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SCHOOL OF CHEMICAL AND BIO- ENGINEERING

Study of Hardness Effect of Clear Juice on the Formation of Evaporator Scale and Optimization of Process Parameters A Case of Metahara Sugar Factory

By

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The undersigned have examined the thesis entitled "**Study of Hardness effect of Clear Juice on the Formation of Evaporator Scale and Optimization of Process Parameters A case of Metahara Sugar Factory"** and submitted in the partial fulfillment of the degree of Master of Science in Chemical Engineering (Process Engineering) complies with the regulation of the University and meets the accepted standards with respect to originality and quality.

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DECLARATION

I declare that this thesis "**Study of Hardness Effect of Clear Juice on the Formation of 'Evaporator Scale and Optimization of Process Parameters A case of Metahara Sugar Factory'** has been composed solely by myself and has not been submitted in any form for another degree, diploma or an award at any University or other institution of the tertiary education. Whenever the contributions of others are involved, every effort is made to indicate this clearly, with due reference to the literature and discussions. Information taken from published and unpublished work of others has been acknowledged in the text and a list of references in my work.

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ABSTRACT

The study focus on the effect of hardness of clear juice for the formation of evaporator scale and optimization of process parameters. The effect of clear juice hardness of the factory have been evaluated in terms of the response variables which include colour, turbidity, calcium oxide, magnesium oxides, reducing sugars and the purity of clear juice out of the clarifier. The reduction of each evaporator scales was monitored by minimizing the colour, turbidity and calcium oxide content of the clear juice. This study was done on lab scale by varying the effect of pH and settling time of clarified juice on the response variables of colour, turbidity and calcium oxide content of clear juice. The statistical analysis was done by using design expert software (7.0.0, version) of the central composite design (CCD). The statistical experimental design combination was done by inserting the highest and lowest values of the factors of the clarified juice. From this analysis method, both factors had an impact on the response variables directly. Both factors had the same significance effects on colour, turbidity and calcium oxide content of the clear juice were found between "(1855.22 - 2369.37) IU, (1720.36 - 2039.67) NTU and, (2142.96 - 2559.68)" ppm, respectively, and pH of (6.7 - 7.2) and a settling time of (3 - 4.66) hours respectively. While purity, reducing sugars and magnesium oxide content of the clear juice were, investigated during the research work. On the other hand, the parameters, which shows the adequacy of the model such as R^2, Adjusted-R^2, predicted-R^2, CV, Press, mean and standard deviation value of each of the three response variables where in the allowable range. Therefore using the optimum values of pH and settling time gives us the optimum output of colour, turbidity and calcium oxide content of clear juice sample, which can be taken as a positive outcome to bring efficient improvement of heat transfer for energy saving and product maximization.

Keywords: pH, settling time, colour, turbidity, calcium oxide, clear juice

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LIST OF SYMBOLS AND ACRONYMS

ANNOVA	Analysis of Variance
Brix	by weight of soluble solid in sugar solution
EDTA	Ethylene di-amine tetra-acetic-acid
EU	Europeans Union
GTP	Growth total product
H.V.A	Handlers-Vereeniging Amsterdam
ICUMSSA	International Commission for Uniform Methods of Sugar Analysis
MEE	Multiple effect evaporator
MJ	Mixed Juice
MSF	Metahara Sugar Factory
NTU	Nephelometric Turbidity Unit
Pol	Polarization
PPM	Parts Per Million
RS	Reducing Sugars
SOP	Standard operational parameters
TCD	Tons of Cane Crushed per day

CHAPTER ONE

1. INTRODUCTION

1.1. Background of the study

Ethiopia is currently working towards being one of the ten top sugar-producing countries in the world. The sugar sector is now regarded as one of the focus areas anticipated to play a vital role in achieving the national GTP. Ethiopia is one of those countries with large potential for sugar production. Utilizing this potential as well as the cheap workforce can be regarded as an excellent opportunity for growth, income generation and socioeconomic impact specifically on employment. To attain the right benefit out of this opportunity, the sugar industry needs to be internationally competitive. This requires running every factory unit by operational standards of the factory. Following standard operational parameter is the main tool in increasing productivity, improving product quality, loss minimization, and brings about ease of operation. To achieve these targets, it is essential to keep all the process parameters of every unit operation within the standard operational limit.

Sugar is a natural sweetener that exists in the leaves of most land plants. However, only sugar cane and sugar beet contain amounts of sugar large enough to be extracted economically on a commercial basis. Sugarcane, botanically known as Saccharum officinarium, is a tall cane shaped plant and is the main source of sugar in tropical countries. Sugar beet, whose botanical name is Beta vulgaris is a tuber with sugar stored in its root and is the main source of sugar in temperate climates. Extracting sugar from sugar cane and sugar beet and manufacturing it into a commercial product involves high energy-consuming processes. Energy is required to provide power to the machinery which extracts the sugar, to heat the process fluid to temperatures at which impurities can easily be removed, to remove excess water and finally to crystallize the sugar by water removal. This high demand for energy makes the overall economy of sugar production highly dependent on the cost of energy (Misheck Gift Mwaba, 2003).

White sugar is produced from sugar cane and sugar beet with a purity of more than 99.8% sucrose (schiweck and Clarke, 1994). However, the remaining 0.2 % or less contains trace

amounts of other carbohydrates, such as polysaccharides, and inorganic and organic non-sugar compounds including colorant polymers. The impurities contribute to a reduction in yield, i.e., Influence the crystallization rate, and lower the sugar quality due to incorporation in the sugar crystal. Color impurities have received much attention due to their complex composition and multitude of chemical pathways, but despite extensive research for more than a century (scheibler, 1869) there are still many unknown aspects concerning the identity, origin, and formation of the undesirable compounds.

Depending on the type of final product to be produced, there are different methods of juice clarification. One such method commonly practiced in the manufacture of plantation white sugar is sulphitation process. In the sulphitation system, clarification is done by heat, lime and Sulphur dioxide gas. The mixed juice is heated to 75^0C, more lime slurry is added to the raw juice and the excess of lime is neutralized by sulfur dioxide gas. The process is called after the name of the acid used for neutralization of excess lime "sulphitation" (Honig P, 1963). The treated juice is heated to slightly above the boiling point and separation of flocculated non-sugars takes place in a continuous settler. From the settler, the clear juice continues in the process, the underflow mud is filtered on continuous drum filter, and the filtrate is recycled to the measured raw juice.

The system of clarification being exercised in Metahara sugar factory is sulphitation process, which requires simultaneous liming with milk of lime prepared to the desired density. Lime slurry is a solution consisting mainly of a coarse suspension and colloidal solution of calcium hydroxide the solubility of calcium hydroxide in water is very low (0.2% at 25^0C). The solubility of lime is greatly increased in sucrose solution so that 10% of sucrose solution dissolves 1.5% Calcium oxide (Mathure, 1978). In the same way, some non-sugar substances present in the juice. The solubility of calcium hydroxides decreases also increase the solubility of lime with the increase in temperature this is a cause of fouling.

Scaling is the accumulation and formation of undesired solid material at phase interfaces (Epistien.N, 1988). In the sugar industry, this will happen to the heat exchanger surfaces of the juice heaters, evaporators, vacuum pans, and centrifugal machines. In a multiple-effect evaporator system, heat is transfer from the vapor chest to the cane sugar juice flowing in the

tubes. During this process, the accumulation of unwanted material on the heat transfer surface takes place, which arises a resistance at the interface of heat transfer. This film resistance layer or deposition of organic and inorganic matter, fibers and other deposits on heat transfer surface are termed as scaling. Scaling was the major unresolved problem in the heat transfer and until now, it cannot be resolved completely. Deposits that are formed on the heat transfer surface consists of various deposits forming minerals. These minerals include some inversely soluble salts such as calcium and magnesium salts. Common deposits forming components are calcium carbonate, calcium sulfate, calcium phosphate, silica, magnesium silicate, iron, zinc phosphate, and zinc hydroxide. Certain elements such as calcium, magnesium, and silica are only moderately soluble in water. Therefore, in this paper, an attempt has been made to perform a detailed analysis regarding the constituents of deposits formed in different sets of a quadruple effect multiple-effect evaporator system of Metehara sugar factory. The sugar industry experiences scaling of evaporators; the extent of Scaling varies from mill to mill, within a season and from season to season for each evaporator. These encrustations were formed from the combined effects of several processes involving inorganic and organic molecules or ions. The type of the scale formed depended on several parameters including the concentrations of scale-forming ions, the amount of dissolved and suspended solids, the PH and the flow properties of the solution. The impurities in the juice deposit onto heating surface areas because heating surface areas have a high temperature at their surface than the surrounding gas and provide nucleation sites for scale growth.

1.2. Statement of the problem

Scaling of heat transferring units in sugar production is a big challenge for the profitability of the plant. Scaling of heat exchangers and evaporators during sugar manufacture reduces productivity and increases energy demand. In the case of Metahara Sugar Factory, scaling is one of the most common problems, first the sugar cane plantation is covered by saline soil, which holds organic and inorganic salts. These salts are coming from the field within the cane and delivered to the factory-processing unit, the juice becomes hard and this would form scale in the tubes of evaporators and their subsequent units. These problems are particularly very serious in the evaporation plant of Metahara Sugar Factory that the scale rate of heat transferring equipment is

very frequent. As a result, the heat exchangers and evaporators tubes don't function properly for more than four days after cleaning. Therefore, the process operation stops every four days for cleaning. The first two sets of evaporators are highly affected by soft salts while hard salts affect the rest three evaporators. As a result, it cause hindering of heat transfer and may incur additional cost for cleaning of evaporators, chemicals and labor force used finally it results in the declining of the factory productivity. Therefore to minimize those mentioned problems we have to measure and evaluate various parameters such as the brix %, pol %, reducing sugars, turbidity, colour, calcium oxide and magnesium oxide content and these parameters shows the degree of hardness of clear juice. Based on this by using different pH range and settling time that are evaluated, this cumulative result will affect the boiling house recovery and minimizes overall factory efficiency.

1.3. Objective of the study

1.3.1. General objective

The main objective of this thesis work was to study the effect of hardness of clear juice for the formation of evaporator scales and optimization of process parameters for low scale formation.

1.3.2. Specific objectives

The specific objectives of this study were to:

- Study the effect of different salts (i.e. calcium oxide and magnesium oxide) on juice clarification process.
- Determine clear juice parameters i.e., (colour, turbidity, Pol %, Brix %, reducing sugar).
- Study the effect of variation of pH and settling time on clear juice clarification.
- Suggest the possible solution for the problem of scaling in the plant.
- Assess the suitability of local lime for juice clarification and their corresponding impurities in relation to the recommended allowable impurities.

1.4. Significances of the study

The main significant in this study provided essential information for sugar producing factories. For any sugar factory, juice clarification system is among the critical unit operations, which can lead to either success or failure of the sugar manufacturing process. Good work performed at the clarification stage pays a considerable benefit in terms of production volume, product quality, reduced cost of production, energy efficiency, etc. The existing method of clarification units has a certain drawbacks. More importantly, incomplete reaction with the juice impurities in the reaction tank creates a formidable challenge to the sugar manufacturing process. In line with this, one of the most outstanding problems that Metahara sugar factory is currently facing is frequent fouling of evaporator and juice heaters tubes and frequent reprocessing of final product mainly due to high clear juice colour, turbidity and calcium oxide. This condition is a major setback, which calls for improvement in the existing clarification system, particularly on the pH control. In addition, after implementation of the technology, it can provide as a benchmark to transfer this technology to other Ethiopians plantation white sugar producing factories. Therefore, the determination of clear juice parameters were important to know the degree of clear juice Hardness in order to prevent scale formation in evaporation plant and plays significant benefit for enhancing boiling house recovery, finally to minimize scale and improvement in the performance of the middle factory.

CHAPTER TWO

2. LITERATURE REVIEW

2.1. Sugar Economy in Ethiopia

Sugar in Ethiopia, serves for direct household consumption and as an intermediate input for other industries like pastries, behaviors like (soft drink, brewery) companies for food processing companies etc. The per capita consumption in Ethiopia is one of the lowest in the world. The current level of per capital consumption is estimated to be about 3.6 kg, which is even below the world average minimum of 5 to 6 kg. The Ethiopian consumption of sugar was forecasted for the coming 10 years; taking into account the Ethiopian population estimated about 100 million in 2009/2010, population growth rate per annum of 2.9% and an annual average economic growth rate of 9%. It is assumed that the per capita sugar consumption could increase at the rate of the economic growth of the nation.

Sugar is produced from cane and beet in over 130 countries in which sugar produced from sugarcane contributes about 65 to 70 % of global production. Brazil, India and China are leading countries and 85% of production was contributed by the top 15 producer countries. Moreover, in the last 50 years these countries were leading the industry. Ethiopia is one of sugar producing country from sugarcane and contributes about 0.18 % of the global sugar production. The use of improved technologies developed through research indifferent sugar producing countries had increased their production and productivity. In Ethiopia, the cane productivity was not maintained from year to year and the potentials are not yet exploited mainly due to the inability to optimize cane production and sugar manufacturing process through the application of improved technologies (Grum A, 2015).

2.2. Production and processing of sugar

Sugar production in Ethiopia was started in 1961/62 E.C when the Wonji/Shoa Sugar Factory was commissioned and produced 15,843 tons of white sugar in the first operation. When sugar cane development began in 1958, Dutch Company, HVA, owned the company. The development of the sugarcane plantation was started on 5000 hectares in the upper reaches of the awash basin

and far from, 110km Southeast of Addis Ababa. Nowadays, there are eight sugars, producing factories in the country. Those of the eight sugar-producing factories have a total production capacity of 280,000 tons of sugar annually. These sugar companies presently produce sugar for the local market as well as for export. White sugar is mainly exported to the neighboring countries such as Djibouti, Kenya, Sudan and the Middle East countries like Yemen and soudiarebia in quantities ranging between 30,000 to 50,000 tons per annum. Raw sugar (partially processed) currently exports to the EU countries under the EBA initiatives in quantities ranging from 14,000 to 15,000 tons.

2.3. General overviews of Metahara Sugar Factory

Metahara Sugar Factory was (established in 1965) is so far among the largest sugar producing factory in Ethiopia. The factory is located 200km South East of the capital, on the Addis-Dire Dawa Djibouti road within the upper Awash Valley. Metahara Sugar Factory has 10,000 hectares under cane plantation, which is wholly owned and managed by the factory and 98% of the cane fields were irrigated by gravity, and electric pumps irrigate 2%. The current installed crushing capacity of the factory is 5000TCD with an annual sugar production of 125,000 tons of plantation white sugar. The factory employees are around 3000 permanent employees and 7000 seasonal employees working mainly on the cane plantation. Currently, the factory generates 6.6MW of electricity from its four boilers has a pressure of (21 bar each) and three turbines (3.3MW capacity each) and consumes all of it. During the off-season, the factory imports power from the national grid. However, the generated power is sufficient for the current factory power requirements.

The main byproduct of the factory, molasses, was used as a raw material for ethanol production, which has a capacity of 50,000l/day. The factory has a plan to double its crushing capacity from 5000 TCD to 10,000 TCD. To meet this capacity, there is a planned expansion in agriculture by an additional 10,000 hectares of plantation area. The land has already been acquired in kesem Area which was 60 Km far from the Factory. The Ministry of Water Resources has been already underway in preparing the irrigation infrastructure for the Kesem fields and seed cane for the area has been planted in a nursery.

2.4. Cane juice

Mixed juice reaches the clarification station as a complex mixture of the integral components of the cane plant. Extraction by crushing and washing operations contribute to the wide variability in the composition of the juice. Juice composition depends not only upon the geographical location, cane variety, season, cultural practice, and maturity at harvest, but also upon the mechanical treatment given to cane during harvesting, transporting, cleaning, and milling (Pieter Honig, 1953).

Table 2. 1: Cane juice composition on soluble dry substances

s/n	Parameters	Juice constituent	g/100g Soluble dry substance	
1	**Sugars**	Sucrose	70.0-90.0	**75.0-94.0**
		Glucose	2.0-4.0	
		Fructose	2.0-4.0	
		Oligosaccharides	0.001-0.05	
2	**Salts**	inorganic acids	1.5-4.5	**3.0-4.5**
		organic acids	1.0-3.0	
3	**Organic acids**	Carboxylic acids	1.1-3.0	**1.5-5.5**
		Amino acids	0.5-2.5	
4	**Other-organic, non-sugars**	Protein	0.5-0.6	
		Starch	0.001-0.18	
		Soluble polysaccharides	0.03-0.50	
		Waxes, fats, phosphatides	0.04-0.15	

Source: *M.Sc. Thesis by Mona Ghazi Zaeib, December 2004*

2.5. Purpose of juice clarification process

Clarification, as used in the sugar industry, refers to the precipitation and removal of all possible non-sugars, organic and inorganic, and the preservation of the maximum sucrose and reducing sugars possible in clarifying juice (Baikow, 1982). There are many methods applied to juice

clarification these include (i) Defecation (the treatment with lime and heat), (ii) Sulphitation (treatment with SO_2), (iii) Carbonation (treatment with CO_2) and (iv) magnesia (treatment with MgO). The overall objectives of these methods are to produce rapid settling of suspended particles in the clarifiers, raise juice pH while lowering the percent volume of sediments or "mud" and producing a clear liquid. The degree of clarification has great bearing on the subsequent stations of the factory, effecting the evaporation, the pan boiling, the centrifuging, the quality of products, and most important of all the yield of sugar (J.C.P.Chen, 1985).

