purity rise on centrifugal machine

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	34.33143	2	17.16572	68.04572	< 0.0001	Significant
A-water %mass.	32.88261	1	32.88261	130.3483	< 0.0001	
A^2	1.44882	1	1.44882	5.743192	0.0283	
Residual	4.288546	17	0.252267			
Lack of Fit	0.382832	4	0.095708	0.31856	0.8605	not significant
Pure Error	3.905714	13	0.30044			
Cor Total	38.61998	19				

Response Surface Quadratic Model is suggested by the sotware. The Model F-value of 68.05 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, A^2 are significant model terms.

The "Lack of Fit F-value" of 0.32 implies the Lack of Fit is not significant relative to the pure error. There is a 86.05% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table 4.25: Post ANOVA Statistics Test purity rise on centrifugal machine

Std. Dev.	0.502262278	R-Squared	0.88895526
Mean	2.409608682	Adj R-Squared	0.87589117
C.V. %	20.84414292	Pred R-Squared	0.84184064
PRESS	6.108111219	Adeq Precision	18.4441522

The "Pred R-Squared" of 0.8418 is in reasonable agreement with the "Adj R-Squared" of 0.8759. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. this ratio of 18.444 indicates an adequate signal. This model can be used to navigate the design space.

II. Response2- DFW Magma Purity

Table 4.26: Analysis of variance table for DFW magma purity [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	276.078	1	276.0783	201.0972	< 0.0001	Significant
A-water	276.078	1	276.0783	201.0972	< 0.0001	
Residual	24.7114	18	1.37286			
Lack of	5.08569	5	1.017139	0.673747	0.6507	not significant
Pure	19.6257	13	1.509676			
Cor Total	300.789	19				

For this response, Response Surface software suggested Linear Model. The Model F-value of 201.10 implies the model is significant. In this case A are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 0.67 implies the Lack of Fit is not significant relative to the pure error. There is a 65.07% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Table 4.27: Post ANOVA Statistics Test for DFW Magma Purity

Std. Dev.	1.171691		R-Squared	0.917845
Mean	83.09132		Adj R-Squared	0.913281
C.V. %	1.410125		Pred R-Squared	0.89279
PRESS	32.24763		Adeq Precision	28.05787

The "Pred R-Squared" of 0.8928 is in reasonable agreement with the "Adj R-Squared" of 0.9133. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 28.058 indicates an adequate signal. This model can be used to navigate the design space.

4.2.4.2 Correlation Matrix for D-Massecuite Centrifugal Separation Optimization

Table 4.28**:** Correlation matrix of factors and responses of D- Massecuite Separation using SPSS

	A-Water% massecuite	R1-Purity rise	R2-DFW magma purity
A-Water% massecuite	1.000	0.923**	0.958**
R1-Purity rise	0.923**	1.000	0.890**
R2-DFW magma purity	0.958**	0.890**	1.000

** Correlation is significant at the 0.01 level (2-tailed).

Table 4.29: Correlation Matrix of Factors [Pearson's r] for D-massecuite separation (using Design Expert-RSM)

	A	A^2
A	1.00	
A^2	0.000	1.00

4.2.4.3 Model equation for the optimization Experiment on D-Massecuite Centrifugal Separation

Equation in terms of Coded Factors:

$$\text{purity rise} = 2.09 + 1.79A + 0.63A^2 \tag{4.14}$$

Equation in terms of Actual Factors:

$$\text{purity rise} = 0.83921 + 0.054807W + 0.031258\,W^2 \tag{4.15}$$

Equation in terms of Coded Factors:

$$\text{DFW pty} = 83.09 + 5.20\,A \tag{4.16}$$

Equation in terms of Actual Factors:

$$\text{DFW pty} = +76.73819+1.15512W \tag{4.17}$$

Diagnostic test

The normal plots of residuals were checked for all responses and all indicated the validity of the model equations. The residual data were almost aligned with the predicted values.