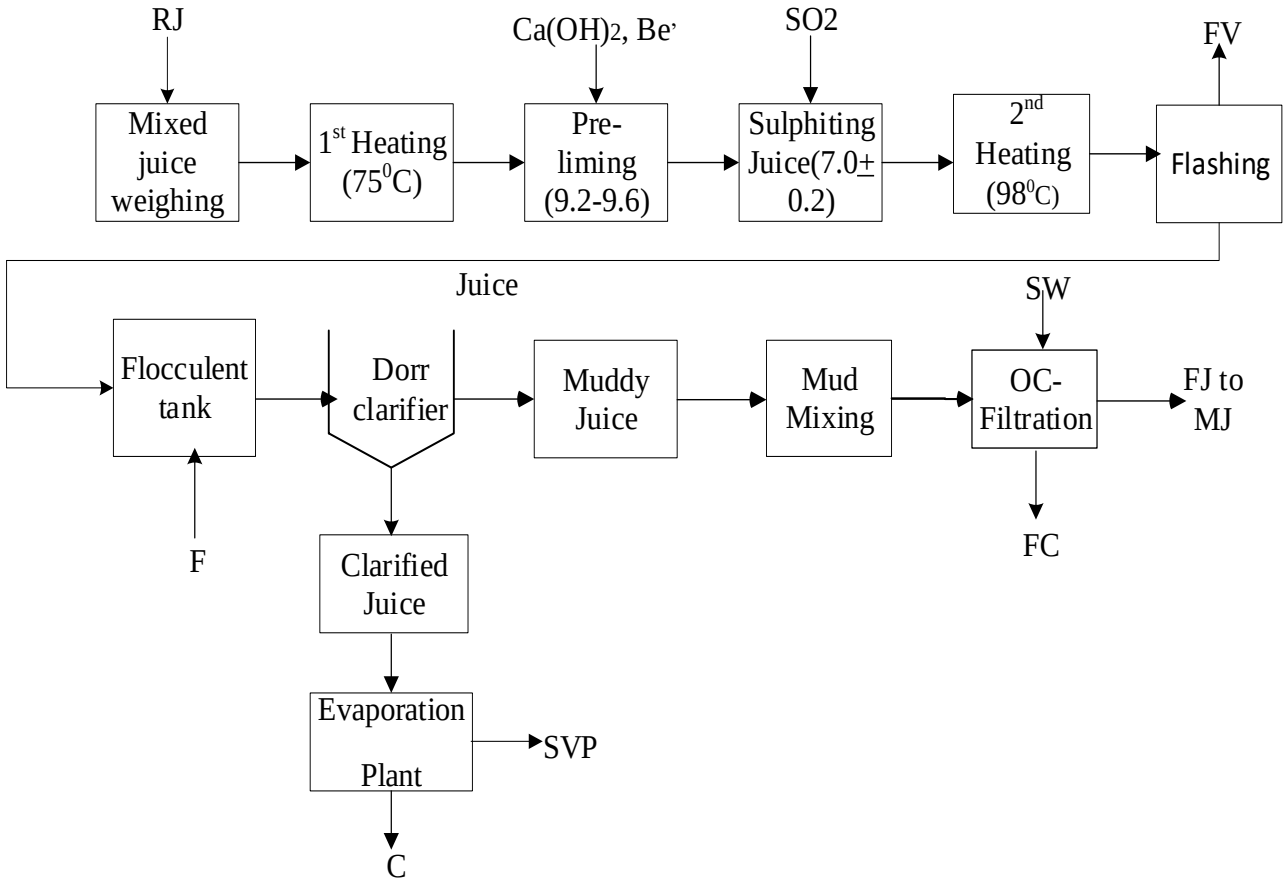


Figure 2. 1: Schematic flow diagram of the juice clarification process

Key Terms:

C-condensates, **F**-flocculent**, FC**-filter cake**, FV**-flash vapor**, FMJ**-filtrate to mixed juice, **RJ**-raw juice**, SVP**- syrup to vacuum pans, **SW**- sweetening of water/Press water

Clarification should produce juice of high clarity with minimum unfavorable effects on the subsequent recovery of sucrose from the clarified juice through maximum removal of non-sugars at the earliest possible stage in the process. During a typical purification process, non-sugar elimination efficiency of 20 to 30% is achieved, and the remaining non-sugars become destabilized to the point where they are harmless to the later operation and finally, end up in molasses. In addition to sufficient addition of lime, temperature, pH, and alkalinity of the juice during liming and sulphitation should carefully controlled to achieve proper non-sugar elimination. The reaction of lime with non-sugars (NS) are complex because, during liming, different non-sugars behave differently. According to their reaction with lime, non-sugars were grouped into the following four groups:

Non-sugars that react with lime to produce a precipitate, Non-sugars that do not react with lime, but they are destabilized, Non-sugars that do not react with lime but they are decomposed and Non- sugars that do not react with lime under any of the conditions. The first three groups are removable non-sugars, and the fourth group contains non-removable non-sugars. Oxalates, phosphates, and sulfates present in the juice are good examples of the first group while Colloids are examples of the second group. Colloids do not precipitate with lime but destabilize. Invert sugar is the best example of the third group. Invert sugars does not react with lime but decomposed by lime to form lactic acid and other acids. The acids then precipitate with lime and removed from the juice by filtration. Raffinose, betaine, and amino acids are the best examples of the fourth group. Non-removable, Non-sugars do not react with lime under any circumstances, so the NRNS stay in the juice throughout the remaining operations and end up in molasses. RNS count for about one-third of the total non-sugars in the juice have the right chemistry to precipitate with lime (Abayneh B, 2012).

NSE is calculated using the following formula:

$$\mathrm{NSE} = \frac{(P_2 - P_1)}{P_2(100 - P_1)} \times 1000 \qquad (2.1)$$

Where, P_1 = Mixed juice purity, P_2 = Clear juice purity

NSE = Non Sugar Elimination

2.5.1. Operation standards for cane and mixed juice

The basic raw material (sugar cane) and mixed juice are required to have their specification that meets certain standards. Such an operational standard is useful in making decisions as to accept reprocess or reject inputs/ or products.

2.5.2. Quality of the cane

The primary source of colour in sugar is the processing of damaged cane and high levels of impurities. The two major areas of plant operation where control is essential for maintaining a high standard quality of sugar, cane quality, and process operation. Immature tops should remove properly since; it contains color-forming compounds that contribute to the dark coloration of sugar crystals. On top of this, the cane should be free of foreign materials especially metals and stones. Mature cane processing is favorable even for maintaining a high standard of sugar quality. The cane should meet the following specifications.

Table 2. 2: Cane quality standards

S/N	Parameters	Standard
1	Recoverable sugar (Field Yield)	$\geq$ 11 %
2	Fiber content	13.5 – 14.5 %
3	Trash content	$\leq$ 3 %
4	Burning to weighing time	$\leq$ 24 hr.
5	RDS of absolute juice	19.5-20.5

***RDS**-Reducing Dry Substances*

2.6. Juice clarification

A critical step in the manufacture of any sugar (Raw, plantation white or refined) is a clarification of the sugar juice. Clarification is the process of removing the constituents of the juice other than sucrose and at the same time, minimizes loss of sucrose and color formation. Clarification is, therefore, an essential step to obtain high yield and high-quality sugar (Dionisi et al.,, 2008).

Juice clarification is done for two purposes, first of all to raise the juice pH and removal of impurities like dissolved inorganic non-sugars and insoluble suspended matters i.e., rendering the juice opaque, viscous and dark in colour and second leaching of the juice to render it brilliant and light in colour for the manufacture of white sugar. To obtain high quality clarified juice, removal of components other than sucrose should be maximized, and losses of sucrose and formation of color should be minimized. Proteinaceous and waxy matter, some silica acid (hydrated), and sesquioxides can be removed by heating, while liming neutralizes acids, forms calcium phosphates, and coagulates colloidal particles. To achieve best clarification efficiency, process parameters at every clarification unit should closely be controlled ((Mathur, 1981; Saska et al. 2010)).

To improve decolonization of the juice, phosphatation and sulphtation can be supported by a flocculating agent (Cartier et al., 1997). Generally added at a level corresponding to 200 - 400 ppm by weight of P_2O_5 on the bases of the sugar in juice or syrup (Rundell and Portage.,1975). Rapid flocculation and sedimentation of suspended particles in primary cane sugar juice has achieved using a high molecular weight anionic polymer flocculants. The performance of flocculants to enhance the flocculation and sedimentation of cane sugar juice particles evaluated by turbidity and settling rate measurements (Doherty, et al., 2003). Flocculation removes insoluble suspended particles and colloidal impurities by aggregating them into flocs. The addition of 3 to 5 ppm of anionic polymer flocculent (APF) into sulfited juice improves the clarity of the juice and mud separation.

2.6.1. Raw juice heating

Juice heating is excited in several vessels, which are called juice heaters or Pre-heaters. In the clarification, the system of all Sugar Factories (solicitation systems) juice heating sequence is as follows. Measured mixed juice was heated to a temperature of 75°C and directed to pre-liming and sulphiting. The main purpose of such heating is the following.

- Due to the application of heat to the juice, a certain organic constituents like proteins are coagulated.
- Heat destroys microorganisms and enzymes preventing loss of sucrose by microbiological Activity.

- Heat accelerates the reaction rate between the juice acids and the lime, which subsequently added.

Table 2. 3: Operational standard for juice heating

S/N	Juice heating	Outlet Temp. in ^{o}C
1	Primary juice heating	70 – 75
2	Secondary juice heating	100 – 102
3	Clear Juice Pre-heating	112 – 115
4	Press water temperature	98 – 100

2.6.2. Defecation process

Lime which, since the beginnings of sugar manufacturing, has reminded the universal basic deficient; the treatment with lime is called "defecation", sulfurous acid SO_2 "sulphtation", phosphoric acid, from P_2O_5"phosphatation,"Carbonic acid, from CO_2 "carbonation" and Magnesia, from (MgO). We shall study the various process separately.

We shall start with defecation; this is the only one of the five methods mentioned which is always practiced. We shall not discuss the chemical effects resulting from the reaction of lime on the juice. Many of the organic acids were eliminated since their lime salts are insoluble albuminoidal matter is coagulated. Part of the pectin and some of the colouring matters were destroyed or rendered insoluble. However, the elimination of impurities is relatively insignificant, the purity of the defecated juice being approximately the same as that of the juice before treatment (E.Hugot, 1986).

2.6.3. Lime quality

The quality of lime used is important in many countries, the only lime obtainable is very impure approximately 60% (CaO) containing much sand and unburned stone. It is recommended that lime containing more than 2% of (MgO) or oxides of iron and aluminum should be avoid. These impurities would cause deposits in at the multiple effect evaporators and magnesia would give in treble defecation. However, magnesia sometimes used systematically with lime that, precisely in order to avoid scale formation in the evaporators.

Table 2. 4: The relation between degrees Baume and lime content of the milk

Degree Baume	Density (g/cm^3)	Calcium Oxide (g/lit)
1	1.007	7.5
2	1.014	16.5
3	1.021	26
5	1.036	46
10	1.074	94
15	1.117	148
20	1.1	206

A good quality of lime should test about 90 - 95% calcium oxide. Hydrated lime $Ca(OH)_2$, is also employed its activity being proportional to its calcium oxide content. Pure hydrated lime contains 56/74 = 76%of calcium oxide. Milk of lime some factories still use lime directly, adding it to the juice in the solid state. The solubility of lime in juice increases with the sugar content and decreases with increasing temperature. At 80°C, calcium oxide dissolves in a juice of sucrose. As the solution is relatively slow and difficult, the use of lime in the pulverized state is not recommended; certain portions of the juice will be over limed, other insufficiently limed. It is of advantage to make first a milk of lime, by mixing the pulverized lime or lump quicklime with water (E.Hugot, 1986).

2.6.4. Milk of lime preparation

Lime can be used in the clarification process as a slaked lump lime, powdered hydrated lime, or powdered quicklime, whichever is most feasible economically destruction (J.C.P.Chen,1985). The lime should be fresh and well burnt with Calcium Oxide content of above 85 %.The addition of lime in solid (powdered) form is still practiced, but it is not recommended because of the slow solubility and certainly, of local over liming and Lime slurry temperature in the slacker should be 98-102^{o}C. Milk of lime is prepared by mixing the lime with water to the desired density, usually, 15 Baume′, 148g (CaO) per liter, 1.116 weights in Kg of one-liter milk of lime, 13.26% (CaO). The density 15 Beo produces great ash elimination and low residual calcium in clarifying juice.

A considerable quantity of soluble impurities can enter the juice if the water contains any hardness or any soluble impurities, so condensate water from the third and fourth evaporators should be used for slaking and diluting to a Baume of $6^{o} - 8^{0}$ (Pieter Honig, 1953).

Figure 2.2: Milk of lime preparation plant of Metahara Sugar Factory

2.6.5. Density of lime

The relation between degrees Baume and lime content of milk of lime is given by the concentration of milk of lime is measured in degree Baume.

Table 2. 5: The lime of allowable impurities

Impurity	Allowable impurity (%)
Magnesium oxide (MgO)	1.0
Silica	1.5
Moisture	2.0
Iron oxide and alumina	2.0
Sulphates	0.5
Carbonic acid	2.0

2.6.6. The effect of high impurities in lime

The following is the effect high impurity in lime on operation:

- High percentage of silica in lime retards the settling of juices and results in the formation of heavier scales inside the tubes of heaters and evaporators.
- High percentage of magnesia retards filtration and rate of settling.
- High percentage of iron affects the colour of the juice. Both iron and aluminum salts cause scaling on the evaporators tubes.

2.7. Sequence of liming and heating

2.7.1. Juice liming process

Liming (defecation) is indispensable and employed in all types of cane juice clarification. Liming in hot juice precipitates matter that is more nitrogenous, saves lime consumption, and prevents the juice from being infected with leuconostoc mesentriodes (Mathur, 1981).

Cold liming: All the lime is added to the cold juice; the limed juice is rapidly heated to the final temperature (almost to the boiling point or just above) the precipitate consists mainly of calcium phosphate, iron and aluminum hydroxides (the advantages of this process are the simplicity and ease of control of the method (Pieter Honig, 1953).

Hot liming: The raw juice is heated and the lime is added after the juice has reached the final temperature. Certain colloids (albumin and hydrous silica) are precipitated with heat and the pH of raw juice (Honig P, 1953).

Intermittent liming: In this process, part of the lime was added to the cold juice to increase the pH from 6.1- 6.4 and then heated to the boiling point or slightly above with further liming to increase the pH to 7.4 - 7.8. The purpose of these modifications to gain the advantages of heating acid juice (colloid precipitation),while avoiding the possible disadvantages of hot liming inversion and destruction (J.C.P.Chen,1985).

Fractional liming and double heating: this process is the most successful of all the intermittent liming practices. It consists of liming the juice to pH 6.4, boiling, liming to pH 7.6, and again boiling and settling; the essential feature is the second heating after the second liming. The advantages found are much greater rapidity of settling, 35% less lime used them for cold liming, greater elimination of non-sugars, reduction in mud volume, and greater clarity of juice.

2.7.2. Effects of pH on juice clarification

The juice should be neutralized with lime to pH > 7 to avoid losses by inversion of sucrose during settling and the concentration of the juice. Excessive liming to pH > 8.5 has to be avoided because this destroys reducing sugars with the attendant color increase, the formation of acid decomposition products (drop in pH), re-solution of some nitrogenous bodies, and the formation of soluble lime salts, which causes sucrose losses and hinders sugar crystallization (Honig P, 1953). The optimum pH to which juice should be limed is dependent on many conditions and varies with the location of the factory, the variety, and maturity of the cane, and other local conditions (James C.P.Chen, 1985).

Generally, the minimum of lime that will give the clear juice with a final reaction close to pH of 7 is most desirable; to neutralize the charge on the fine suspended particles in the juice to facilitate coagulation and settling. In addition, pH is important to the rate at which certain reaction occurs especially the precipitation of calcium phosphate. The juice pH was shown to have implications on the inversion losses, color formation, loss of sugar, sugar quality, and scaling in subsequent process (Rein, 2002). Also, some studies have indicated that sucrose loss as a percentage of total sucrose per hour in Clarifiers decreases as the pH increases, and ranges from 3% at a pH of 5 to 0.1% at a pH of 6.5 (saska, 2002).

2.7.3. Effects of time on juice clarification

The retention time of juice in the Clarifiers has a great effect on the juice and its components. Retention of juice at an elevated temperature leads to inversion and reducing sugars degradation from organic acids, resulting in purity and pH drop (J.C.P.Chen, 1985). If the juice is refractory or contains a large proportion of suspended matter, it may be logical to hold the juice in the clarifier for longer periods. However, excessive capacity clarifies that hold juice for long periods result in higher levels of inversion (Baikow, 1982). The minimum retention time recommended for the Rapi Dorr 444 clarifier is 2 hours and a maximum of 3 hours. The retention time may vary according to the type of liming technique utilized as some methods may result in a slower settling rate or the reactions of the deficiencies in the juice may be slower and require more time than other methods.

2.7.4. Effects of temperature on juice clarification

Temperatures just above the boiling point say (103-105ºC), is the maximum for good practice and if the temperature drops below 75ºC there is a chance of loss due to micro-activity (drop in pH). The effect of temperature in juice clarification is to accelerate the rate at which chemical reactions occur in the juice. The effect of temperature in juice clarification is best described as a comparison between hot and cold liming. Investigations carried out into the effects of cold versus hot liming indicated that hot liming was more favorable as it removed more colour, dextran, oligosaccharides, and produced clarified juice with lower calcium levels, but results in higher mud levels and a slower floc settling rate (Eggleston.G, 2000). However, the main advantage of hot liming is the reduction of scale formation in juice heaters and evaporators because of increased calcium removal (Doherty et. al, 2002).

2.8. Chemical reactions occurring in simple juice clarification

A wide range of chemical and physical reactions takes place in the juice. They can be classified as those due to pH change, due to changes in concentration and due to the increase in temperature. These reactions can be subdivided into chemical reactions and physical reactions. The major chemical reaction is that of the cation with the phosphate ion to form phosphate

intermediates and to precipitate tri-calcium phosphates $Ca_3(PO_4)_2$; Protein is denatured by heating (denaturing temperature is varied by pH). It then coats the solid particles and the phosphate flocks and imparts an apparent negative charge. Hot liming procedures result in the most complete at the point for many proteins is below pH of 6; Amino acids are not changed much by clarification, though at high pH they can react with reducing sugars to give increased color; Reducing sugars are also stable, they can be destroyed at high pH and temperatures in excess of 100^0C. The rate is just significant at a pH of six but increases by a factor of five for each pH increase; Sucrose is inverted at low pH, high temperature; Waxes, gums, and pectin has are denatured but otherwise unchanged (Peter Rein, 2007).

2.8.1. Chemical reactions of clarification

In a complex system as represented by cane juice the chemical reactions that occur during clarification are not fully understood. Fundamentally, reactions will take place preferentially, which will result in a system of lower free energy content. Defecation, as outlined earlier is the most used method of clarification, therefore in looking at the reactions in clarification; defecation will be the clarification method of choice. The solubility of calcium oxide in water is about 0.12% at 25ºc, but it is greatly increasing in a solution of sucrose. The solubility decreases with increase in temperature. Calcium hydroxide is a relatively strong base of a divalent metal ionizes in solution in two steps:

$$Ca(OH)_2 \longleftrightarrow CaOH^+ + 2OH^- \quad (2.2)$$

$$CaOH^+ \longleftrightarrow Ca^{+2} + OH^- \quad (2.3)$$

The chemical activity of compound depends principally upon the activity of the Ca^{+2} and the OH^- ions, although the $CaOH^+$ ions may take part in the reaction also. Because the secondary ionization is small, the concentration of Ca^{+2} is low, with less than 10% of calcium as Ca^{+2}in sucrose solution containing 0.3-0.5%calcium oxide (Honig P, 1953). The most important factor in efficient clarification is the phosphate content of the juice. In mixed juice, the phosphates are inorganic as well as organic. The inorganic phosphates exist as free phosphate ions, whereas the organics exist in the form of phospholipids, phosphoproteins, nucleotide phosphates, and hexose

phosphates. Understandably, only the free phosphate ions take part in juice clarification. If the inorganic phosphate level in raw juice is less than 300 ppm, the juice cannot properly clarified, and the addition of phosphate is required (James C.P.Chen,, 1985).