4.2.4.4 Response surface plots analysis D-Massecuite Centrifugal Separation

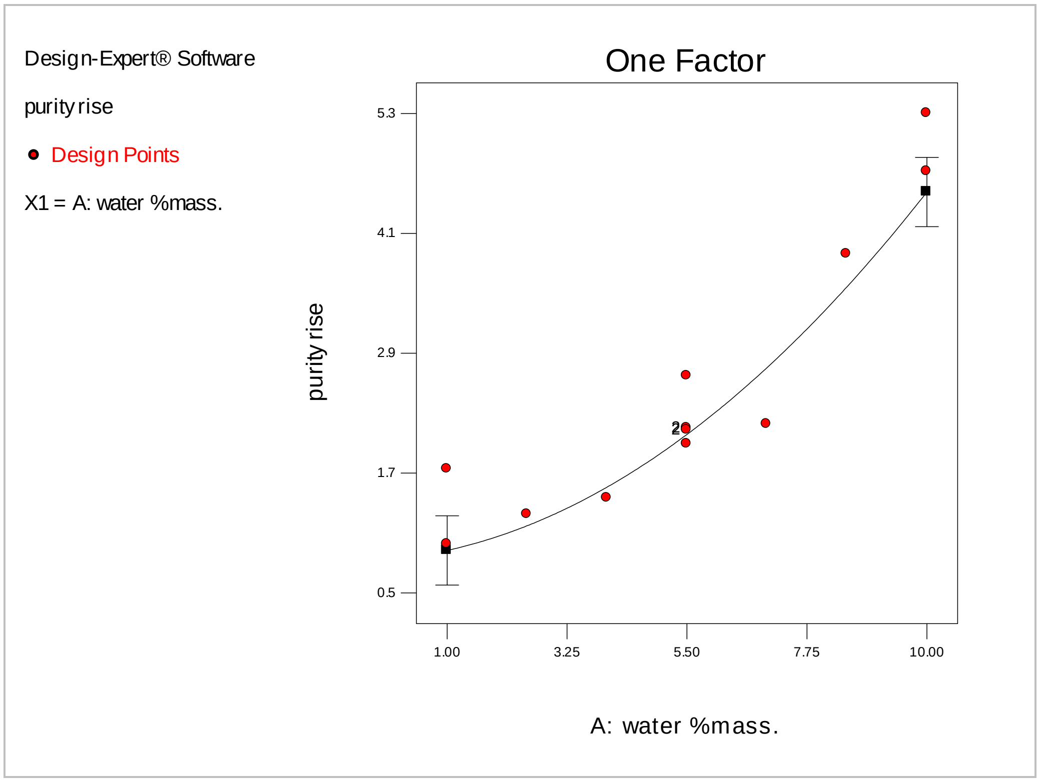

Figure 4.13: Response surface plot for purity drop across centrifugal machine

There is a second order relationship between purity drop across centrifugal machine and the amount of spray water added.

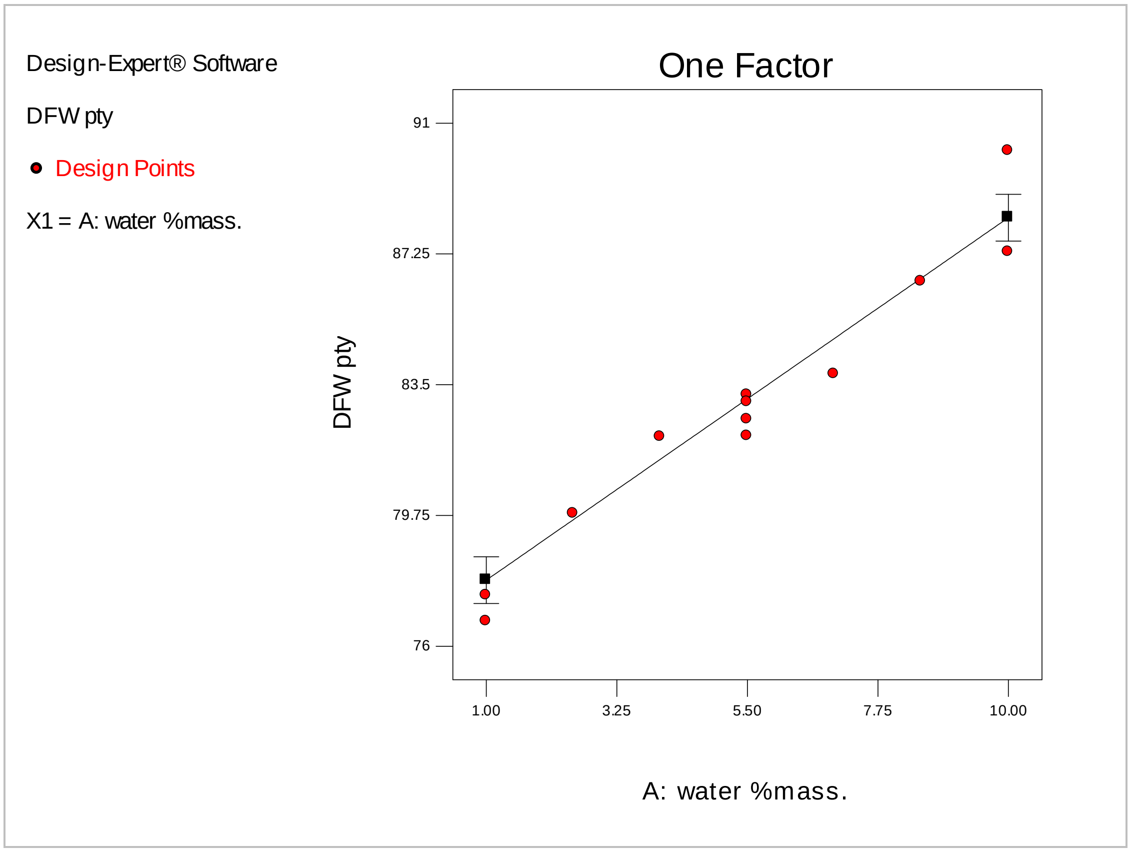

Figure 4.14 : Response surface plot for DFW magma purity

The purity of DFW magma increases almost linearly with increase in amount of spray water application.

4.2.4.5 Optimization Solutions for Optimization across Massecuite Centrifugal Machine

Table 4.30: optimization solution for D-massecuite separation

Number	Water %mass.	purity rise	DFW Magma pty	Desirability	
1	6.33	2.437	84.047	0.558	Selected

One solution is obtained with desirability of 55.8%. The optimum spray water is 6.33% massecuite. At this spray water, the optimum purity rise and DFW magma purity are 2.44 and 84 units respectively.

4.3 Summary of Optimization Results

As already seen under the data analysis part, the optimum parameters for D-massecuite boiling were massecute purity of 56.25^0, brix of 102^0 and seed volume of 2000ml. At these optimum parameters, the nutsch (mother liquor) purity and purity drop at pan discharge were 35.52^0 and 20.73units respectively. A nutsch purity reduction of 36.05 – 35.52 = 0.53 is obtained by optimization of massecuite boiling. As the brix increases the nutsch purity decreases due to less water in the massecuite and hence less sucrose remains in the solution. A brixs of more than 102 is not practical due to less fluidity of the massecuite causing difficulty in discharging from pan, pumping and agitation by crystallizers. With increase in purity of massecuite, its mother liquor (nutsch) purity also increases proportionally. But as masecuite purity decreases below 56.25, the crystallization of sucrose is not effective due to high non sucrose content which causes high viscosity and reduces crystallization rate.