In addition, the amount of inorganic phosphate left in clarifying juice should not be less than 10 ppm; otherwise, there exists a considerable danger that the juice has been over limed, a condition accompanied by a higher non-sugar content of clarified juice, increased lime salts, and poor resulting sugars (Honig.P, 1953). The reaction of lime and phosphate in juice leads to precipitation of Calcium phosphate. This reaction is rather complex due to the reactions of phosphoric acid, calcium hydroxide, and the presence of other organic and inorganic compounds. Calcium phosphate reaction rate is very slow and the precipitate is formed in two forms, tri-calcium phosphate, $Ca_3(PO_4)_2$ and calcium hydrogen phosphate $CaHPO_4$, as shown by the following reactions.

$$Ca^{+2} + HPO_4^{-2} \longrightarrow CaHPO_4\downarrow \quad (2.4)$$

$$3Ca^{+2} + 2PO_4^{-3} \longrightarrow Ca_3(PO_4)_2\downarrow \quad (2.5)$$

The formation of calcium hydrogen phosphate has a faster reaction rate, and as it approaches completion, the rate of the formation of tri-calcium phosphate increases. The rate of both reactions were greatly increased at high temperatures and increase the hydrogen ion (H^+) concentration, which has been suggested as a contributor to the drop in the pH observed when the juice is heated. Approximately 80-90% of the phosphate present in juice precipitated with lime. It was shown that at a pH of about 8.5, about 99% of inorganic phosphate added to juice is precipitated, but only 90% of phosphorus occurring in the cane plant is precipitated (Honig P, 1953). Reactions of sucrose and reducing sugars are undesirable in the clarification process. Reaction of sucrose that may occur includes the hydrolysis of sucrose to reducing sugars (D-Fructose and D-glucose) as follows:

$$C_{12}H_{22}O_{11} + H_2O \longrightarrow C_6H_{12}O_6 + C_6H_{12}O_6 \quad (2.6)$$

Sucrose D-glucose D-fructose

The reaction is irreversible and accelerated at low pH and high temperature. However, this reaction is negligible at pH above 7.2 the sucrose loss during hydrolysis is irreversible and hence this reaction must be kept to a minimum during clarification.

Table 2. 6: Influence of pH, temperature, and time, on the destruction of RS and sucrose

Influence of ↓ On	pH		Temperature	Time
	<7,acidic	>7, alkaline		
Reducing Sugars	No Destruction	Destruction is Moderate, if temperature is <55°C;decomposition Products Uncolored Destruction is considerable if temperature is > 55°C;formation of Many Organic acids and colored Compounds	Generally Raising temperatures accelerates The Rate of reactions accelerates	The quantity of Formed Decomposition products depends on the length of the time exposure
Sucrose	Destruction by Inversion; formation of Invert sugar	No Destruction		

Source: *Pieter Honig. (1953) Principles of sugar technology*

Reducing sugars, on the other hand, are naturally occurring in cane juice as glucose, fructose, and mannose. Mannose found only in minute quantities, but together the reducing sugars are the second most abundant component in juice calculated on dissolved solids. Unlike sucrose, reducing sugars are very stable at low pH, but readily oxidized under alkaline conditions of pH above eight. The oxidation of reducing sugars is undesirable since the products of the oxidation are acids and brown colored compounds that impart colour of crystals (Honig P, 1953). Reducing sugars combine with amino acids to from undesirable highly colored products.

2.9. pH control in sugar industry

The sugar industries, before the introduction of the pH method for measuring the reactions of sugar juices and intermediate liquors, the only means available were test paper or titration with standard acid or alkali to an endpoint depending on some indicator. These methods measured only the quantity of acidity or alkalinity, whereas the hydrogen ion concentration or pH is the measure of the intensity of the acidity or alkalinity. Because the sucrose inverting power of an acid is a direct function of dissociation (i.e. If the amount of hydrogen ion concentration in the solution) pH control in sugar industries is highly emphasized.

2.9.1. Conditions for efficient pH control

In order to measure effective pH control in the factory the operation should have fulfills steady flow of mixed juice, uniform concentration milk of lime, effective mixing of the juice with the reagent, optimum reaction time of each treatment, use of pH electrodes withstanding high temperature, calibration of pH electrodes at regular intervals and automatic pH control system.

2.9.2. Role of phosphate in juice clarification

The phosphate content of the juice is the most important factor in efficient clarification. In sugar cane, the phosphates are found as inorganic as well as organic forms. The inorganic phosphate exists as free phosphate ions, whereas the organics exists in the form of phospholipids, phosphoproteins, and hexophophates, etc. Only the free phosphate ions (inorganic form) take part in juice clarification. Therefore, juices with an adequate quantity of inorganic phosphate are most desirable. During cane growing, if fertilizers are not properly applied, there may be more organic phosphate in the juice than inorganic phosphate. Then the juice may not respond well to clarification. It is demonstrated that if the inorganic phosphate level in raw juice is less than 300 ppm (W/V), the juice cannot be properly clarified and the addition of phosphate is required. In the phosphate deficient cane juice, the addition of soluble phosphate to mixed juice to bring percentage to the minimum (0.03%) triple superphosphate (48%) is generally used. Dicalcium phosphate, ammonium phosphate or syrupy phosphoric acid may be used for this purpose. The controlling factor is the relative cost per unit.

2.10. Juice Sulphitation

The production of sulfur dioxide occurs when sulfur is burned in a current of air. To obtain complete combustion of sulfur, a weight of air equal to eight or nine times the weight of sugar would have to be introduced. In any type of sulfur burner, the air supplied to the furnace should be dry to avoid the formation of sulfuric acid. The sulfuric acid causes corrosion of pipeline and formation of calcium sulfate in the juice, which leads to increased rates of scaling in heat transfer equipment.

In order to achieve successful sulphitation the following requirements are considered; Steady juice flow, proportioning devices for milk of lime, efficient arrangement of mixing and absorption of sulfur dioxide, sufficient capacity of the reaction tank to ensure the required reaction, correct pH control on liming and splitting.

2.10.1. Harmful effects of SO_2 consumption with sugar

Sulfur dioxide has traditionally been used in food processing and produces storage to minimize colour formation due to browning reactions associated with amino acids interacting with invert sugars in the Millard reaction. Sugar beet processors routinely use sulphur dioxide in process streams for the same purpose. Among sugar cane, processors worldwide there is mixed interest in the usage of sulphitation. In the United States, sulphitation has rarely been used in cane raw sugar factories since the 1950's. Today, there is renewed interest in the effectiveness of sulfur dioxide as a colour retardant as many US factories are considering the production of high quality low colour raw sugar to be sold as a food grade sugar (Anonymous, 1996).

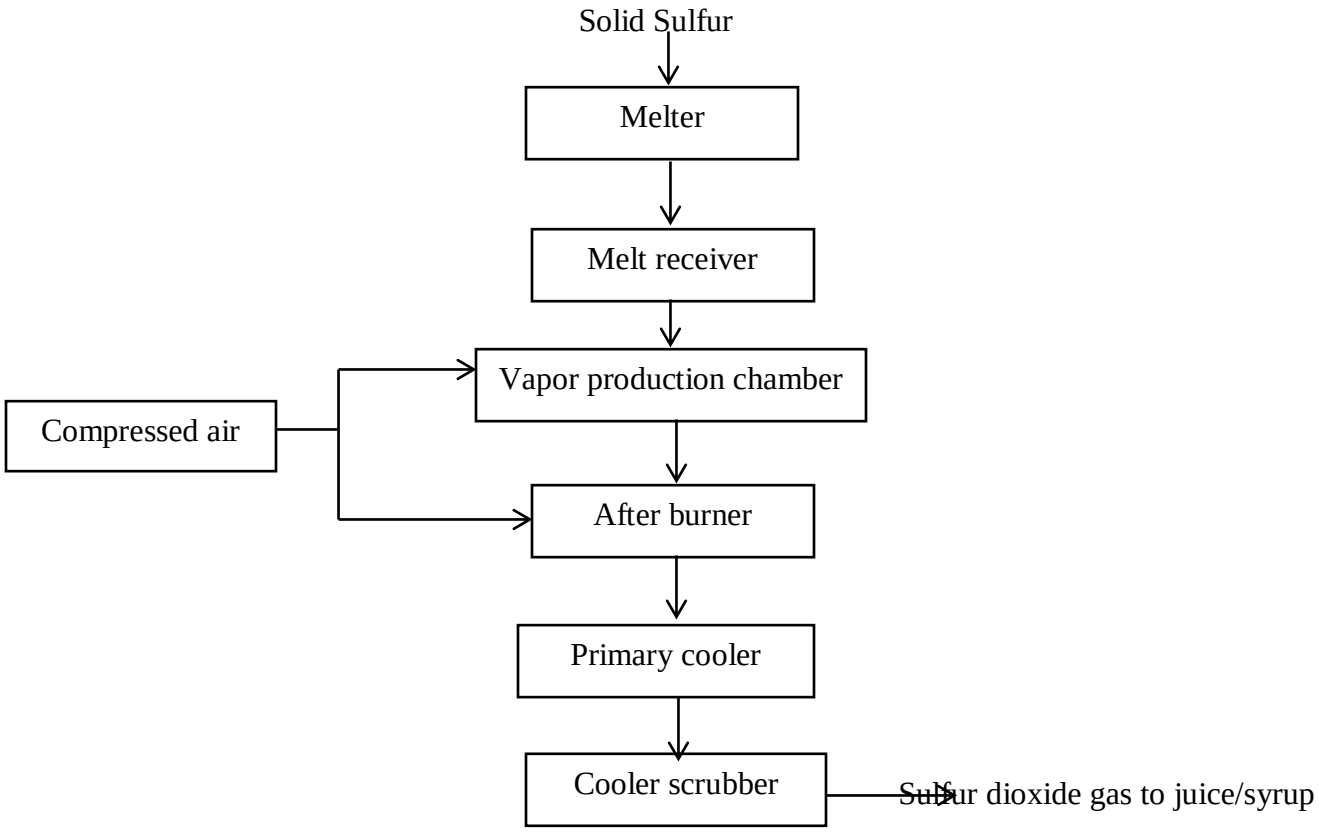


Figure 2. 3: Flow diagram of sulfur dioxide (SO_2) gas generating unit

2.10.2. Action of SO_2 gas on juice clarification

Sulfur dioxide neutralizes the excess quantity of lime added and precipitates calcium sulfite

$$Ca(OH)_2 + H_2SO_3 \longrightarrow CaSO_3 + 2H_2O \quad (2.7)$$

If more sulfur dioxide gas is passed and the juice is made slightly acidic, part of calcium sulfite is converted into soluble calcium bisulfite, which is undesired.

$$CaSO_3 + H_2SO_3 \longrightarrow Ca(HSO_3)_2 \quad (2.8)$$

Further addition of lime increases the pH and precipitation of calcium sulfate starts again. The Precipitation is complete when all the acidity is neutralized by lime.

$$2H_2SO_3 + 2Ca(OH)_2 \longrightarrow 2CaSO_3 + 4H_2O \quad (2.9)$$

If highly acidic juice containing the soluble calcium bisulphate in solution is boiled, it again decomposes into precipitated calcium sulfate and sulfur dioxide.

$$Ca(SHO_3)_2 \longrightarrow CaSO_3 + SO_2 \quad (2.10)$$

Sulphur dioxide bleach the juice by acting on the colouring matter. Sulfurous acid is a strong bleaching agent. It bleaches the colouring matter originally present in the cane juice. It prevents or slows down the colour formation in later stages of the processing. It reduces the ferric salts which are highly coloured compounds formed by the action of iron of the equipment with polyphenols into colourless ferrous compounds. Sulfur dioxide gas decreases the viscosity of the juices. The lower the viscosity of juice, the easier is the crystallization of low sulphur dioxide content, of fewer than 14 %, above 16 % sublimation is likely to occur.

2.10.3. Preliming (alkaline sulphitation) – Wonji Shoa Experience

The mixing of lime and juice is done with the help of a power driven agitating device, a good mixing performance results can be observed by determining the pH of the treated juice sample repeatedly at the same flow rate. The action of lime on cane juice is already noted. Great care must be taken is its action on the reducing sugars of cane juice, which forms harmful coloured decomposition products. This negative effect is controlled by limiting the temperature and evaluation and optimization of process conditions for lime saccharate clarification retention time of juice in the pre liming tank. The retention time should be minimum possible with effective mixing (less than a minute). The juice is pre limed to pH of 9.5 - 10.0. The excess of lime is neutralized by sulfur dioxide gas in the sulphitation tank.

2.10.4. Simultaneous sulphitation (neutral sulphitation) – Metahara Experience

In the simultaneous sulphitation system, the lime remains in contact with the juice for a very short period. The raw juice is heated to 75 ^{0}C and directed to the sulphitation vessel where lime milk and sulfur dioxide gas are admitted in the sulphitation tank. The juice was limed at the inlet to the sulphitator to pH of 8.5-9.5 and neutralized with sulfur dioxide gas. The advantage of the system is that the time in which reducing sugars exposed to higher alkalinity is negligible and reaction of lime with reducing sugars is very limited.

2.10.5 Shock liming modification–Finchaa Experience

The mixed juice heated to 75 ^{0}C is first prelimed to pH of 7.2–7.5. After a very short period, the same juice is subjected to second liming (shock liming) where its pH is raised to 9.2 - 9.5 and gassed with sulfur dioxide simultaneously in a suitably designed sulphitator tank. After gas treatment, the pH is regulated until 7.2-7.5 again heated to 100-102 ^{0}C in secondary juice heaters and directed to settlers through the flash tank.

2.11. The flash tank

To avoid formation of convection currents and get rid of air and gas bubbles the treated juice fed to the clarifier must be at constant temperature. To obtain these conditions, treated juice is heated to slightly above boiling point (98 – 102 ^{0}C). When this juice is subjected to atmospheric pressure in the flash tank, it will flash (auto evaporation) and its temperature will go down to standard for which convection current in the clarifier is protected. The flashing process will get rid of air or gas bubbles contained in the juice. The treated juice from the secondary heating, which is under the pressure provided by sulphited juice pump, enters the flash vessel at atmospheric pressure. The juice enters tangentially into the flash tank. The air bubbles attached to the suspended particles are there by removed; otherwise, they would prevent the settling.

2.12. Flocculants

Flocculants form a bridge between two or more particles uniting the solid particles into a loose porous state. A high molecular weight flocculants were used at clarification, resulting in a fast bridging of very small particles present in the juice, whilst lower molecular weight flocculants are recommended at filtration of mud where flocks are small and have a higher resistance to shear (Edey, 1999).

When flocculants (anionic) were applied in mud filtration, the following advantages were achieved:

- Insoluble solid content of the filtrate was reduced from 2.0 to 0.4 %.
- The moisture content in the filter cake was reduced from 79.5 to 75.6.
- The percentage of Pol in mud was reduced by about 30 %.

2.13. The juice clarifier

The clarifying agents added and forms precipitate in the clarification. The separation of this precipitate or mud was done in a specially designed tank called the clarifier. The precipitate settles at the bottom and the clear juice was extracted at the top. A good clarifier should produce good juice quality, should have short retention time and more efficient mud removed. Any accumulation of mud that was not properly scraped away will crust over rapidly and cool in temperature that permits bacterial action and souring, resulting sugar loss. Retention of juice at an elevated temperature leads to inversion and reducing sugar degradation to form organic acid, resulting in purity and pH drop. On the other hand, if the temperature drops below 75 ^{0}C, there is a chance of loss due to micro activity. For a better control, the difference in pH between mud and clarified juice should not be more than 0.2 units.

2.13.1. Juice settling

In the clarifier, the suspended solids are allowed to settle out from the juice. The juice flows upward at a velocity low enough to allow the precipitate to settle. The precipitate are falling down against rising streams if juice going to the overflow point. Obviously, for effective separation, the settling rate must be faster than the up-flow streams of juice so that net direction of particle movement is downward. If juice is drawn from only one compartment of the clarifier, a very high upward velocities of juice exists which restricts the falling rate of the precipitate or particles entertainment to the over flow clear juice occurs. The Dorr clarifiers have a settling tray, which are supposed to segregate the setting juice into compartments. Liquid - Solid separation occurs utilizing the cross sectional area of each tray. This system reduces the overall upflow velocity dividing it by the number of settling trays.

2.14. Dorr clarifier

The model consists of four compartments with separate provisions for feed, over flow take-off and mud withdrawal, allowing the unit to operate as four totally independent Clarifiers enclosed in a common housing. The juice was introduced at the top center of each compartment through a central hollow shaft. As the feed enters each compartment, it strikes the deflector baffle, and then

flows outward at a decreasing velocity, with minimized turbulence. The scraper was arranged on the rotating central shaft in each compartment, which moves the settled mud to the mud discharge boot at the bottom of the tray to be withdrawn separately. Each compartment has its own overflow piping to remove the clarified juice at multiple points around the periphery. A single overflow box collects the clarified juice from all compartments. The clear juice from each compartment were taken off by means of the overflow tubes due to hydrostatic head. The overflow tubes are adjustable, in height, which enables to regulate clear juice flow rate from each compartment. The muddy juice was taken off by means of hydrostatic head (P.Rein, 2007).

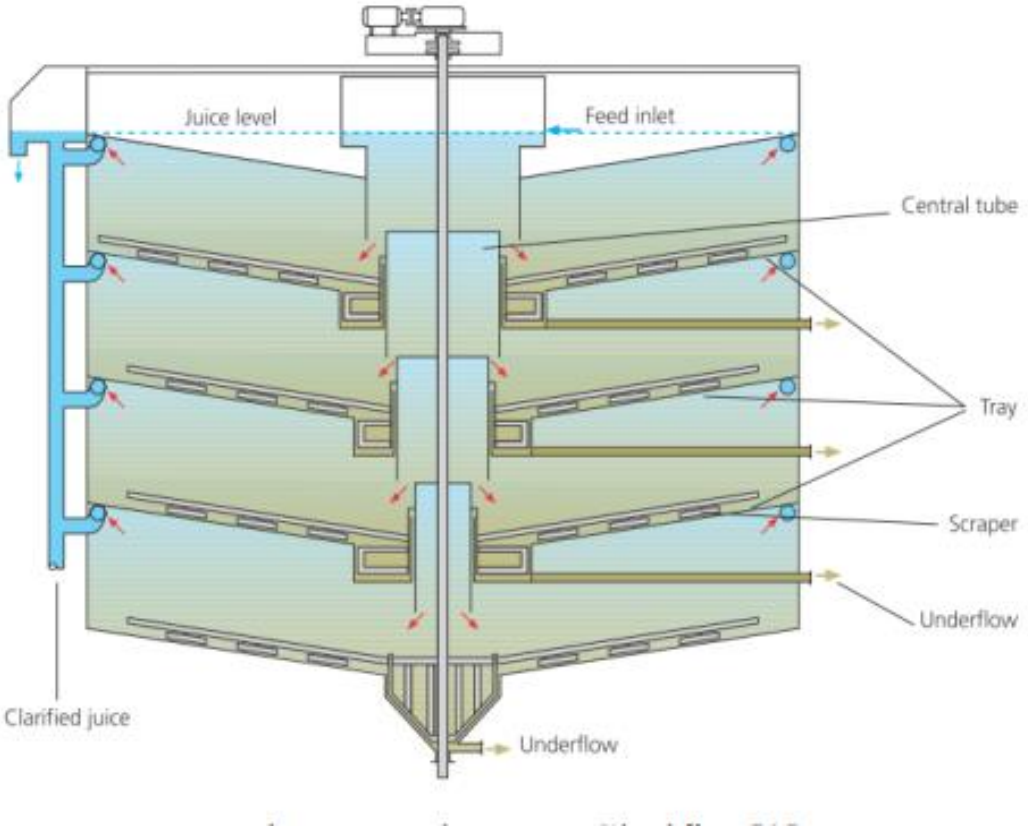


Figure 2. 4: The Dorr Clarifier

2.14.1. Short retention time clarifier

The short retention time clarifier or rapid clarifier is a single tray settler characterized by a short retention time that usually ranges between 20 to 45 minutes. The first generation of these type clarifiers were developed in the seventies by the Sugar Research Institute (SRI) and it has become one of the most popular clarifiers in the industry (P. Rein, 2007). Similarly, the Audubon Sugar Institute (ASI) recently developed a clarifier with Turbulence Reduction Devices, which has shown a better performance, lower residence times and less cost of implementation compared to other clarifiers (V. Kochergin and C. Gaudet,, 2011).