Due to the availability of more total surface area to absorb more sucrose molecules from the solution, purity drop increases as seed volume increases. However there is a practical limit to the maximum of seed volume beyond which two negative impacts will be encountered. The final massecuite crystal size will be very small allowing passage through the sieves of centrifugal machines. The second effect is the increase in viscosity and solidity of the massecuite causing handling problem in pan, crystallizers and pumps. Seed volume of 2000ml is taken as maximum limit.

At masseciute cooling, the optimum parameters for high purity drop were cooling temperature of 44^0C and cooling time of 34.5h. At these optimum temperature and time, the purity drop obtained was 6.37units which was well within the standard limit. But the purity drop obtained was at the cost of masecuite flow rate. With the existing total crystallizers volume of $220m^3$, this optimum retention time allows a massecuite flow rate of $220m^3/34.5h = 6.4m^3/h$. In terms of mass flow rate it is equal to 9.6t/h (specific gravity of D-massecuite = 1.5). The massecuite production rate of MSF is 16 – 20t/h. Hence additional capacity crystallizers are required to get standard purity drop of greater than 6.0 units by cooling.

Purity drop across massecuite reheater is influenced by reheating temperature, cooling temperature and reheating time. In this experiment cooling and reheating temperatures were taken as independent variables and reheating time was obtained indirectly from massecute flow rate and reheater volume. As the temperature difference between cooling and reheating increases, the purity rise also increases due to requirement of high heating water temperature or high reheating time. There is an optimum point at which minimum purity rise can be achieved. The optimum cooling and reheating temperatures to minimize the purity rise across reheater were 45^0C and 52.5^0C respectively. The minimum possible purity rise and maximum possible massecute flow rate obtained at the optimized parameters were 0.48 units and 19t/h respectively. The optimum reheating time is calculated from the flow rate of massecuite (19t/h), working volume of the reheater ($3.5m^3$) and D-massecuite specific gravity (1.5). It is equal to 2x3.5/(19/1.5) =0.538h = 32.3minutes. This value is marginal compared to the standard limits of about 30minutes.

Optimization solution for D-massecuite cooling was calculated for the existing crystallizers' capacity by putting a target cooling temperature of 45^0C and cooling time of 17.37h in design expert program (both values were taken from reheating optimization solution). Cooling time is equal to 220X1.5/19 =17.37h. The optimum solution is shown in table 4.31.

Table 4.31 : Massecuite cooling optimum solution for the existing factory setup

Number	Cooling Temperature	Cooling time	Purity Drop	Desirability	
1	45	17.37	5.01353	0.816822	Selected

A maximum purity drop of 5.01 units was obtained for the existing cooling crystallizers. The overall purity drop for the existing crystallizers and with additional capacity of cooling crystallizer (cooling time of 34.5h) is compared as shown in table 4.32.

For D- masscuite separation, minimum purity rise across and maximum DFW magma purity are desired. The higher the purity rise, the higher is the loss of sucrose with final molasses. The lower the purity of DFW magma purity, the higher is recirculation of molasses (non sucrose) back to the boiling house. Non sucrose recirculation also needs equal attention since it reduces boiling house efficiency, increase steam consumption and reduce final product quality. Therefore, a compromise is needed between these two parameters. The main factors which influence these parameters, at fixed centrifugal machine design, is the amount of spray water applied to the centrifugal machines. The higher the amount of water, the better the DFW magma purity is, but final molasses purity will increase due to more dissolution of sugar crystals.

Keeping the massecuite reheating temperature constant, optimization was carried out on massecuite separation by changing the flow rate of spray water. Purity rise of 2.44 and DFW magma purity of 84 were obtained at optimum spray water of 6.33%massecuite.

Table 4.32: comparison of purity drops for existing cooling crystallizer's capacity and extra cooling capacity

With the existing crystallizer capacity				With cooling time of 34.5h		
Station	Massecuite purity	Purity drop	Nutsch purity	Massecuite purity	Purity drop	Nutsch purity
Boiling	56.25	20.73	35.52	56.25	20.73	35.52
Cooling	56.25	5.0	30.52	56.25	6.37	29.15
reheating	56.25	-0.48	30.99	56.25	-0.48	29.63
Centrifugal separation	56.25	-2.44	33.43	56.25	-2.44	32.07

4.4 Reduction in Purity of Final Molasses due to the Optimization at each Process Step

4.4.1 With the Existing Crystallizes Capacity (220m³)

Table 4.33 : Gain in purity drop due to optimization with existing crystallizer capacity

D- Massecuite Station	Purity drop/nutsch purity*		
	Before optimization	After optimization	Gain in purity drop
Boiling	36.05*	35.52*	0.53
Cooling	4.21	5.01	0.80
Reheating	-0.97	-0.48	0.49
Separation	-2.05	-2.44	-0.39
Overall purity drop			1.43

*At boiling nutsch purity is considered since there is different massecuite purity before and after optimization

Gain in Purity drop = nutsch before – nutsch after

= purity drop after – purity drop before

Final molasses purity before optimixzation = 36.05-4.21+0.97+2.05 =34.86

Final molasses obtained by optimization = 35.52-5.01+0.48+2.44 = 33.43

Reduction in final purity = 34.86 – 33.43 = 1.43 units (overall purity reduction due to optimization with existing crystallizers).