Table 2. 7: Standard parameters of clarified juice

s/n	Parameters	Units	Standard
1	Brix	%	13 - 14.5
2	Purity	%	≥ 86
3	pH	pH	7.0 – 7.2
4	Reducing sugar	%	< 0.5
5	Calcium Oxide	Ppm	< 900
6	Color	ICUMSA	5000 – 10,000
7	Turbidity	NTU	700 - 1500

*** *ICUMSA* –** International Commission of Uniform Method of Sugar Analysis**. *ppm-** parts per million

2.15. Filtration

Flocculated mud settles in the clarification process in the lower and the underflow is removed at a steady rate. The mud has then to be filtered to recover the juice from the soil and mud solids, mostly with the addition of fine bagasse particles, known as bagacillo, as a filtration aid. Filtrated is returned to the juice heating system. The operation of the filter stage is complex, requiring the interaction of good equipment control and conditioning of filter feeders. The combined interaction of wash water addition, milk of lime addition, filtrate recycles, filter and filter flocculant addition, maintaining the optimum fiber (bagacillo) ratio, filter head setting, boot level, drum speeds and agitator speeds, as well as attention to the training of operators, has to be

tuned for optimum results with regards to pol loss and filter operations. The settled flocculated mud impurities are extracted from the clarifier to recover trapped sucrose.

2.16. Juice turbidity

Turbidity is the measurement of scattered light that results from the interaction of incident light with particulate material in a fluid sample. It is an expression of the optical properties of a sample that causes light rays to be scattered and absorbed and rather than transmitted in a straight line through the sample. Turbidity of juice is often caused by the presence of suspended and dissolved solids. Particulate material is typically undesirable in the clarified juice disturbances, poor pH control, and poor frequent start-up and shutdown incidents were major process juice from a health perspective and its removal is often required when the final sugar is intended for consumption. Both process variables and cane quality were causes of high juice turbidities. In particular, flow variables contributors to poor turbidities. Poor cane quality consistently resulted in higher juice turbidity. As reported by (Doherty et. al, 2002) lower turbidity was achieved with lime saccharide rather than milk of lime.

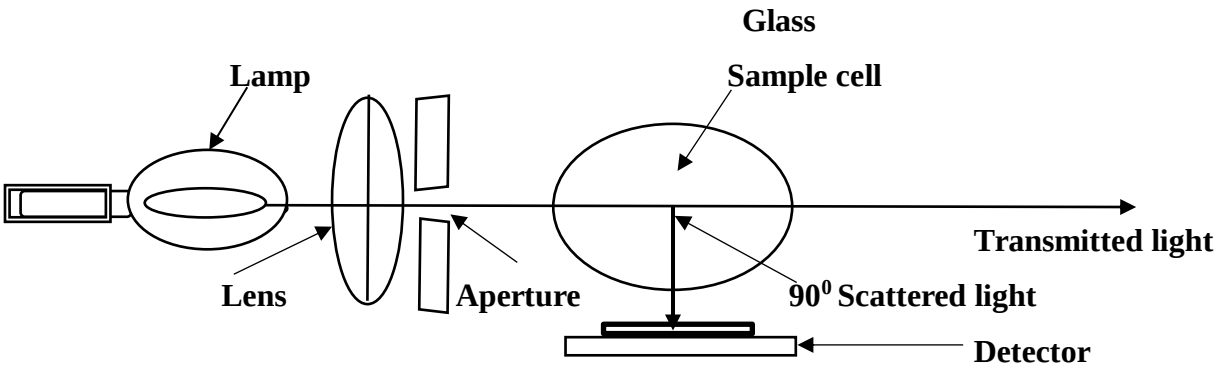


Figure 2. 5: Online Turbidity Meter

2.17. The juice colour

Colour in sugar process streams consist of a complex mixture of compounds. They are intro-duced naturally from the cane plant produced during processing in the factory. The compounds formed have different molecular weights, chemical structures and properties because of degradation and polymerization reactions caused by changes in process parameters such as pH

and temperature. The colorants that are difficult to remove are mainly hydrophobic in nature and persist throughout the sugar manufacturing process including within the sugar crystals. Moreover, their behavior and reactivity at various stages of the sugar manufacturing process is extremely complex. Therefore, it is important to understand the process parameter that contributes to the formation of color in order to develop technologies suitable for the subsequent removal of color during processing**.**

2.18. Evaporation plant

Evaporation is a key unit operation in a sugar mill and is the principal factor that determines its energy efficiency. It is the major user of steam in concentrating the clarified juice to a dissolved solids content of about 65 to 68 Bx^{0}' the way the evaporator station is configured determines the amount of steam that the factory requires and so the arrangement of the evaporators is a most important consideration. Multiple effect evaporation enables the steam requirement to be reduced and the bulk of evaporation of water is done by this means. The limit on syrup concentration is the approach to saturation at which point starts. In theory, this would limit the RDS to about 72 but in practice a safety margin of at least two is chosen, to allow some leeway in control and to allow for some cooling syrup in storage without crystallization occurring.

2.18.1. Juice evaporation under vacuum in the multiple effect evaporators

Across a multiple-effect evaporators, the operating pressure and correspondingly the boiling point of juice decreases from the first to the last effect. Evaporation of juice under such condition presents the following main advantages: It increases the driving force for heat transfer, the total temperature difference between the steam and the juice, which in turn increases the rate of heat transfer thereby increasing the evaporation rate.

- Low temperature operations generally require less energy (heat) than high temperature operations, i.e., it saves energy.
- Low temperature operations are less dangerous as the juice becomes more and more concentrated and viscous from the point of view of:

✓ Inversion is favored at high temperatures.

✓ Caramalization (Coloration)**:-** It is the charring of sugar manifested at high temperature

operations.

Such low temperature operation increases the evaporation capacity of an evaporator. Evaporation in the multiple - effect evaporators is not only due to the heat supplied to the heating surface of the Calandria but also occurs as a result of the flash that takes place when the juice passes from one body into another where lower pressure exists, as a consequence of progressively increasing vacuum and decreasing temperature in the evaporator set.

2.18.2. Sucrose losses in evaporators

Serious losses of sucrose can occur at the evaporator station because of high temperatures. The highest temperature occurs in the first effect of the evaporator. Because inversion and other degradation reactions are temperature dependent, this is important; the dependence on temperature is exponential and so average and localized temperatures are of great importance. Here it is important to point out that the rate of inversion doubles for every 6.3 rise in temperature. From an inversion point of view, is important also to keep the pH up, above 7 and preferably above 7.4 entering the first effect. However, the effect of temperature is far more significant. For this reason, it is desirable to keep the profile through the evaporation as low as possible (Rein P, 1986).

Table 2. 8: Standard pressure, temp., and brix of quadruple effect evaporators

Effect No.	Vapor Pressure in kg/cm^2 (abs)	Vapor Temperature ^oC	Brix
Pre boiler/ Vapor cell	1.0 -1.4	108 –111	19.8
1^{st} effect	1.4	108 – 111	26.6
2^{nd} effect	0.86	94 – 98	34.7
3^{rd} effect	0.55	82-85	49.7
4^{th} effect	0.158	54 – 60	65 – 68

2.18.3. Inversion

It is the transformation of sucrose (non- reducing sugar) into reducing sugars, glucose and fructose. These inverted sugars are "lost" in the final molasses, as they never crystallize under any circumstances in the vacuum pans and crystallizers.

Inversion is mainly a function of two factors: pH and Temperature**.**

- As temperature increases, the rate of inversion increases and Vice versa. It is pronounced at temperatures above 100 ºC.
- The lower the pH (or the higher the acidity) of juice, the higher the rate of inversion and vice versa. At pH lower than 6.5, inversion becomes more and more significant.

2.18.4. Scaling of evaporators calanderia tubes

Scales (incrustations) reduce heat transmission from the heating medium (steam/Vapors) to the juice thereby decreasing the evaporation rate and consequently the crushing rate of the factory. In operation, the tubes of a multiple effect becomes fouled in two ways: First scale is formed on the outside, a deposit of oil carried by the steam is formed and inside, scales derived from the juice are deposited. The exterior deposit of oil on Calandria tubes hardly exists except in the case of factories, which still use reciprocating steam engines, particularly if these were lubricated by atomization of oil at the steam entry. Scales inside the tubes form the most troublesome deposit. The scales formed inside the Calandria tubes originate from, Materials in suspension in juice poorly separated by clarification. These materials deposit mainly in the first vessel (vapor cell). Non- sugars in solution, which become insoluble as the juice becomes concentrated.

Table 2. 9: The composition of scales formed inside the calanderia tubes

Salts	Degree of Scale
Calcium Salts	Phosphate, Sulphate, Oxalate, and Carbonate
Metallic Oxides	Oxides of Magnesium, Aluminum and Iron
Silica	This forms the greater part of the deposit in the last vessel
Sulphites	Are common in factories using sulphitation process

2.19. 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). 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 excellent 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. 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 to constructing approximation models based on physical experimented observations (Boxetal., Montogomery). The main advantage of RSM is the reduced number of experimental runs needed to provide sufficient information for statistical acceptable results (Montogomery, 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 the response.

2.20. Suggested Solution for the Problems

The primary objective of effective juice clarification is the removal of maximum quantity of non-sugars that have harmful effect on recovery of commercial sugar at the earliest possible stage. The main objectives of cane juice clarification are:

- To separate soluble and insoluble matter that can precipitate.
- To reduce colour & turbidity of the juice
- To produce clear juice of correct pH (7.0 ± 0.1)
- To kill or inactivate microorganisms in the juice by heat treatment.

The importance of effective clarification are:

- Sucrose recovery is raised as the loss through final molasses is reduced
- Hardness of the clarified juice is minimized as the fouling effect of evaporators and heat exchangers are minimized
- The quality of the final product (sugar) is improved
- Operational consistency in the boiling house will be favorable with higher capacity.
- In juice clarification process, the elimination of non-sugars has to be done in such a way that.
- Neither sucrose nor reducing sugars were destroyed in considerable quantity.

CHAPTER THREE

3. MATERIALS AND METHODS

The methodology that would be followed to evaluate the performance of juice clarification plant was going to be conducted by primary and secondary data collection methods. Primary data collection means performing a laboratory work and then analyzing and discussing the results we obtained. When it comes to secondary methods, it includes literature survey, internet serf, data and information gathering, industry survey, peer discussion and interview.

3.1. Raw materials

Raw material collection and preparation of sample for making the experiment were clear juice from raw juice receiving tank after the removal of Dorr clarifier of (MSF) and the prepared milk lime from lime preparation plant were taken. Then the brix of lime were measured by lime Baume, refractometer, polarometer saccharometer, and spectrophotometer. After the sample was collected and packed in a polyethylene bag, transportation was carried out by bus from the factory to research station laboratory because of in order to prevent the juice from inversion and inversion is high temperature dependent, finally the sample was stored at room temperature in a research laboratory until further processing at lab scale.

The chemicals required for this research were neutral lead acetate, basic lead acetate, potassium oxalate, Methyl blue indicator, Mercuric chloride, Acid washed kieseguhr, mercuric iodide (HgI_2), Fehling's solution "A", Fehling's solutions "B, Ethylene diamine tetra acetic acid (EDTA), Ammonium buffer solution, oxalic acid, iodine solution, concentrated hydrochloric acid (HCl) and concentrated sulfuric acid (H_2SO_4) are used as laboratory reagents.

3.2. Methods

3.2.1. Study variables

Study variables were variables that studied during the research work; thus, variables are dependent or independent variables. The variables were the effect of pH, settling time, reducing sugar, Pol (sucrose) % and Brix %, colour, turbidity, calcium and magnesium oxide content. The

first two are factors or input (independent variables) and the rest were output, which is called response variables. Variation or change on input variables was affected the output variables that mean a change in pH would affect mainly some of the output variables RS, Pol %, Brix %, calcium oxide and magnesium oxide of clear juice, on the other settling time is mainly affecting the Brix %, Pol % (i.e. Purity), colour and turbidity of the clear juice.

3.2.2. Laboratory experimental setup

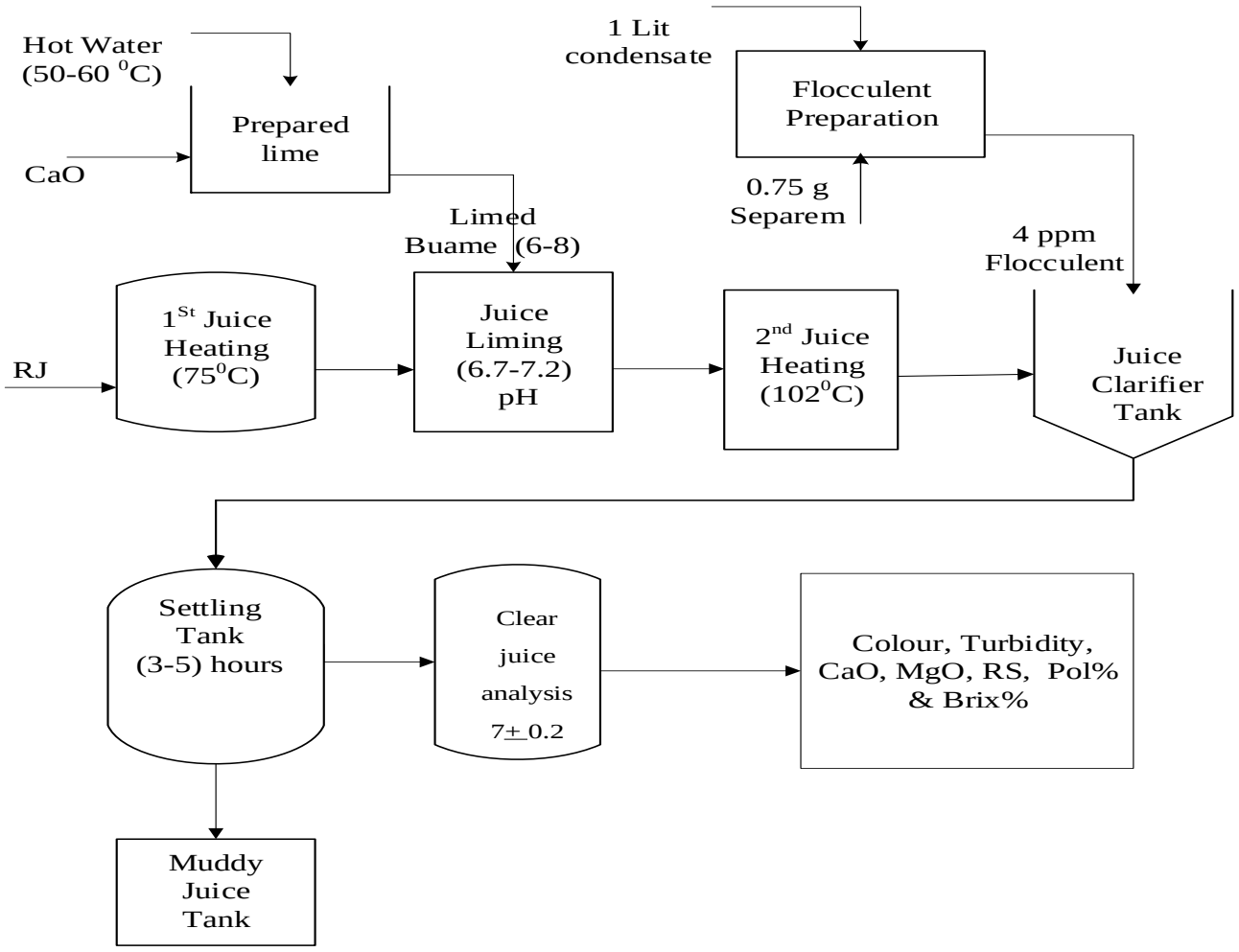


Figure 3. 1: Lab scale juice clarification process

Key Terms: CaO - Calcium Oxide, **MgO** - Magnesium Oxide, **RJ**-Raw Juice, **RS** - Reducing Sugars

3.2.3. The study design of quality parameters

Two portion of study had been designed on this study, first the study the existing process parameters like pol, Brix and pH of mixed, primary juice heating, secondary juice heating, sulphited juice and clear juice analysis. During the study the quantitative analysis of clear juice process parameters was identified, this was done by analyzing mixed and clear juice samples on each clarifications sections. Secondly, after identifying the most loss areas study the effect of hardness of clear juice for the formation of evaporators scale and optimization of process parameters were done. The study was done by varying the application of lime of proper controlling of pH and settling time on the raw juice for the output of colour, turbidity reducing sugars, pol%, Brix% and calcium magnesium oxide content of clarified juice.

3.2.4. Sample receptacles

Plastic, stainless steel or copper receptacles were used. They were cylindrical in shape to facilitate handling and cleaning and of sufficient capacity to hold enough samples for the desired sampled period. The containers were cleaned, sterilized by means of a steam jet and dried before use. At least two sets of containers were available so that they were drained completely after washed. Their lids tightly fitted to prevent evaporation of the samples. Permanent labels (marking) were painted on the containers for easy identification (Kassa H, 2010).

3.2.5. Design of sample taking devices

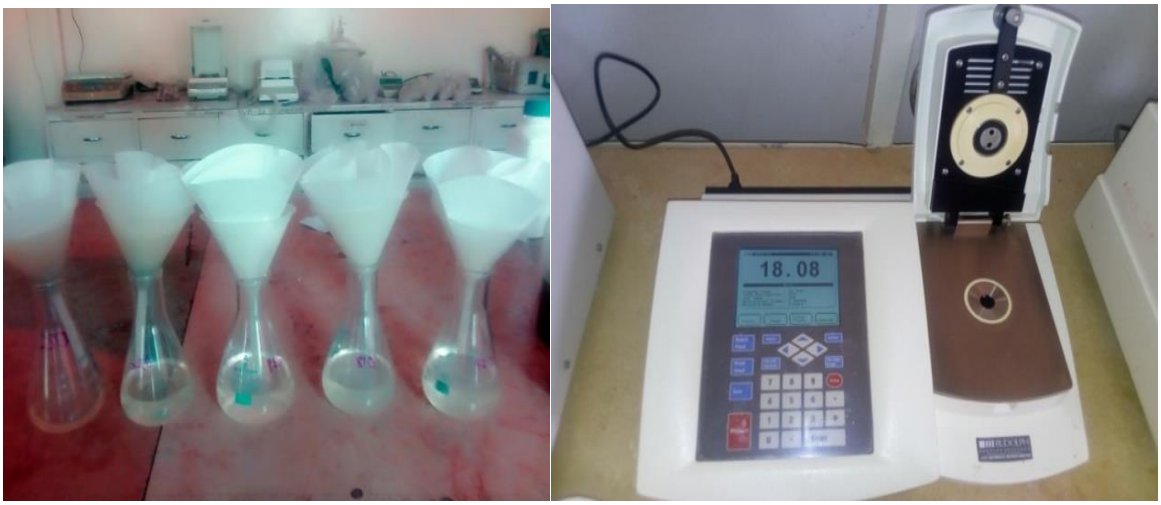
Sampling devices were designed according (Kassa.H, 2010) methods. The sample taken was representative and proportional to the total quantity of the material processed during the sampling interval. The sampling devices were self-cleanly as possible and can be cleaned easily; they were not subjected to mechanical fail as well. It also protected from evaporation or moisture absorption. In the process of juice, sampling the container was rinsed with a portion of the samples before filling up. All receiving vessels and carries were well fitted lids and preservative were added to the bucket at the rate of 0.5 ml mercuric chloride per liter of the expected final volume, at the state of each sampled period. At the end of every sampled period, the sampling apparatus and containers were conscientiously cleaned and dried. The samples were thoroughly

mixed before transferring to another container, i.e., when transferred to carrier vessels or after each hour replaces the sample packet with a cleaned one, and conveyed the sample to the laboratory. Then the samples were again mixed in the laboratory before screened into smaller vessels.

3.3. Determination of factory products

3.3.1. Determination of Brix

Method: - The unit of Brix, which has been in common use in the sugar industry for many years, is intended to represent the dry substance content % m/m. The convenience in the use of Brix hydrometer has lost favor in sugar industries due to the rough approximate nature of the results obtained especially in low purity sugar products. Today sugar industries of the world have replaced the hydrometer measurement by the refractometric method in which the result of the latter is closer to the true dry substance. However, the refractometric dry substance (RDS %) is still higher than the true dry substance obtained by official method oven drying. However, the term Brix continues to be used in parts of the sugar industry, even though ICUMSA recommended that it use to be discontinued. The method of determination of refractometric dry substance in sugar products were accepted by ICUMSA.



(a) Clarified juice analyses (b) refractometer instrument

Figure 3. 2: Clarified juice and refractometer instrument

3.3.2. Determination of Pol

Method: - The Polarimetry method of determination of Pol in sugar products is the officially accepted method in the sugar work throughout the sugar world. The method was based on the New International sugar scale adopted by ICUMSA (International Commission for Uniform Methods of Sugar Analysis). In 1986, that is 100 ^{0}Z points is the optical rotation of the normal weight of sucrose (26 g weighed in air with brass weight), dissolved in 100 ml, polarized at the wavelength of the green line of the mercury isotope ^{198}Hg, wavelength = 546.2271 nm at 20 ^{0}C, in a tube length of 200 mm. For sugar products, which need clarification, the Horne's dry lead method of clarificationof the sample is preferable which is used almost exclusively in all modern sugar industries. In the Horn's method of clarification of the sample, the presence of insoluble matter in the juice is of no consequence. The Schmitz's method demands the establishment of correct volume on two occasions, 100 and 110 ml and evolves and error due to the volume of lead precipitate. In all cases, care should be taken to avoid the use of excessive lead acetate for clarification. The danger of over leading is greater in dry lead than in wet lead, particularly if the dry lead is not finely divided and readily soluble. In lead clarification, there always some change of volume error and some combination with any laevulose present. However, still lead acetate is accepted as the best clarifying agent for general laboratory purpose.

Table 3. 1: Specific rotation of the main sugars

S/N	Description	Angle of rotation
1	Sucrose	+ 66.54 ^{0}arc
2	Dextrose	+ 52.50 ^{0}arc
3	Laevulose	- 92.50 ^{0}arc
4	Invert	- 20 ^{0}arc

**(+) degree rotation in clockwise direction. *(-) degree rotation in anti-clockwise direction*

The value of pol obtained by direct polarization is the resultant of optical rotation of all optically active substances present in the solution. It can indicate the true measure of sucrose only when no other optically active substance present in the solution rather than sucrose. For any substance of known specific rotation, the concentration of that substance in a solution may be determined

by observing the rotation of polarized light as the solution is tested in a polaroscopic tube of known length with the correct combination of temperature and light. This is the basic principle of calibration of the modern polaroscope known as Sacchrometer. A saccharometer is a polarometer graduated not in angular degree, but with an evenly divided scale calibrated to read from -100^0 to $+100^0$Z.

$$\text{Pol\%} = \text{Pol Reading} \times \text{Pol Factor} \quad \textbf{(3.1)}$$

(a) Clarified juice (b) Polarometeric instrument

Figure 3. 3: Clarified juice and Polarometer instrument

3.3.3. Determination of juice purity

After determination of Pol % and Brix % of the juice sample, the purity of juice sample was Calculated using the formula:

$$\text{Purity} = \frac{Pol}{Brix} \times 100\% \quad (3.2)$$

3.3.4. Determination Reducing Sugars

Procedure: The reducing sugar content of juices is determined using the method of Eynon and Lane in which a sample of juice containing about 0.15 to 0.3g RS/100 cm^3 is titrated against the Fehling's solution. It has been found that calcium interferes with the determination and it is recommended that EDTA be used as sequestrate. Following the method of (ICUMSA, 2007)), neutral lead acetate, Fehling's solution 'A' and Fehling's solution 'B' were prepared and standardized after which analysis of reducing sugars was carried using the solutions as follows:
The sample of juice was filtered for analysis through the piece of gauze to remove any solid particles, which might block the tip of the pipette or burette. About 50g of the screened sample was added into a clean volumetric flask, together with 10ml of EDTA (4%) and made to the mark with distilled water. The 50ml burette was rinsed with the diluted juice before filling and adjusting to zero. Then, 5ml Fehling's A solution and 5ml Fehling's B solution were pipped into the boiling flask and 15ml diluted juice from the burette was added and the flask was placed an on a fast hot plate for boiling in not more than 2.25 min. Three or Four drops of methylene blue were added and the addition of solution from the burette was continued until the indicator is completely decolorized. The boiling liquid changes to the bright orange appearance, which it had before the addition of the indicator.

3.3.5. Determination of Colour

Sugar colorants are generally classified in two groups: colors that occur naturally e.g. plant pigments and colour that are formed during the process e.g. caramels, melanoidins. These components can be removed using different processes like affinition, carbonation or application of ion exchange resins, which are usually carried out in a sugar refinery. In the case of juice clarification, this operation is not intended to remove colour. However, some colour reduction can be expected and the parameter can serve as an additional source of information for the complete assessment of the clarifier's performance. Therefore, we measured the colour of the filtrate before and after clarification using the ICUMSA methods. The method consists in taking an aliquot of juice, approximately 5 grams and mixing it with approximately 95 grams of water. Then the solution was adjusted to pH 7.0 ± 0.1 by adding a solution of sodium hydroxide or

hydrochloric acid. This step is very important because colorants are sensible to pH changes. Then, the sample is passed through a 0.45 µm filter to remove any trace of turbidity that might affect the reading, especially at the wavelength used in the 52 method (420 nm) that accounts for colour and turbidity. Then, the refractometric dry substance (RDS) of the solution is measured and the sample was analyzed using a spectrophotometer adjusted to a wavelength of 420 nm.

$$\text{ICUMSA}_{420}\ \text{Color Unit} = \frac{A}{B \times C} \times 1000 \qquad (3.3)$$

Where: A- Absorbance

B - Cell Length (cm)

C - Concentration of total solids (g/ml)

(a) Spectrophotometer (b) Clarified juice after filtration

Figure 3. 4: Spectrophotometer and clarified juice

3.3.6. Determination of turbidity

The turbidity of clarified juice is a measure of the effectiveness of the clarification process. The turbidity of the clear juice was determined according to the method 3.5 of (SASTA, 2005). The ICUMSA colors of unfiltered and filtered samples are measured at 420nm and the difference between the readings is the turbidity of unfiltered juice (MSIRI, 1991).

The Brix of clarified juice was initially measured and the conversion of the original Brix in to 2^o Brix was carried out. After conversion, the required gram of clear juice was weighed and filled with distilled water until the weight became hundred grams. This sample divided into two equal portions and the first portion was subjected to filtering through a Buchner funnel with What man NO.5 filter paper using kieselguhr filter aid. Then absorbency was measured at 420nm, by filtering the sample solution with kieslguhr (1% on solids) through filter paper, and the pH was adjusted to 7 ± 0.2 with diluted hydrochloric acid or sodium hydroxide, before absorbency reading, the effect of absorption of light scattering by particles contributing to turbidity is eliminated. So the difference between absorbency readings before (A_1) and after (A_2) filtration $A_1 - A_2$ is the turbidity of the juice (Kassa, 2010).

$$\text{Turbidity of clarified juice} = C2 - C1 = \frac{(A2-A1)}{0.0201} x1000 \quad (3.4)$$

Where: A_2 = absorbency before filtration /non filtered juice, A_1 = absorbency after filtration/filtered juice

3.3.7. Determination of Calcium oxide content

Method: The concentration of calcium and magnesium in mixed juice, clear juice and syrup can determined approximately by complexometric titration against EDTA solution.For exact determination, precipitation with oxalate and titration with potassium permanganate is recommended. However, the titration with EDTA gives accurate enough results for actory control (ICUMSA GS7, 1994).

Principle: - EDTA-ethyldiamine tetra acetic acid has got the property of strong complexity, ability of metal ions forming stable soluble internal complex salts; each molecule of EDTA forms one metal ion irrespective of its valence. When sugar solutions are titrated with EDTA

calcium and magnesium together are determined using a complex of heavy metals and the dye Erichrom black T as an indicator of magnesium at pH = 10. Calcium is precipitated out of the solution by potassium oxalate. The subsequent titration against EDTA gives a magnesium concentration of the solution. The pH of the solution during EDTA titration is important and limits of ±0.05 units for successful determination. To achieve such narrow limits of pH control, buffer solutions are frequently used.

For determination of MgO concentration, 100ml of the juice sample was pipetted into a 200ml volumetric flask, with the addition of 25ml of 5% potassium oxalate solution, and made up to mark with distilled water. The solution was mixed thoroughly and allowed to stand for 1 h to ensure complete precipitation of the calcium oxalate 10ml of the two solutions were pipetted into a 250ml Erlenmeyer flask and diluted to about 50ml with distilled water. Then, 2ml ammonia buffer solution, 8-drops diethyl–dithiol- carbonate solution and 8 drops of Eriochrome black T indicator were added. The solution was then stirred with a magnetic stirrer and titrated against standard EDTA solution until the pink colour of the solution first changes to light green.

1ml standard EDTA 0.574mgCaO = 0.413MgO

If titre for CaO + MgO determination = a ml

Titre for Magnesium Oxide determination = b ml.

Hence titre for Calcium Oxide determination = (a-b) ml

If a liquid of diluted juice used for titration = C ml

$$\text{CaO in 1 litter juice} = \frac{0.574(a-b)\times 1000\times \text{dilution factor}(2)}{C} \quad (3.5)$$

$$\text{MgO in1 litter juice} = \frac{0.413(b)\times 1000\times \text{Dilution factor}(2)}{C} \quad (3.6)$$

Where: a = EDTA is taken for titration in the form calcium and magnesium oxide,

b = EDTA is taken for titration of magnesium oxide

C = Diluted juice used for titration.

3.4. Determination of pH

Following the manufacturer's directions, calibrate the pH meter using the 4.00 and 7.00 pH buffer solutions (compensated for a temperature different from 20°C) while stirring at a constant rate. Calibrations should done at the beginning of each day or shift using fresh buffer solutions only. The buffer solutions should be at room temperature. The pH value determines whether the clarified juice is hard or soft. Measurement of pH is to determine the corrosiveness of the clear juice is there any impact on the multiple effect evaporator tubes or not. The measurement, pH meter is standardized with the buffer solution within the specified range of scale. If the addition of lime in the juice increases, the amount of pH recorded becomes higher, pH meter with glass electrode was used for pH measurement. Therefore, the clear juice samples was taken, measures, and put in in the measuring beaker.

3.5 Statistical analysis

The data were analyzed and modeled using the design expert software 7.0.0. Second degree polynomial models were generated. The significant terms in the models were identified by analysis of variance (ANOVA) for each response. Significance was judged if the probability level of the F-statistic calculated from the data was less than 0.05. The model adequacy was checked by R^2, adj R^2, pred R^2 and, Lack of Fit test. The tables of summary of fit, ANOVA, lack of fit and parameter estimates, which were generated by the software, were presented in the discussion part. Contour, interaction and 3D plots were developed to search the optimum conditions for the three main responses namely, the clear juice calcium oxide, the turbidities and colour.

CHAPTER FOUR

4. RESULTS AND DISCUSION

4.1. Laboratory result analysis

The analysis methods that have been used in this study for each sample have been listed in the table below. The determination steps of each parameter referred to the Ethiopian Sugar Factory, laboratory manual for sugar production by (Kassa H, 2010).A digital refractometer and polarometer were used to measure the Brix and pol content of the juice samples respectively from the clear juice-receiving tank. The juices were analyzed as soon as possible or else preservation was added and kept into the refrigerator to minimize inversion or degradation of sucrose by the action of microorganisms, as the time goes reproduction of microorganisms on sucrose solution is very high. The most important parameters that have done in clear juice laboratory analysis results were; Brix %, pol %, colour, turbidity, calcium oxide, magnesium oxide and reducing sugars for each sample.

Table 4. 1: Laboratory analytical result for mixed, primary juice heating and sulphited juice

Mixed juice					Primary juice heating				Sulphited juice			
s/n	Brix %	Pol**%**	Pty%	pH	Brix%	Pol	Pty%	pH	Brix%	Pol%	Pty%	pH
1	19.3	16.46	85.31	5.4	19.2	16.3	84.8	5.8	18.52	15.9	85.8	6.9
2	19.73	16.99	86.12	5.3	19.31	16.5	85.7	5.5	14.05	12.4	87.97	7.2
3	18.85	16.33	86.62	5.4	19.41	16.5	85.21	6.3	19.21	16.5	85.89	6.9
4	13.73	11.6	84.27	5.3	19.35	16.5	85.02	5.6	19.03	16.3	85.65	7.5
5	19.24	16.64	86.64	5.5	13.94	11.9	85.4	5.8	13.79	11.7	84.83	7.8
6	18.91	16.3	86.86	5.2	13.56	11.6	86	5.4	18.12	15.54	84.84	7.3
7	19.16	16.68	87.68	5.5	23.5	20.1	85.4	5.7	22.82	19.58	85.81	7.0
8	18.95	16.46	86.81	5.4	13.5	11.7	86.4	5.6	19.15	16.05	83.8	7.1
9	18.91	16.04	84.88	5.5	14.5	11.6	82	6.3	18.77	16.15	86.04	7.4
10	18.21	15.61	85.61	5.7	17	14.7	86.3	6.	11.57	9.74	84.18	7.4
11	19.15	16.33	85.27	5.6	13.45	11.3	84.4	6.9	15.5	13.09	84.52	6.5

***Pty**-Purity of mixed, primary juice heating and sulphiting juice.*

4.1.1. Result analysis of mixed, first heating and sulphited juice

Table, 4.1 shown above the mixed juice, primary juice heating and sulphited juice analytical parameters obtained during the crushing season of the research work were as pH, purity, and pol (sucrose) were good and data analyzed were in the range of standard operations (SOP). However, the mixed juice, primary juice heating, and sulphited juice brix is not the range of standard operations parameters. These may bring a great impact and difficulty to control in the evaporation plant and vacuum pan tubes during the processing of syrup in the boiling house because of the fast formation of sugar crystals at that stage. When crystals were formed in the evaporation plant, there are losses through vapor lines during the juice extractions system. This makes caramalization (colouration) it is the charring of sugar manifested at high temperature operations.

4.1.2. Result analysis of secondary juice heating, and clarified juice

Table 4. 2: Laboratory analytical result for second heating and clear juice

	Secondary juice heating				Clarified juice			
S/N	Brix%	Pol%	Pty%	pH	Brix%	Pol%	Pty%	pH
1	18.26	15.33	84.10	6.9	17.86	15.05	84.27	6.9
2	18.37	15.35	83.56	6.9	17.34	14.7	84.77	6.7
3	19.25	16.4	85.19	7.2	17.87	14.82	82.99	6.8
4	19.3	16.5	85.70	7.0	17.48	14.8	84.66	6.7
5	14.65	12.35	84.4	7.3	18.92	16.05	84.83	6.9
6	16.55	13.84	83.8	7.4	17.72	14.95	84.41	6.8
7	23.71	20.29	85.6	6.9	17.87	15.1	84.49	6.8
8	20.57	17.21	83.66	7.3	18.48	15.4	83.33	6.9
9	19.61	16.3	83.12	7.5	18.1	15.2	83.97	7.0
10	12.8	10.66	83.2	7.8	18.23	15.55	85.29	7.0
11	14.7	12.1	82.3	6.7	15.54	13.09	84.3	6.8

***Pty**-Purity of secondary juice heating and clarified juice*

Table 4.2 above show mixed, primary juice heating and sulphited juice, the secondary juice heating and clarified juice analytical parameters obtained during the crushing season of the thesis work were, pH, purity, and pol (sucrose)% were good or within the standard. However, the Brix of such juices was not the range of standard operations or somewhat difficult to accept. These may bring a great impact on controlling in the upcoming units like evaporation plant and vacuum pan during sugar boiling this may come the fast formation of sugar crystals. When crystals were formed in the evaporation plant, the formation of inversion may take place because inversion of sucrose due to the combined effect of pH and temperature. Across a multiple-effect evaporators, the operating pressure and correspondingly the boiling point of juice decreases from the first to the last effect.Evaporation of juice under such conditions presents low temperature operations generally require less energy (heat) than high temperature operations, i.e., it saves energy. Low-temperature operations are less dangerous as the juice becomes more and more concentrated and viscous from the point of inversion.