4.4.2 With Extra Crystallizer Capacity (413m^3 Capacity)

Table 4.34: Gain in purity drop due to optimization with extra crystallizer capacity

D- Massecuite Station	Purity drop/nutsch purity*		
	Before optimization	After optimization	Gain in purity drop
Boiling	36.05*	35.52*	0.53
Cooling	4.21	6.37	2.16
Reheating	-0.97	-0.48	0.49
Separation	-2.05	-2.44	-0.39
Overall purity drop			2.79

- Final molasses obtainable before optimization of the massecuite purity = 36.05-4.21+0.97+2.05 =34.86 units
- Final molasses obtained by optimization = 35.52-6.37+0.48+2.44 = 32.07
- Reduction in final purity = 34.86 – 32.05= 2.69 units (overall purity reduction due to optimization plus additional crystallizer capacity).
- Final molasses purity reduction due to extra crystallizer capacity alone = 33.43-32.07 = 1.36units.

4.5 Techno- Economic Gain by Purity Reduction of Final Molasses

The gain in terms of the amount of additional sugar (final product) obtained due to reduction in the purity of final molasses by applying process optimization on D-massecuite boiling, cooling, reheating and separation can be calculated from solid balance across crystallization and separation plants. For the crystallization and separation plant the input is syrup and the outputs are final molasses and final product. The aim of the process optimization is to reduce the sugar lost with final molasses and to increase the amount of sugar going to final product from certain amount of sucrose input by the syrup. In order to calculate the amount of sugar produced from a fixed amount of sucrose input by syrup, first we have to know the amount of syrup generated in MSF from certain amount of cane crushed by carrying out material balance for the sugar factory (MSF).

4.5.1 General Material Balance on Metahara Sugar Factory

Average Data of MSF-2005/2006 – 2009/2010)

Parameter	Value	Remark
Cane crushed	5366.7 t/ milling days	weighing
	223.61 t/h	weighing
milling days	220.5	@Time efficiency of 87.45%
Sucrose % cane	12.90	lab. analysis
Fibre% cane	13.88	lab. analysis
Fiber%bagasse	48.20	lab. analysis
Bagasse % cane	28.80	calculation
Imbibition % cane	29.03	flow meter
Mixed juice%cane	100.23	weighing
Filter cake% cane	3.445	weighing
Sweetening off water	3.5	flow meter
Milk of lime % cane	2.25	weighing
Clear juice% cane	102.53	calculation
Clear juice brix	15.9	lab. analysis
Syrup purity	83.51	lab. analysis
Syrup Brix	61.7	lab. analysis
Final molasses purity	34.45	lab. analysis
Final product (sugar)	99.8	lab. analysis