4.1.3 Lime quality

Table 4. 3: Analytical results of lime purity

S/N	Available CaO (%)
1	84.0
2	86.0
3	90.3
4	84.7
5	86.3
6	86.1
7	60.2
8	80.5
9	86.8
10	85.4

The lime quality is one of the performance indicators for white sugar production process. A good quality of lime should contain over 90-95 % CaCO3 and less than 2 % insoluble matter (E.Hugot, 1986).However, the factory has been still using below the recommended level of available lime which is not recommended for clarification. The allowable impurity of SiO_2, sulfate as SO3, iron oxide, and aluminum oxide in this lime is not significant that much because its composition of purity was far below the standard limits. However, the degree of only available calcium oxide has a great significance effect but still the level of calcium oxide has below the standard this would boast in the colour, turbidity and calcium oxide content clear juice hardness finally would result also the accumulation of scale in the tubes of evaporators

4.1.4. Laboratory analysis for magnesium, purity and reducing sugars

Table 4. 4: Clear juice analytical result of purity, magnesium oxide and reducing sugars

S/N	pH	ST (hour)	Brix %	Pol %	Purity%	MgO (ppm)	RS (%)
1	6.70	3.00	11.73	10.21	87.05	479	0.7
2	7.20	3.00	10.89	9.22	84.65	512	0.56
3	6.70	5.00	10.33	11.31	84.81	496	0.6
4	7.20	5.00	12.30	10.87	88.36	529	0.52
5	6.60	4.00	13.39	11.40	85.17	438	0.73
6	7.30	4.00	13.69	12.21	89.19	553	0.48
7	6.95	2.59	17.54	15.22	86.77	446	0.66
8	6.95	5.41	14.27	12.47	87.37	487	0.61
9	6.95	4.00	16.32	14.33	87.80	347	0.62
10	6.95	4.00	14.60	12.79	87.59	430	0.65
11	6.95	4.00	14.92	12.94	86.74	446	0.68
12	6.95	4.00	13.60	11.87	87.27	281	0.68
13	6.95	4.00	15.11	13.24	87.63	413	0.65

Reducing sugars are also not in the range of standard operations in Table 4.4. These shows there are inversions thermal treatment of reducing sugars in the presence of amino acids and denaturing of proteins (and other organics, such as gums and waxes). Proteins in aqueous phase cause colored compounds to appear in a few minutes and the darkening increases with retention time (Knerr et.al, 2001).

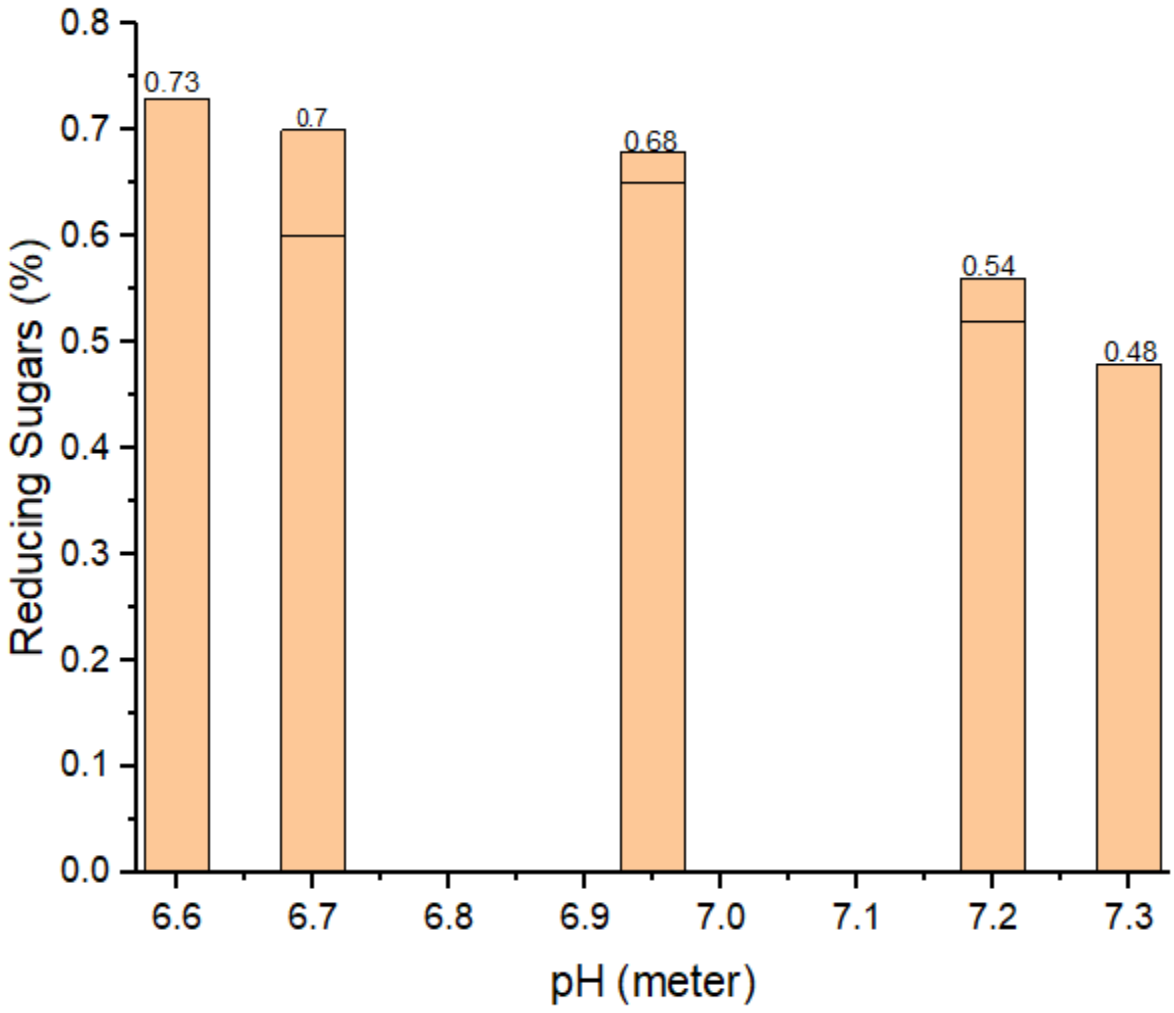


Figure 4. 1: Effect of PH on reducing sugars in clear juice

When the decrease in the amount pH of an acidic state of pH 6.6 in Table 4.4 above becomes increasing in the value of reducing sugars of 0.73 % and this becomes the formation of inversion also facilitates sucrose loss. While the higher amount of pH of 7.3 the minimum in the reducing sugars that were observed with a value of 0.48 % this becomes the relatively below the

recommended level and this amount of reducing sugars gives a significant amount of colour formation. Degradation reducing sugars to organic acids due to high pH and temperature and precipitation of organic and inorganic salts. Therefore the greater the amount of excessive liming leads to hinder crystal formation in the sugar crystallization so, to avoid such criteria optimum reducing sugars were required at optimum pH and settling time.

4.2. Design optimization of colour, turbidity and calcium oxide content

Response surface methodology of complete composite design was implemented to identify the optimum colour, turbidity, and calcium oxide content of clear juice. Two factors at two level response variables were employed; the factors were pH and the settling rate of the clear juice. The design of optimization was conducted on the central composite design method, the design of an experimental combination was held by inserting the lowest and highest values on the design expert software.

Table 4. 5: Central composited for optimization of response variables

Std	Run	Block	Factor 1 PH	Factor 2 (ST)	Response 1 Colour	Response 2 Turbidity	Response 3 CaO
1	5	Block 1	6.70	3.00	*	*	*
2	10	Block 1	7.20	3.00	*	*	*
3	6	Block 1	6.70	5.00	*	*	*
4	9	Block 1	7.20	5.00	*	*	*
5	7	Block 1	6.60	4.00	*	*	*
6	3	Block 1	7.30	4.00	*	*	*
7	11	Block 1	6.95	2.59	*	*	*
8	13	Block 1	6.95	5.41	*	*	*
9	2	Block 1	6.95	4.00	*	*	*
10	12	Block 1	6.95	4.00	*	*	*
11	4	Block 1	6.95	4.00	*	*	*
12	1	Block 1	6.95	4.00	*	*	*
13	8	Block 1	6.95	4.00	*	*	*

4.2.1. Design optimization for response variables of clear juice

The analysis result of all the three of the responses as such in Table 4.6. The samples were taken for each design combination and analyzed for responses of colour, turbidity and calcium oxide content of clear juice. The analysis was done as usual based on the handbook of laboratory methods and chemical control for Ethiopian sugar factories (Kassa H, 2010). The sampling system for each combination of the juice samples were sampled at the same time and analysis were done in parallel. The sampling system was by varying the application of lime and constant application of flocculent and measured the time taken for settling. For each thirteen combinations, the representative samples were taken, but the sampling period was different.

Table 4. 6: Clear juice analysis result of response variables

Std	Run	Block	Factor 1 PH	Factor 2 (ST)	Response 1 Colour	Response 2 Turbidity	Response and 3 CaO
1	5	Block 1	6.70	3.00	2268	2089	2388
2	10	Block 1	7.20	3.00	1625	1537	2387
3	6	Block 1	6.70	5.00	2506	2338	1963
4	9	Block 1	7.20	5.00	3213	2833	2833
5	7	Block 1	6.60	4.00	2328	1479	2396
6	3	Block 1	7.30	4.00	2642	2338	3237
7	11	Block 1	6.95	2.59	2300	2042	2452
8	13	Block 1	6.95	5.41	2938	3138	2273
9	2	Block 1	6.95	4.00	2054	1846	2235
10	12	Block 1	6.95	4.00	2063	1895	2502
11	4	Block 1	6.95	4.00	2641	2467	2640
12	1	Block 1	6.95	4.00	2336	2398	2599
13	8	Block 1	6.95	4.00	2683	2041	2629

4.3. ANOVA results for colour of clear juice

Linear versus 2F1 model was suggested by the design either programs to make this response but did not use both at the same time, so a linear model was taken to test for its adequacy to describe its variation with independent variables. From the ANOVA test in Table 4.7, shown below, the Model F-value of 7.11 implies the model is significant. There is only a 0.95% chance that a "Model F-value" this large could occur due to disturbance.

Values of "Prob > F" less than 0.0500 indicates model terms are significant. In this case B, AB 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 the model.

Table 4. 7: ANOVA test for colour of clear juice

Source	Sum of squares	DF	Mean-square	F-value	Prob > F	
Model	1.418E+006	3	4.728E+005	7.11	0.0095	Significant
A-pH	32266.01	1	32266.01	0.49	0.5037	
B-ST	9.304E+005	1	9.304E+005	13.99	0.0046	
AB	4.556E+005	1	4.556E+005	6.85	0.0279	
Residuals	5.986E+005	9	66505.70			
Lack of Fit	2.329E+005	5	46589.21	0.51	0.7612	Not Significant
Pure Error	3.656E+005	4	91401.30			
Cor Total	2.017E+006	12				

****ST***-Settling Time

Lack-of-Fit is the variation due to the model's inadequacy. The "Lack of Fit F-value" of 0.51 implies the lack of Fit is not significant relative to the pure error. There is a 76.12% chance that a "Lack of Fit F-value" this large could occur due to disturbance. Non-significant lack of fit is good because we want the model to fit. This non-significance lack of fit shows the proposed model fits with the experimental data and the independent variables or parameters have a desirable effect on the response. This conclusion was deduced by comparing the p-value of the individual and combined factors with the percentage of probability of the model (i.e. $\alpha = 0.05$).

The lesser the p-value than α-value has implied the larger significance effect of the factor on the response.

Table 4. 8: Checking of data and adequacy of model of colour

Std. Dev.	257.89	R-Squared	0.7037
Mean	2430.54	Adj R-Squared	0.6050
C.V.	10.61	Pred R-Squared	0.4392
PRESS	1.135E+006	Adeq Precision	9.501

The "Pred R-Squared" of 0.4392 is in reasonable agreement with the "Adj R-Squared of 0.6050, "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 9.501 indicates an adequate signal. This model can be used to navigate the design space (Montgomery, 2001). Coefficient of variation is a measure expressing the standard deviation as a percentage of the mean, smaller values of CV give better results. The PRESS (Predicted Error Sum of Squares) measure of how a particular model fits each point in the design.

4.3.1. Equation for optimization of colour of clear juice

Model equations were 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.

Final equation in terms of coded factors:

$$\text{Colour} = +2430.54 + 63.51 \times \text{A} + 341.03 \times \text{B} + 337.50 \times \text{A} \times \text{B} \quad (4.1)$$

Final equation in terms of actual factors:

$$\text{Colour} = +36830.88521 - 5145.96847 \times (\text{pH}) - 9041.46647 \times (\text{Settling Time}) + 1350.00000 \times (\text{pH}) \times (\text{Settling Time}) \quad (4.2)$$

The coefficient of determination of correlation is 0.7037, which is 70.37 %, it indicates that the degree of the fitting is good and the reliability of the trend is good. The model reaches a very significant level of the probability value ($p < 0.01$). In addition, the regression coefficient and the

regression intercept have reached a significant level ($p < 0.01$). Increasing the negative terms will decrease the responses and at the same time, decreasing the positive terms will increase the responses.

4.3.1.1. Normality of the data

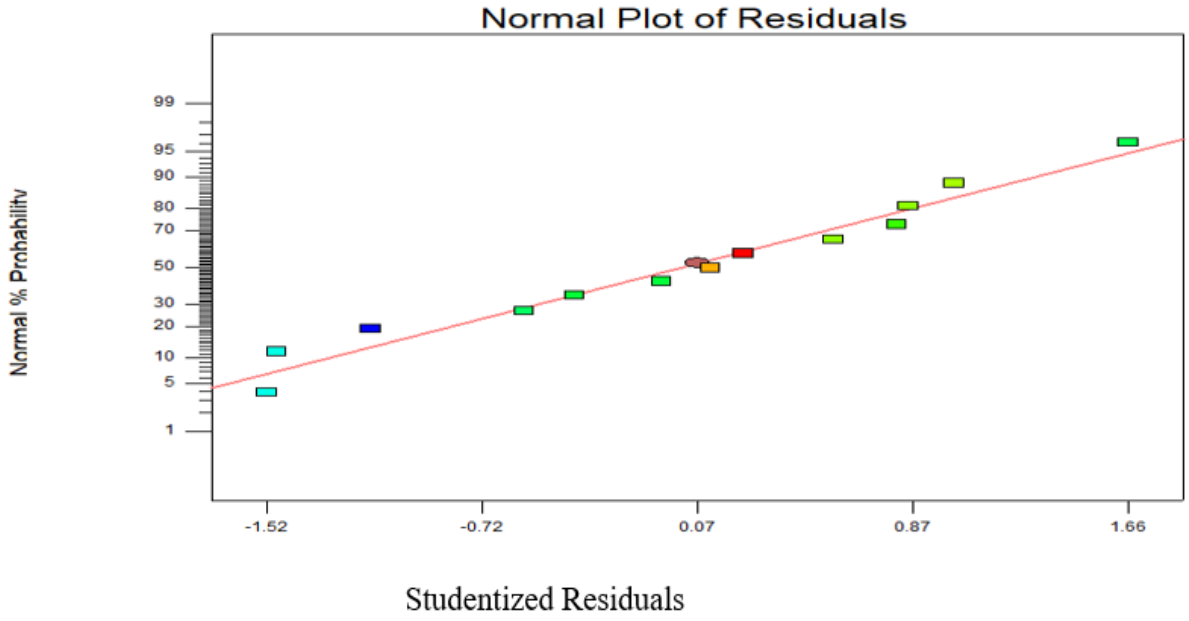


Figure 4. 2: Normal probability plot of colour

The normality of the data was done using the normal probability plot. The normal probability plot of the residuals for colour is shown in Figure: 4.2, above which reveals that the residuals are falling on the straight line. This means that the errors are distributed normally. Therefore, the normality assumption was satisfied as the residual plot approximated along a straight line.

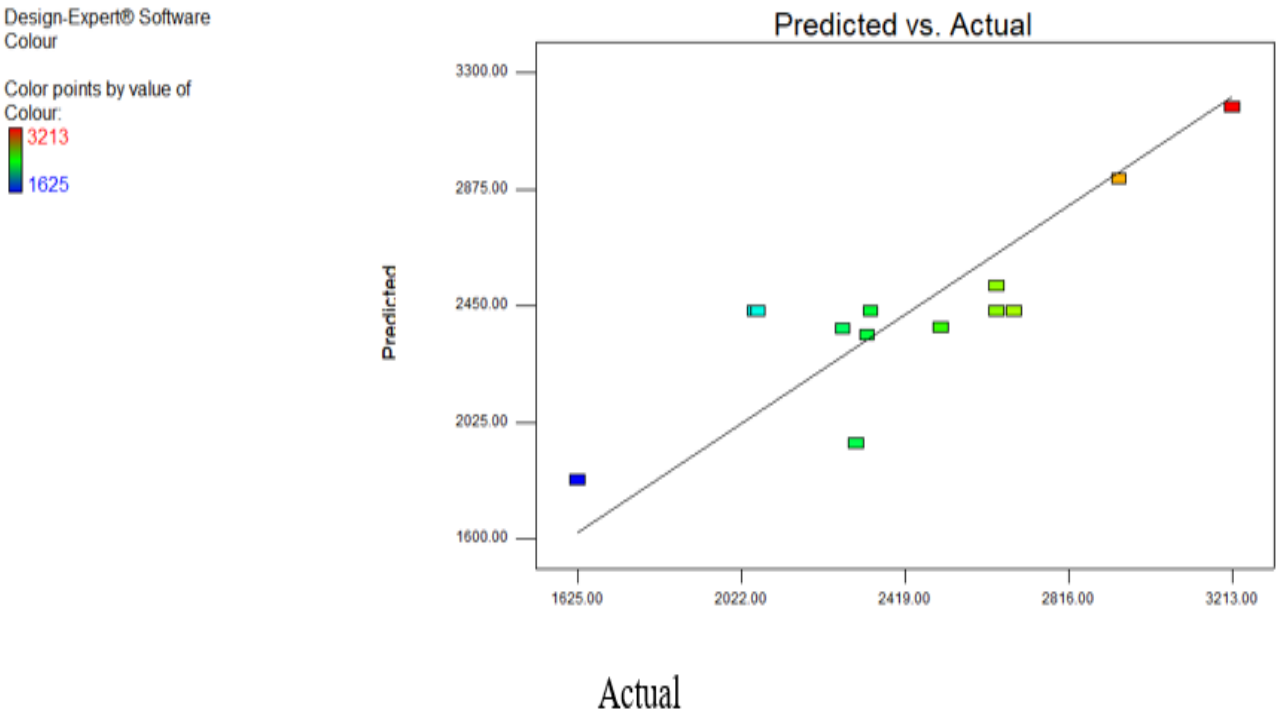


Figure 4. 3: Predicated versus actual plot

Figure: 4.3 indicates the relationship between the actual and predicted values of the colour. A residual is the difference between an observation and its estimated (or fitted) value from the statistical model being studied. This figure shows that the developed model is adequate. Because, the residuals in the prediction of each response are small, have a tendency to be close to the diagonal line.

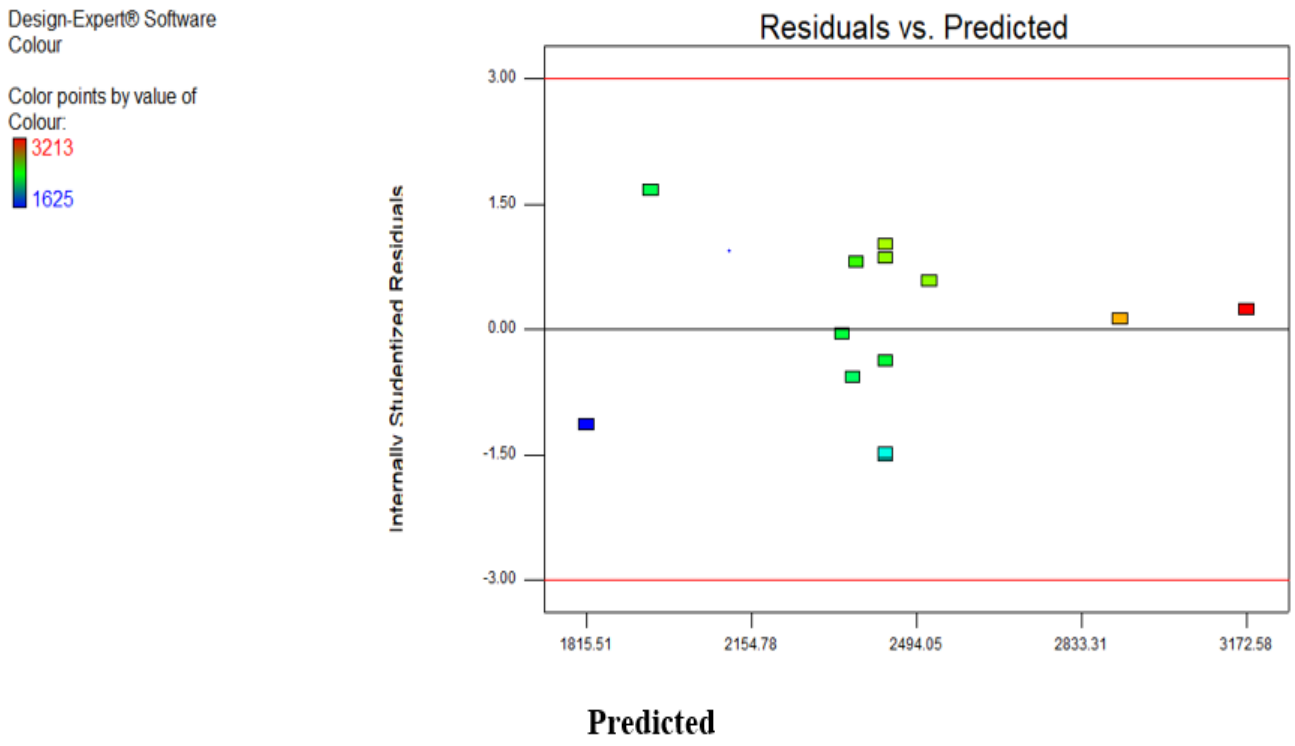


Figure 4. 4: Residual versus predicted plot of colour

Figure: 4.4 presents a plot of residuals versus the predicted response. The general impression is that the residuals distribute randomly on the display, telling that the variance of the original observation is constant for all values of colour. Figure: 4.2 and Figure: 4.4 are satisfactory, based on these two plots we conclude that the empirical model is adequate to describe the active ingredient of colour by response surface.

4.3.1.2. Independency of the data

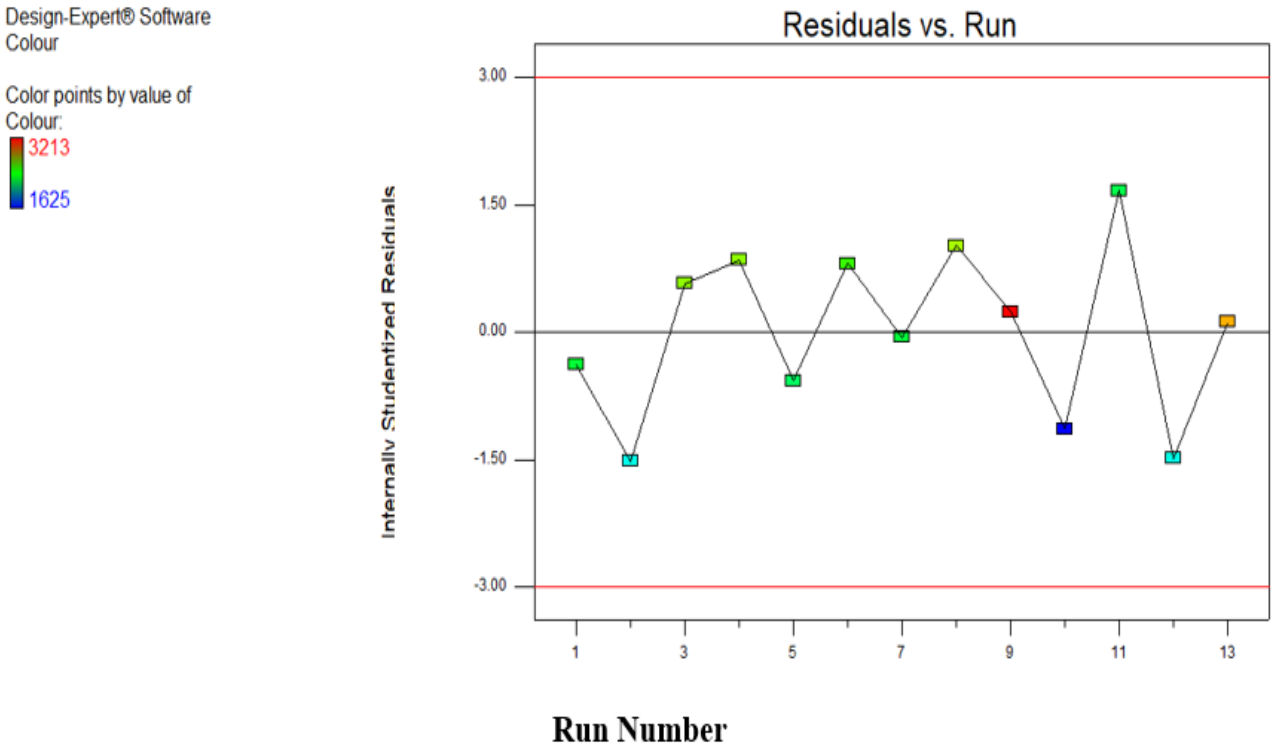


Figure 4. 5: Residual versus run plot of colour

The independency of the data was tested by plotting a graph between the residuals and the run order. The residual plot for colour is shown in Figure: 4.5 above which reveals that there was no predictable pattern observed because all the run residues lay on or between the levels of -3 to 3.

4.3.2. Interaction graph of colour of clear juice

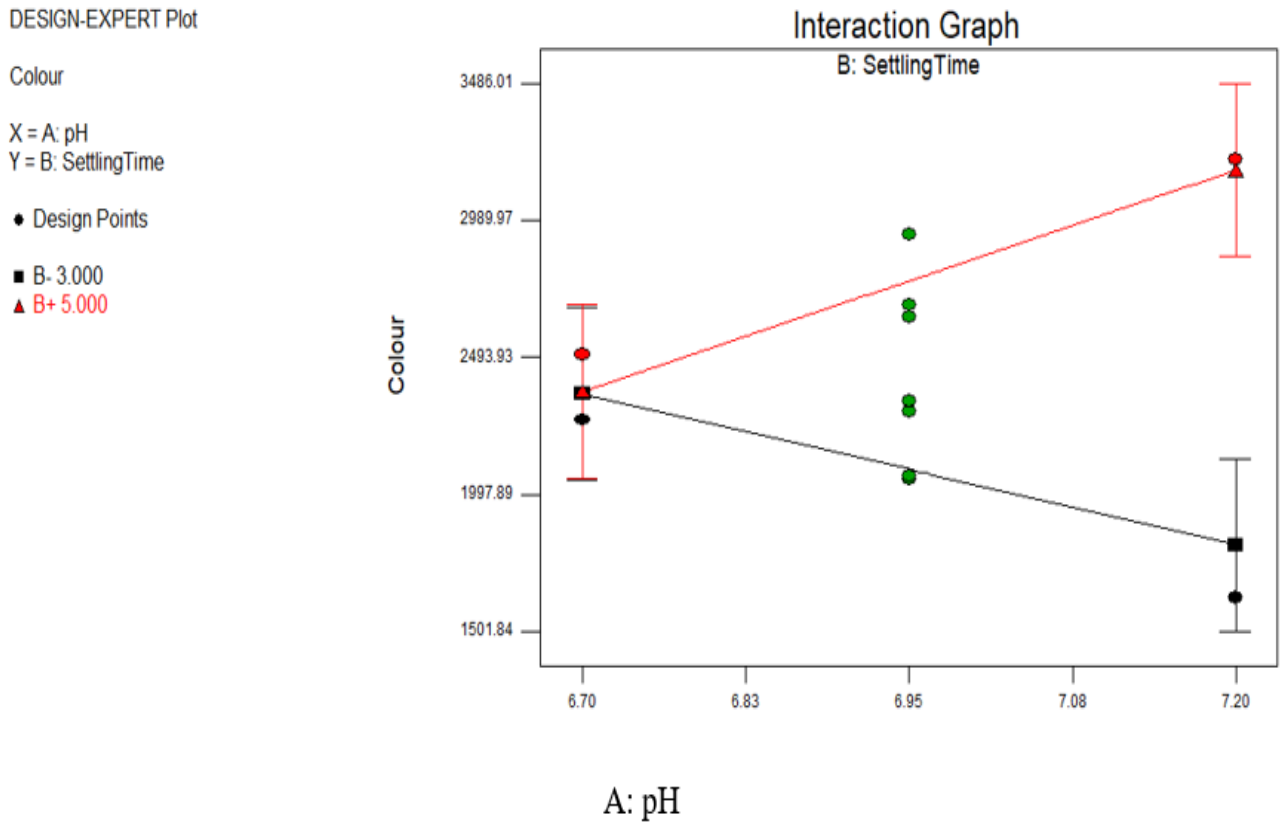


Figure 4. 6: Interaction graph of colour of clear juice

For the Figure: 4.6 shown above that, the interaction effect of pH with settling time on colour. At low levels of pH and settling time, virtually there is no difference in colour. As pH increases the gap of between low level of settling time and high level of settling time plots gets bigger and bigger because the colour increases more when the settling time increases from 3 to 5 hours with the pH from 6.7 to 7.2 and the maximum colour found was 3213 IU this is obtained at the pH of 7.2 and the settling time of 5 hours. While the minimum colour of clear juice ware 1625 IU this is obtained at the pH of 7.2 and a settling time of 3 hours. When the settling time below 3 hours the colour of the clear juice becomes decreased even if the pH of the clear juice was increased from 6.7 - 7.2. For the interval of 6.7 - 7.2 pH and at high level (five and above) of settling time, colour becomes increases with high potential. However, at a pH of 6.7 the colour value of clear

juice did not affect a great extent at settling time of either 3 or 5 hours. From this result, we can conclude that colour will disturbed greatly by settling time and the interaction factors.

4.3.3. Contour Plot for colour of clear juice

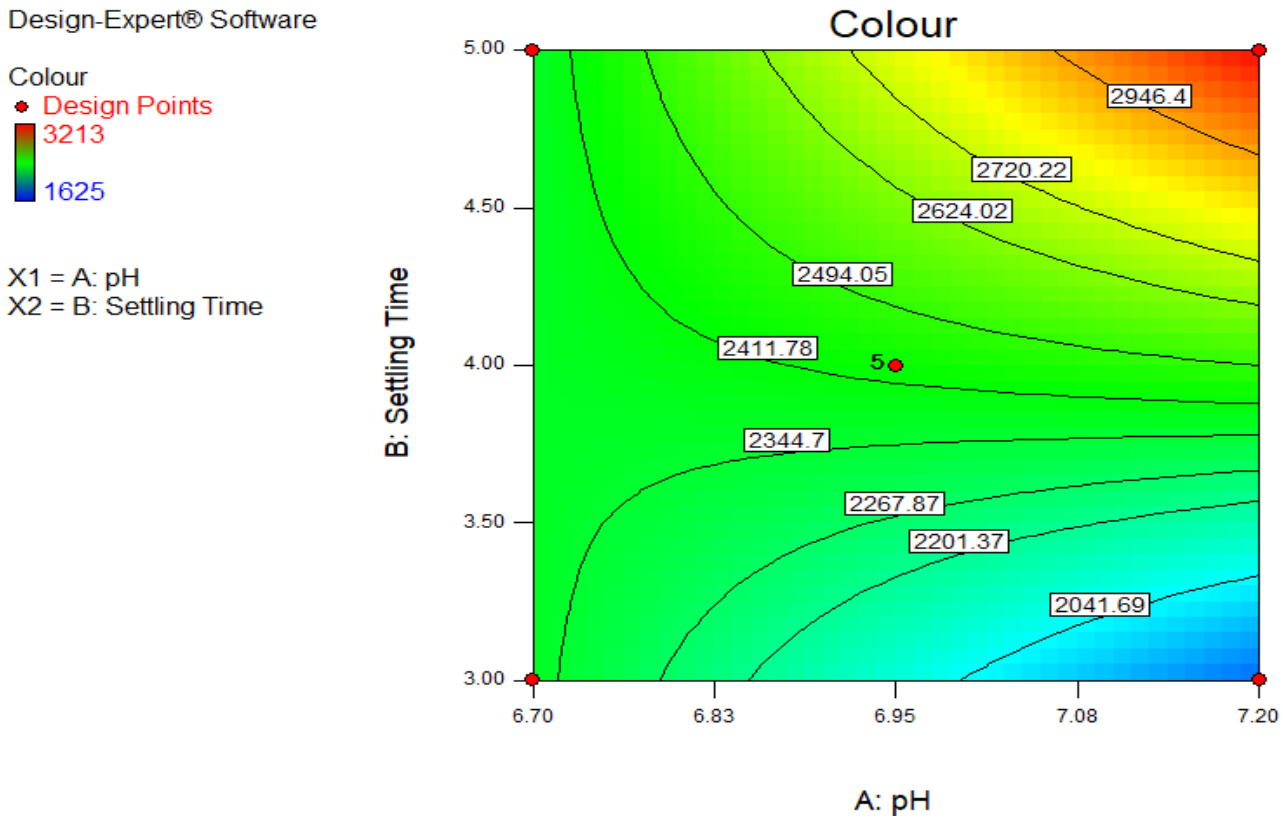


Figure 4. 7: Contour Plot of colour of clear juice

As it was from, Figure 4.7 above observed that as the settling time increases with pH increases the colour of the clear juice becomes increased. This is due to an increase in the colouring matter in raw juices. At the same time for the low level of the factors, (i.e. settling time and pH the observed colour was minimum as they were compared with that of the high levels). Also at a low level of pH and high level of settling, time the colour of clear juice becomes higher this is due to the increasing in colour forming compounds that come from the field and also due to factory processing these colouring compounds are said to be formation flavonoids and phenolic compounds. However, high level of pH and low level of settling time the colour of the clear juice

becomes minimum that is at the point of pH of 7.2 and a settling time of 3 hours which is the minimum colour recorded was 1625 IU.

Table 4. 9: Numerical optimization range of colour on clear juice analysis

Constraints Name	Goal	Lower Limit	Upper limit	Lower Weight	Upper weight	Importance
pH	is in range	6.7	7.2	1	1	3
ST	is in range	3	5	1	1	3
Colour	minimize	1625	3213	1	1	3
Turbidity	minimize	1479	3138	1	1	3
CaO	minimize	1963	3237	1	1	3

**ST- Settling Time,*CaO – Calcium Oxide Content*

The result of optimum data analysis using design expert 7.0.0 software of three solutions were observed indicated that the following parameters were selected as the optimum values of the factor levels and their response variables. in this study having an optimum range of colour, turbidity and calcium oxide content and a hundred percent desirability. The results were shown in the table 4.10 below.

Table 4. 10: Optimization values (solutions) of colour of clear juice

Number	pH	ST (hours)	Colour (ICUMSA)	Turbidity (NTU)	Calcium Oxide (ppm)	Desirability (%)	
1	7.20	3.00	1815.52	1720.36	2559.68	0.737	Selected
2	6.70	4.66	2369.37	2040.56	2142.06	0.671	
3	6.70	4.65	2369.32	2034.65	2148.04	0.671	

****ST** - Settling Time*

From the above three optimization solutions were obtained and the optimum outputs of the colour of clear juice were 1815.52, 2369.37 and 2369.32 with the desirability of 73.7%, 67.1% and 67.1 % respectively. From the first run, the optimum colour of clear juice was 1815.52 IU this was obtained at an optimum pH of 7.20 and settling time of 3 hours. Also at the corresponding turbidity and calcium oxide contents of clear juice were 1720.36 NTU and 2559.68 ppm respectively was gained from this optimization result shown that minimum colour and turbidity was obtained at a neutral PH and minimum settling time of 3 hours. In the case of second run, the optimum colour of the juice was 2369.37 IU, for this the optimum pH and settling time was 6.7 and 4.66 hours respectively, and the corresponding turbidity and calcium oxide contents were 2040.56 NTU and 2142.06 ppm respectively. From this optimization result shown that, the attainable calcium oxide content of clear juice was at minimum pH of 6.7 and optimum settling time of 4.66 hours. Finally, from the last run the selected optimum colour of the clear juice was 2369.32 IU, this was found at a pH and settling time of 6.70 and 4.65 hours respectively, and their corresponding turbidity and calcium oxide contents were 2034.65 NTU and 2148.04 ppm. So we can conclude from this optimization result at higher PH and settling time the value of colour and turbidity becomes minimum relative to that of higher level so that is the aim of this research work in order to minimize the effect of scale formation along the evaporator plant. On the other way, the amount of calcium oxide contents becomes high relative to the low level of pH and settling time.

4.4. Response Surface Methodology

Response surface methodology of central composite design (CCD) of Design-Expert 7.0.0 software was used to determine the optimal conditions of the selected response of the process. Response surface methodology is a mathematical modeling tool used to predict the output relationship with respect to the multi-input parameters. The model predicts the value of the unknown output for any desirable input. Central composite design uses the method of least-squares regression to fit the data to a certain model. The adequacy of the model was determined by evaluating the lack of fit; that was obtained from ANOVA. The statistical significance of the model and model variables were determined at a 95% confidence interval ($\alpha = 0.05$).

The result found after the data was put in design-expert software in a standard order, there has been found a single model that can be possibly fit or satisfy the significance of the factors A quadratic model was suggested, even though it has a lower R-Squared (R^2) and adjusted R-Squared (Adj-R^2) values than a cubic model. This is because the cubic model is aliased, which means that the effects of each variable that cause different signals become indistinguishable. As the RSM, if a p-value of lack of fit, $p>0.05$ (non-significant) it implies that the proposed model fit the experimental data and the independent variables or parameters has considerable effects on the dependent (response) variables. Based on this fact, the lack of fit (p-value = 0.5389) of quadratic models has been found best-fitted model among all with 53.89 % of lack of fit.

4.5. ANOVA result for turbidity of clear juice

The design program for this response to test for its adequacy and to describe its variation with independent variables suggested quadratic and 2F1 model, the Model F-value of 5.56 implies the model is significant. There is only a 2.19 % chance that a "Model F-Value" this large could occur due to noise.