Material balance for each unit operation

i. Extraction Plant

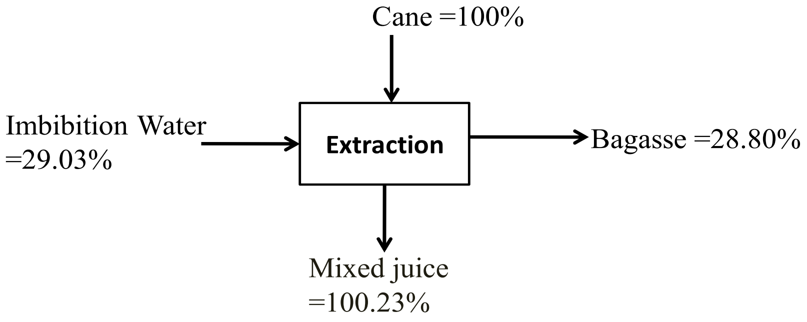

Figure 4.15: Material balance around extraction plant

ii. Juice Clarification Plant

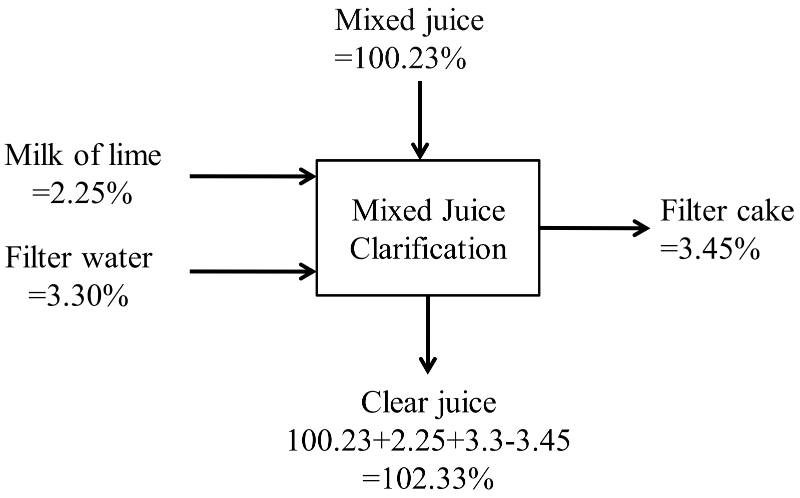

Figure 4.16: Material balance around clarification plant

iii. Evaporation plant

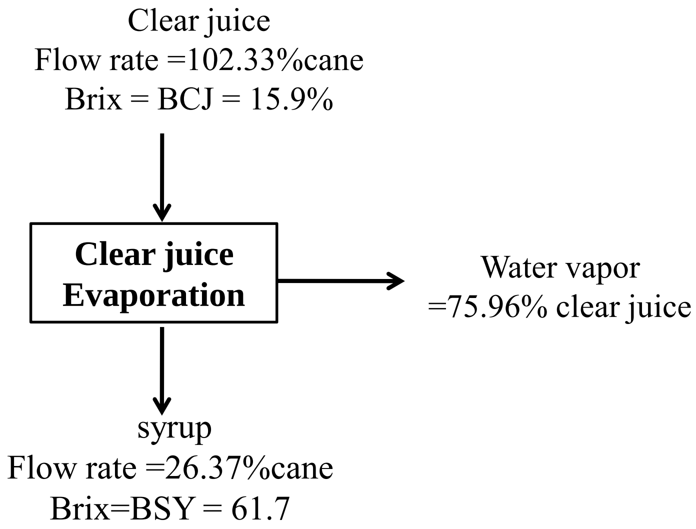

Figure 4.17: Material balance around evaporation plant

Solid balance across evaporator

Solid in clear juice = solid in syrup

BCJ x Flow rate of clear juice = BSY x Flow rate of syrup

i. Crystallization and separation plant

The solid in the syrup is divided into solid in final product and solid final molasses. The solid balance can easily be calculated from purities of products by using Cobeze Diagram as shown by figure 4.5.

Where: Pi = purity (%)

Si= solid flow rate (T/H)

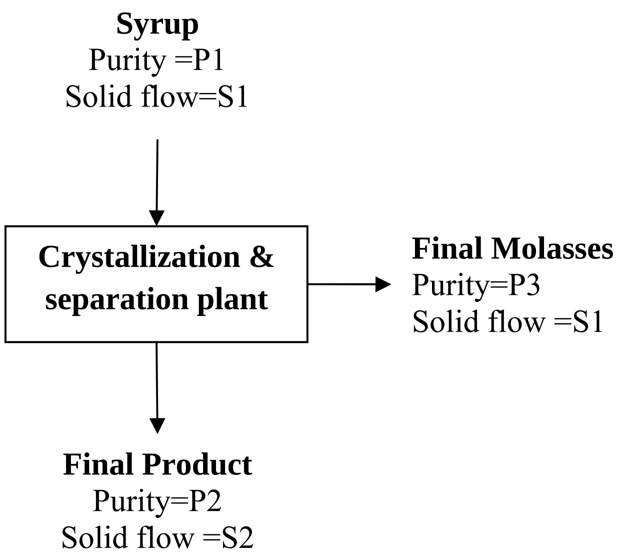

Figure 4.18: Input – output around crystallization and separation plant

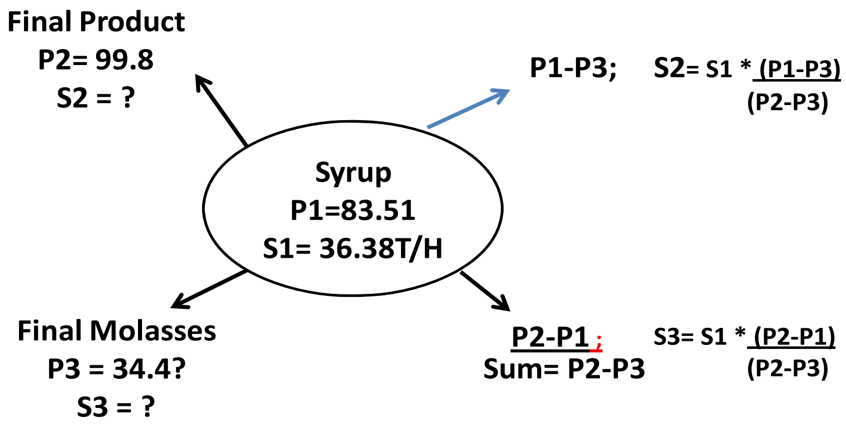

Figure 4.19: The Cobenze Diagram

4.5.2 Financial Gain by Purity Reduction of Final Molasses

The gain in terms of the amount of additional sugar (final product) obtained due to reduction in the purity of final molasses by applying process optimization on D-massecuite boiling, cooling, reheating and separation can be calculated from solid balance across crystallization and separation plants. Cobenze Diagram is used to carry out the solid balance.

a) Solid balance for the final molasses purity obtained before optimization (34.86^0)

- Solid flow of final product =36.38x(83.51-33.43)/(99.8-33.43) = 27.451t/h
- Solid flow of Final molasses =36.38 – 27.451 = 8.929t/h

b) Solid balance for final molasses purity of 33.43^0 (obtained by optimization with the existing crystallizer's capacity)

- Solid flow of final product =36.38x(83.51-34.86)/(99.8-33.43) = 27.254t/h
- Solid flow of Final molasses =36.38 – 27.451 = 8.929t/h

c) Gain in final product due to reduction of final molasses from 34.86 to 33.43

- Sugar gain = 27.451 – 27.254 = 0.1966t/h = 4.72t/day
- For 220.5 milling days per campaign the gain is = 4.72x220.5 = 1040.5 **t/year**
- For sugar price of 13,000Birr/ton, the gain in monetary term is =1040.5 x13000 **=13,526,919 Birr/year.**

d) **Additional gain by replacing the existing crystallizer with veridical type high capacity crystallizer to obtain enough cooling time (and final molasses purity of 32.07^0)**

- Solid flow of final product at final molasses purity of 33.43^0 is equals to 27.451t/h
- Solid flow of final product at final molasses purity of 32.07^0 is equal to 36.38x(83.51-32.07)/(99.8-33.43) = 27.63t/h
- The sugar gain due reduction in final molasses purity by 1.36 is = 27.63 -27.451 = 0.179t/h = 4.3 t/day = 4.3x220.5 = **948.8t/year.**
- The gain in terms of money = 948.8x13000=**12,334825Birr/year**

4.5.3 Technological Benefit by Replacing the Existing Crystallizers with Vertical Type

Now a days, recent technology led sugar industries are no more using horizontal type crystallizers. Compared to traditional horizontal units, vertical crystallizers provide significant benefits such as:

- Larger volumes and smaller footprint
- Possibility of outdoor installation
- Higher cooling surface to volume ratios
- A better ability to handle highly viscous massecuite
- Minimizes over flow losses
- Allows plug flow (first in – first out principle)
- Requires less space/compact design
- Less power consumption
- Requires no supporting stages
- Easily automated
- Requires less manpower and maintenance cost

Factories are currently aiming for maximum sugar recovery levels in response to increasing prices on international markets. One profitable plant investment strategy with a very rapid payback period is to boost a factory's low grade station crystallizer capacity by installing vertical cooling crystallizers. A number of factories have been able to achieve an improvement of more than four units in their final molasses purity through such an investment and as a consequence they have recovered their investment costs in a matter of months.

5 Conclusions and Recommendations

5.1 Conclusions

This thesis mainly focused on the optimization of D-massecuite station operational parameters that could lead to reduction of sucrose loss with final molasses by minimizing its purity. After assessment of the existing D-massecuite processing parameters, optimization of the parameters was carried out at all process steps to minimize sucrose loss with final molasses.

The Optimization experiments have been successfully designed, analyzed, proper data are generated and statistically validated. RSM has been employed to optimize the parameters since it is an indispensable tool for Process Optimization.

The study clearly indicates that there is a potential to minimize sucrose loss with final molasses at MSF by optimizing process parameters at D-massecuite boiling, cooling, reheating and centrifugal separation. The existing final molasses purity of 34.86 can be reduced to 33.43, i.e overall purity drop of 1.43 can be obtained by optimization and strict control of D- massecuite process parameters. This results in annual saving of 1040.5 tons of sugar or 13,526,919 Birr/year.

In addition to the strict parameter control and application of optimization, if we replace the existing crystallizers with the new vertical type to get retention time of about 34.5 hrs and cooling temperature of 44^0C, the final molasses purity can be reduced to 32.05 which is an additional gain of 948.8tons of sugar per year. This is equivalent to saving of additional 12,334825Birr/year.

Except for cooling crystallizers all the unit operations in MSF related to D-massecuite processing have enough capacity to give standard purity drop by proper control of process parameters. At pan boiling, by applying the required amount of seed, doing proper purity balance for every batch and brixing up the massecuite to the required value before discharging, it is possible to get the required purity drop.

5.2 Recommendations

According to the experimental results and practical observations, the following recommendations to be implemented in Metahara Sugar Factory to reduce loss of sucrose with final molasses:

- Put in practice and strictly control the optimum process parameters obtained by this thesis for D-massecuite boiling, cooling, reheating and centrifugal separation.
- Implement periodic sampling and nutsching of the D-massecuite on discharge from the pan, at inlet and out let of each cooling crystallizers, after reheating and on entering the centrifugal, compare and interpret the purity analysis results of the various Nutsch samples to gauge crystallization (massecuite exhaustion) performance.
- Provide temperature control system for massecute reheating
- Training or awareness to be given for operators and other workers at pan, crystallizer, reheater and centrifugal stations about purity balance and the effect of parameters control on loss minimization.
- Implement centrifugals screen inspection and replacement program. The purity rise across the centrifugal machines was higher despite optimization of spray water amount. There is no screen inspection and replacement program for MSF. The screens are changed only when they are torn (detached). Normally, centrifugal screens get worn out after serving for some months and allow passage of some sugar crystal with final molasses. As a rule there should be inspection and replacement program of screens every time, to avoid loss of crystal sugar through the worn out sieves.
- Replacement of the existing under capacity and old horizontal crystallizers with of vertical crystallizer (s) of sufficient cooling capacity to obtain standard purity drop across cooling crystallizer. The vertical crystallizers allow proper cooling by allowing plug flow of massecuite in which first in first out rule is attained. Since crystallization by cooling is the last desugarization phase in the sugar production process, any process control errors would have an irreversible effect on the amount of sugar lost to the molasses. This is why optimized cooling crystallization is so important.

5.3 Recommended future study

Determination of Target Purity of Final Molasses for MSF: The optimum final purity obtained by this research is a relative value. It is minimum value compared to the existing final molasses purity. But there is no evidence to conclude that the achieved final molasses purity is the lowest possible purity for MSF. For final molasses, the “Target Purity” (or equilibrium purity) refers to 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 for a fixed composition of non-sucrose substances. Depending on the prevailing conditions, each factory has its own target purity which could be obtained experimentally by conducting boiling and crystallization in model pans and crystallizers at different boiling and cooling time and temperatures. Further research is recommended to determine the target purity of final molasses for MSF.

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Appendices

Appendix-A: Detail Brix and Purity Analysis of D- Massecuite and its Mother Liquor at Different Process Steps before Optimization

I. First Battery crystallizers

Crystallizer#12 inlet

Temperature ^{0}C	Mass. Brix	Mass. Purity	Nutsch brix	Nutsch purity	Nurity drop
62	102.24	56.46	103.00	35.53	20.93
67	107.88	55.26	104.60	34.03	21.23
62	101.10	56.50	103.00	34.85	21.65
59	101.90	53.06	102.40	35.25	17.81
66	101.46	57.77	101.90	37.39	20.38
65	101.04	57.72	99.70	39.62	18.10
66	100.74	60.51	102.30	39.30	21.21
57	102.42	58.23	102.40	36.72	21.51
66	100.14	60.27	100.40	38.04	22.23
63	102.06	57.44	101.70	35.10	22.34
64	100.86	57.22	104.00	35.20	22.02
65	101.10	56.08	101.90	35.42	20.66
66	102.42	54.95	103.80	33.80	21.15
50	99.96	56.60	103.30	33.30	23.30
60	101.46	56.30	101.70	36.77	19.53
63	101.79	56.96	102.41	36.02	20.94

crystallizer#12 outlet/ crystallizer#13 inlet

53	102.42	54.66	107.2	33.12	21.54
43	102.42	56.35	104.2	31.3	25.05
55	102	55.76	102.102	31.4	24.36
55	102.4	54.2	103.6	33.4	20.8
59	101.3	56.52	103	34.08	22.44
55	100.38	58.4	101	35.24	23.16
59	102.24	56.75	102.6	33.72	23.03
54	100.92	58.68	99.9	35.23	23.45
60	99.72	60.47	99.1	36.83	23.64
47	101.64	57.2	101.4	35.2	22
55	101.58	57.23	101.5	33.1	24.13
62	101.1	56.38	100.5	32.34	24.04
60	101.94	55.97	101.1	33.04	22.93
49	100.8	56.25	99	34.04	22.21
58	101.58	56.23	104.2	34.17	22.06
55	101.5	56.7	102.0	33.7	23.0

Crystallizer#13 outlet/crystallizer#14 inlet

Temperature ^{0}C	**Mass. Brix**	**Mass. Purity**	**Nutsch brix**	**Nutsch purity**	**Nurity drop**
48	103.26	55.08	105.6	30.37	24.71
52	101.64	57.38	102	32.45	24.93
53	101.28	53.55	103.6	29.54	24.01
53	101.28	55.86	102	31.96	23.9
58	101.16	57.06	101.5	35.07	21.99
58	101.74	58.49	99.6	38.85	19.64
53	101.28	58.59	101.4	31.95	26.64
46	101.16	58.84	100.6	30.42	28.42
62	100.62	59.83	100.6	36.28	23.55
51	101.28	58.17	99.9	32.43	25.74
48	101.52	56.03	101.1	30.46	25.57
41	100.38	56.78	99.9	30.69	26.09
54		56.32	101.7	30.38	25.94
40	100.86	54.37	101.7	29.6	24.77
56	102.06	55.73	103.1	32.4	23.33
52	101.37	56.81	101.62	32.19	24.62

Crystallizer#14 outlet/Reheater inlet

44	103.32	54.76	104.60	28.88	25.88
46	101.88	56.36	103.20	31.10	25.26
50	101.16	56.11	103.60	30.98	25.13
51	100.80	56.96	102.90	32.65	24.31
57	100.56	57.10	101.00	33.76	23.34
56	106.26	60.53	100.60	35.49	25.04
50	100.50	58.15	102.00	31.27	26.88
43	100.50	58.32	100.10	30.07	28.25
47	100.38	59.41	99.30	32.63	26.78
58	100.56	63.42	100.91	33.69	29.73
41	101.52	57.44	101.60	31.10	26.34
42	99.54	54.79	102.00	30.00	24.79
41	101.28	55.27	88.00	32.39	22.89
57	102.78	53.53	103.70	31.44	22.09
49	100.56	54.71	100.60	29.42	25.29
49	101.44	57.12	100.94	31.66	25.47

Reheater outlet/centrifugal machine inlet

43	104.04	55.13	104.90	30.60	24.53
55	101.88	55.83	104.00	32.30	23.53
49	101.28	56.55	100.60	30.41	26.14
56	101.88	54.95	101.70	35.99	18.96
57	100.86	58.12	93.00	37.85	20.27
56	100.68	58.52	99.90	35.74	22.79
56	100.44	59.26	101.60	33.37	25.89
51	100.80	58.15	100.90	33.20	24.95
55	99.06	58.39	99.10	30.88	27.51
48	100.86	57.22	101.20	32.06	25.16
59	100.86	56.10	100.00	32.00	24.10
58	99.78	57.13	104.20	29.37	27.76
64	101.46	54.88	90.90	30.80	24.08
44	100.98	55.73	101.60	30.78	24.95
60	101.46	56.30	102.10	31.44	24.86
54	101.09	56.82	100.38	32.45	24.36

Centrifugal machine outlet (Final molasses)		
Temperature	Brix	Purity
50	97.9	32.97
50	98.2	32.5
52	97.7	32.64
51	97.3	32.64
58	95.8	39.98
68	100.02	33.69
50	95.6	36.29
51	94.2	34.7
55	96.5	34.715
49	95.2	33.3
50	95	35.47
58	97.3	34.94
46	95.9	31.49
AVERAGE= 52.9	**96.9**	**34.1**

III. Second Battery Crystallizers

Crystallizer#15 inlet					
Temperature ^{0}C	**Mass. Brix**	**Mass. Purity**	**Nutsch brix**	**Nutsch purity**	**Nurity drop**
62	101.40	57.22	104.00	36.15	21.07
66	101.28	54.44	104.20	35.12	19.32
59	98.64	58.76	100.10	39.66	19.10
66	101.70	59.87	104.20	35.60	24.27
63	100.50	60.96	99.90	38.24	22.72
65	100.86	60.14	102.60	36.64	23.50
59	100.20	57.78	101.40	36.49	21.29
66	101.64	54.78	99.90	33.13	21.65
63	101.76	55.01	101.20	36.26	18.75
61	100.20	54.73	102.50	36.20	18.54
57	100.62	58.26	100.40	33.57	24.69
62	100.80	57.45	101.85	36.10	21.35
Crystallizer#15 outlet/crystallizer#16 inlet					
60	102.36	55.28	104.50	34.55	20.73
61	102.54	53.19	102.60	34.21	18.98
53	99.66	57.62	99.90	37.13	20.49
54	101.16	58.66	106.40	32.52	26.14
65	100.98	58.76	101.00	37.13	21.63
54	101.46	58.66	101.10	35.31	23.35
58	102.06	58.14	99.70	35.30	22.84
60	101.22	55.30	99.90	34.53	20.77
60	100.98	55.14	101.20	36.46	18.68
60	101.10	54.24	102.70	34.18	20.07
55	100.62	58.26	100.40	33.57	24.69
58	101.29	56.66	101.76	34.99	21.67
Crystallizer#16 outlet/crystallizer#17 inlet					
53	103.32	55.05	108.30	32.40	22.65
57	102.84	53.91	104.60	32.60	21.31
53	100.14	55.18	100.00	32.90	22.28
55	102.96	56.06	105.20	32.41	23.65
60	100.80	59.88	101.20	33.00	26.88
48	101.28	58.59	102.10	33.30	25.29
48	99.84	60.40	101.00	34.65	25.75
62	100.44	54.30	102.20	33.36	20.94
41	100.86	55.21	101.50	30.15	25.06
44	102.06	53.73	102.00	30.00	23.73
52	101.5	56.2	102.8	32.5	23.8

Crystallizer#17 outlet/reheater inlet					
Temperature ^0C	**Mass. Brix**	**Mass. Purity**	**Nutsch brix**	**Nutsch purity**	**Nurity drop**
50	104.40	54.40		104.40	54.40
44	101.10	53.77	102.80	31.71	22.06
47	100.02	56.99	100.40	36.25	20.74
52	101.04	59.02	100.9	33.59	25.43
56	101.22	58.8	100.8	34.22	24.58
48	101.28	59.18	88.6	31.6	27.58
54	100.86	58.715	100.7	35.45	23.265
62	100.26	56.43	101.1	33.83	22.6
49	101.52	55.437	99.6	30.72	24.717
44	101.94	55.33	101.50	30.15	25.18
51	101.4	56.8	100.1	32.9	23.9
Reheater outlet/centrifugal machine inlet					
53	103.8	55.433	107.2	31.25	24.183
54	99.84	55.05	107.36	30.644	24.406
57	100.5	57.25	99.6	35.34	21.91
60	100.68	55.065	103	33.59	21.475
58	102.96	57.26	100.1	31.37	25.89
56	100.98	58.94	99.6	34.13	24.81
54	100.26	57.39	101.2	35.27	22.12
55	100.5	59.04	101.6	35.43	23.61
59	101.28	55.27	102.1	33.3	21.97
50	101.04	55.107	100.5	32.83	22.277
58	100.74	54.43716498	99.7	32.49749248	21.9396725
60	101.46	56.3	102.1	31.44	24.86
56	101.2	56.4	102.0	33.1	23.3

Centrifugal machine inlet /Final Molasses		
Temperature	Brix	Purity
53	100.6	33.99
54	97.3	34.64
54	97.4	36.14
56	92.2	36.55
55	99.8	33.72
55	99.3	38.94
57	95.2	38.55
64	97.6	36.58
56	99.6	36.68
52	96.2	30.35
Average = 55.6	**97.52**	**35.61**

Appendix – B: Factory Performance Figures for Metahara Sugar Factory (Quantity in Quintals)

		2005/06	2006/07	2007/08	2009/10	2010/11	Average	%cane
Sugar cane crushed		11,478,087	11,704,193	11305414	12,130,022	12,032,489	11,730,041	100.00
Net mixed juice		10,639,893	10,648,613	10,557,959	10,961,901	10841273	10,729,928	91.47
Bagasse		3,283,732	3,400,418	3,210,388	3,460,642	3,465,996	3,364,235	28.68
Final molasses		413,434	414,553	410,822	424,823	393,288	411,384	3.51
Filter cake		414,551	426,757	386,137	376,335	393,288	399,414	3.41
Sugar production	Domestic	1,047,080	1,264,025	1,252,661	1200825	1,200,349	1,192,988	10.17
	Export or Bottling Companies	206,076		5,0000	100,979		71,411	0.61
	Total	1,253,156	1,264,025	1302661	1,301,804	1,200,349	1,264,399	10.78
Sucrose %cane		13.2	12.9	13.8	12.7	11.9	12.9	
Quintals sucrose in cane		1509368.4	1508670.5	1562408.2	1542938.8	1427053.2	1510087.8	12.9
Sucrose loss with	Bagasse	80751.2	84485.5	80307.8	85478.8	83197.2	82844.1	0.706
	Filter cake	8603.4	8146.8	7030.8	7406.1	8134.2	7864.3	0.067
	Final molasses	138861.9	139250.3	142491.6	143647.6	130718.1	138993.9	1.185
	undetermined	6339.3	6487.3	6562.1	6326.0	6279.0	6398.8	0.05455
Total sucrose loss		234555.9	238369.9	236392.4	242858.6	228328.5	236101.0	15.6