Table 4. 11: ANOVA result for turbidity of Clear juice

Source	SS	df	Mean square	F - Value	Probe > F	
Model	2.131E+006	5	4.262E+005	5.56	0.0219	significant
A-pH	1.676E+005	1	1.676E+005	2.19	0.1827	
B-ST	1.197E+006	1	1.197E+006	15.63	0.0055	
AB	2.741E+005	1	2.741E+005	3.58	0.1005	
A^2	1.052E+005	1	1.052E+005	1.37	0.2797	
B^2	3.300E+005	1	3.300E+005	4.31	0.0766	
Residual	5.364E+005	7	76626.24			
Lack of Fit	2.072E+005	3	69063.50	0.84	0.5389	Not-significant
Pure Error	3.292E+005	4	82298.30			
Cor Total	2.667E+006	12				

***ST**- Settling Time, ***SS**-Sum of Squares

Values of "Probe > F" less than 0.0500 indicates model terms are significant. In this case, only B is significant model terms. Values greater than 0.1000 indicate that the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model.

The "Lack of Fit F-value" of 0.84 implies the Lack of Fit is not significant relative to the pure error. There is a 53.89 % chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good because we want the model to fit

Table 4. 12: Model adequacy result for turbidity of clear juice

Std. Dev.	276.81	R squared	0.7989
Mean	2187.77	Adj R squared	0.6553
C.V.	12.65	Pred R squared	0.4548
PRESS	1.988E+006	Adeq precision	7.622

The "Pred R squared" of 0.4548 is in reasonable agreement with the "Adj R squared" of 0.6553. "Adeq precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 7.622 indicates an adequate signal. This model was used to navigate the design space (Montgomery, 2001).

4.5.1. Equation optimization for turbidity of clear juice

Model equations were 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. The coefficient of determination of correlation is 0.7989, which is 79.89 %, it indicates that degree of fitting is good and the reliability of the trend is good. The model reaches a significant level of the probability value ($p < 0.01$). In addition, the regression coefficient and the regression intercept have reached a significant level ($p < 0.01$). Increasing the negative terms will decrease the responses and decreasing the positive terms will increase the responses.

Final equation in terms of coded factors:

$$\text{Turbidity} = +2129.40 + 144.73 \times A \times 2386.87 \times B + 261.75 \times A \times B - 122.95 \times A2 + 217.80 \times B2 \quad (4.3)$$

Where: A & B are factors A=pH, B= Settling Time

Final equation in terms of actual factors:

$$\text{Turbidity} = -65870.75487 + 23734.98473 \times \text{pH} - 8632.17774 \times (\text{Settling Time}) + 1047.00000 \times (\text{pH}) \times (\text{Settling Time}) - 1967.20000 \times (\text{pH}^2) + 217.80000 \times (\text{Settling Time}^2) \quad (4.4)$$

4.5.2. Diagnostic Test for the Responses

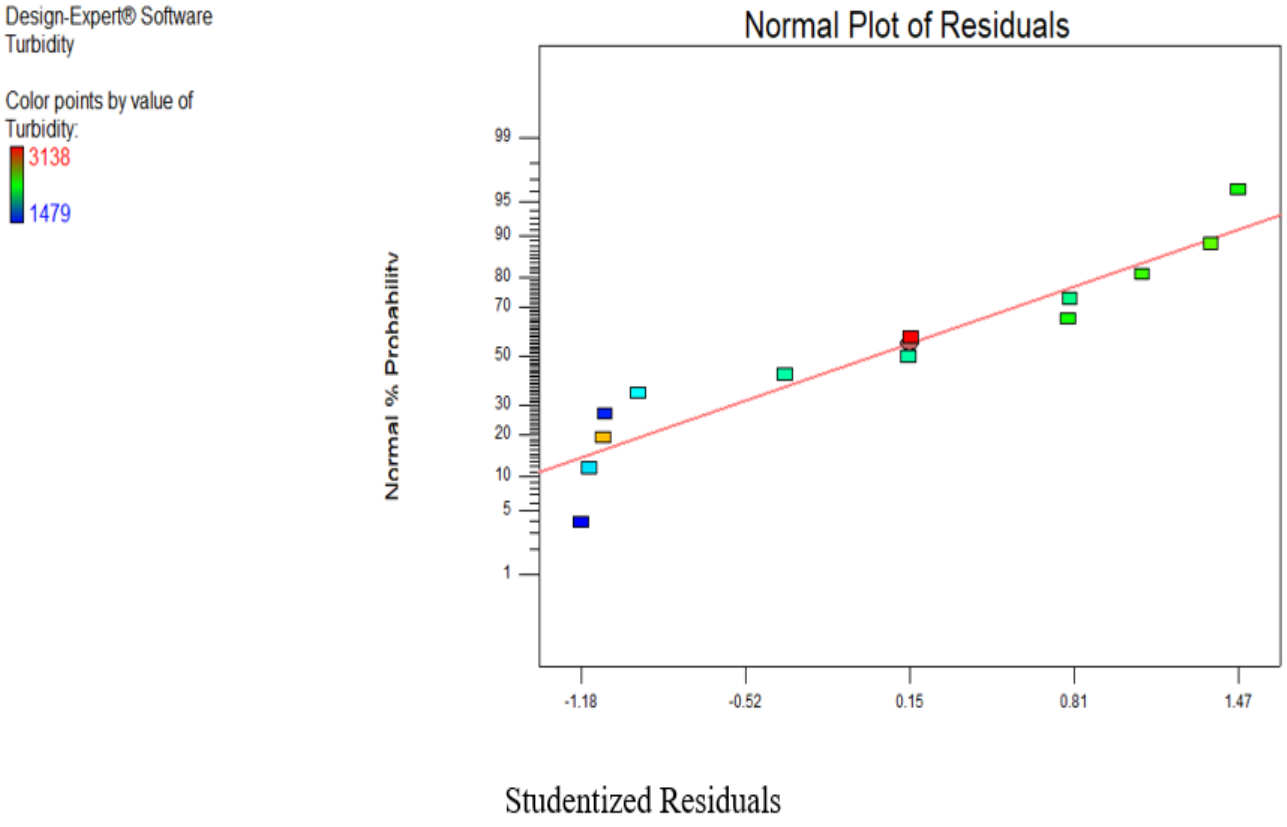


Figure 4. 8: Normal probability plot for turbidity

All diagnostic plots were also tested for all responses for adequacy of the models (normal plot of residuals, residual vs predicted value, residual vs. factor, studentzed residuals, etc.).For example, Figure 4.8 above shows how precisely the turbidity of clear juice is modeled, because all the points are lined up correctly and the deviation of points for colour from normality is insignificant. Similar results has observed for other responses i.e. (turbidity and calcium oxide content).

4.5.3. Interaction graph for turbidity of clear juice

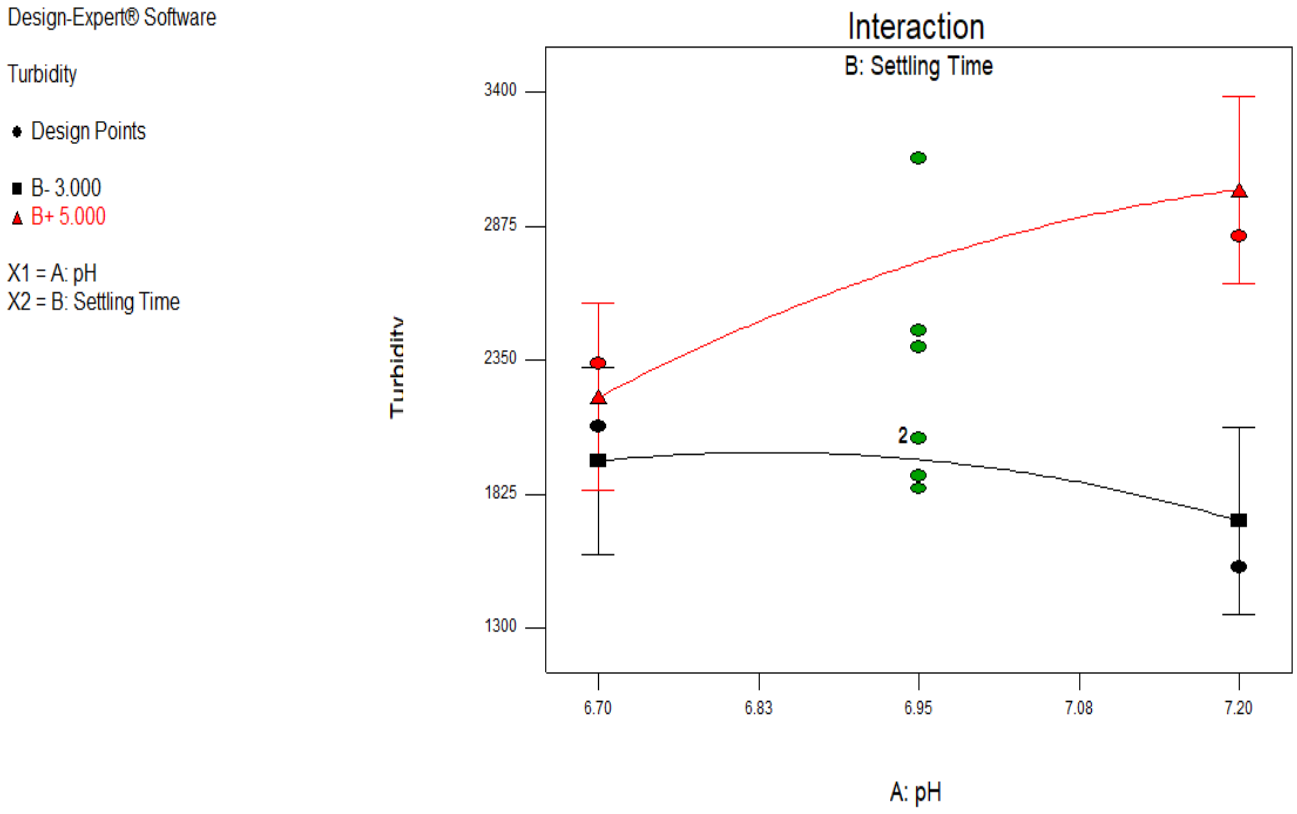


Figure 4. 9: Interaction graph for turbidity of clear juice

As the surface plot in the Figure 4.9 above indicates that when increasing the pH from 6.7 to 7.2 the interaction of turbidity with the settling time and pH is not significant from the graph. When increasing the pH from 6.7 to 7.2 increasing turbidity for high level settling time and a value of 3138 NTU this was gained at a pH of 6.95 and a settling time of 5.41 hours while, with decreasing the amount of low level settling time, then the turbidity of clear juice becomes a

decreasing trend from a PH of 6.95 - 7.2 so the minimum turbidity that observed were 1479 NTU this was gained at avalue of pH of 6.6 and a settling time of 4 hours. When pH is held constant at 6.95 and with decreases settling time result in decreasing the turbidity content of the juice. As increasing the pH beyond 6.95 to 7.2 and decreasing at low levels of settling time, then the turbidity content of the juice becomes decreases.

4.5.4. Contour plot of turbidity for clear juice

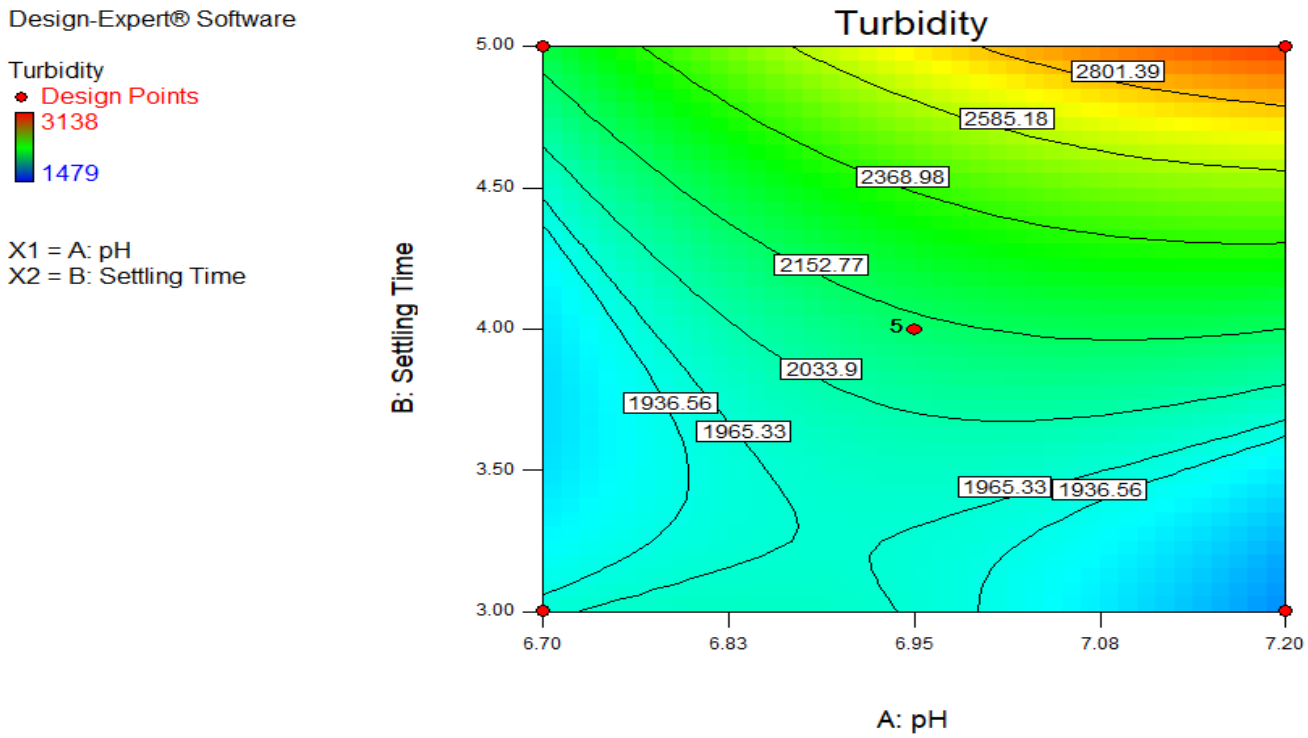


Figure 4. 10: Contour plot for turbidity of clear juice

At a constant pH of clear juice of 6.7 turbidity of clear juice, becomes minimum as relating to with variable pH and settling times of clear juice. As the contour plot in the Figure 4.10 above,indicates that when increasing the pH from 6.7 to 7.2. The interaction of turbidity with the settling time and pH is not significant from the above graph. However, it was increased for increase of pH and at a higher level of settling time. In addition, it also decreased for at a low settling time, even though the pH of raw juices has been increased. In general, it can be

concluded that settling time was a more significant factor than a pH of raw juices as compared from the interaction graph.

4.6. ANOVA results of calcium oxide content of clear juice

The design program for this response to test for its adequacy and to describe its variation with independent variables suggested quadratic model.

Table 4. 13: ANOVA test for calcium oxide contents of clear juice

Source	Sum of squares	df	Mean-quare	F-value	Prob > F	
Model	9.349E+005	5	1.870E+005	6.12	0.0171	Significant
A-pH	5.296E+005	1	5.296E+005	17.35	0.0042	
B-ST	6736.37	1	6736.37	0.22	0.6528	
AB	1.897E+005	1	1.897E+005	6.21	0.0414	
A^2	67579.59	1	67579.59	2.21	0.1804	
B^2	1.148E+005	1	1.148E+005	3.76	0.0937	
Residual	2.137E+005	7	30529.97			
Lack of Fit	99643.81	3	33214.60	1.16	0.4267	Not significant
Pure Error	1.141E+005	4	28516.50			
Cor Total	1.149E+006	12				

From the ANOVA result of the design expert the Model F-value of 6.12 implies the model is significant. There is only a 1.71 % chance that a "Model F-Value" this large could occur due to noise (personal error or disturbance). The predicted response fits well with those of experimentally obtained responses. All the factors, i.e,. pH and settling time had a significant effect individually on the calcium oxide this indicates that, the variation in magnitude of the individual factors directly affects the calcium oxide. Values of "Prob > F" less than 0.0500 indicates model terms are significant. In this case A, AB 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 the model.

Lack-of-Fit is the variation due to the model inadequacy. The "Lack of Fit F-value" of 1.16 implies the Lack of Fit is not significant relative to the pure error. There is a 42.67 % chance that a "Lack of Fit F-value" this large could occur due to noise. Non significant lack of fit is good because we want the model to fit. This non-significance lack of fit shows the proposed model fit the experimental data and the independent variables or parameters have a desirable effect on the response. This conclusion was deducted by comparing the p-value of the individual and combined factors with the percent of probability of the model (i.e. $\alpha = 0.05$). The lesser the p-value than α value has implied the larger significance effect of the factor on the response.

4.6.1. Checking of data and adequacy of model of calcium oxide

Table 4. 14: Model adequacy result for calcium oxide content of clear juice

Std. Dev.	174.73	R-Squared	0.8139
Mean	2502.62	Adj R-Squared	0.6810
C.V.	6.98	Pred R-Squared	0.4579
PRESS	8.868E+005	Adeq Precision	9.224

The above value shows that an adequate signal for the model can be used to direct the design space. The "Pred R-Squared" of 0.4579 is in reasonable agreement with the "Adj R-Squared" of 0.6810; i.e. the difference is less than 0.2. The value of correlation coefficient (R^2) of the model was 0.8139, which expresses the quality of fitness. A high (R^2) indicates that the variation could be accounted for by the data satisfactorily fitting the model. This shows that only 1.34% of the total variation in the outcome parameter assessed are unexplained by the observed model, and expresses well enough quadratic fits to navigate the design space. The “Adeq Precision" measures the signal to disturbance ratio due to random error. “A ratio greater than 4 is desirable. In this case, a ratio of 9.224 indicates an adequate signal. This model canbeused to navigate the design space.

The R^2 should be at least 0.80 for a good fit of a model even R^2 values depends on the nature of the analysis. The R^2 value obtained in the present study for these response variables was higher

than 0.80, indicating that the regression models explained the result well. Coefficient of variation (CV) is a measure expressing the standard deviation as a percentage of the mean, smaller values of CV give better result. The coefficient of variation (CV) of less than 10 (i.e. 6.98) indicated that the model was acceptable. The PRESS (Predicted Residual Sum of Squares) measure of how a particular model fits each point in the design.

Final equation in terms of coded factors:

$$\text{Calcium Oxide} = +2521.00 + 257.29 \times A - 29.02 \times B + 217.75 \times A \times B + 98.56 \times (A^2) - 128.44 \times (B^2) \quad (4.5)$$

Where: A and B are factors A- pH, B- Settling Time

Final equation in terms of actual factors

$$\text{Calcium Oxide} = +93816.13583 - 24375.12320 \times (\text{pH}) - 5054.96803 \times (\text{Settling Time}) + 871.00000 \times \text{pH} \times (\text{Settling Time}) + 1577.00000 \times (\text{pH})^2 - \text{settling Tme} \quad (4.6)$$

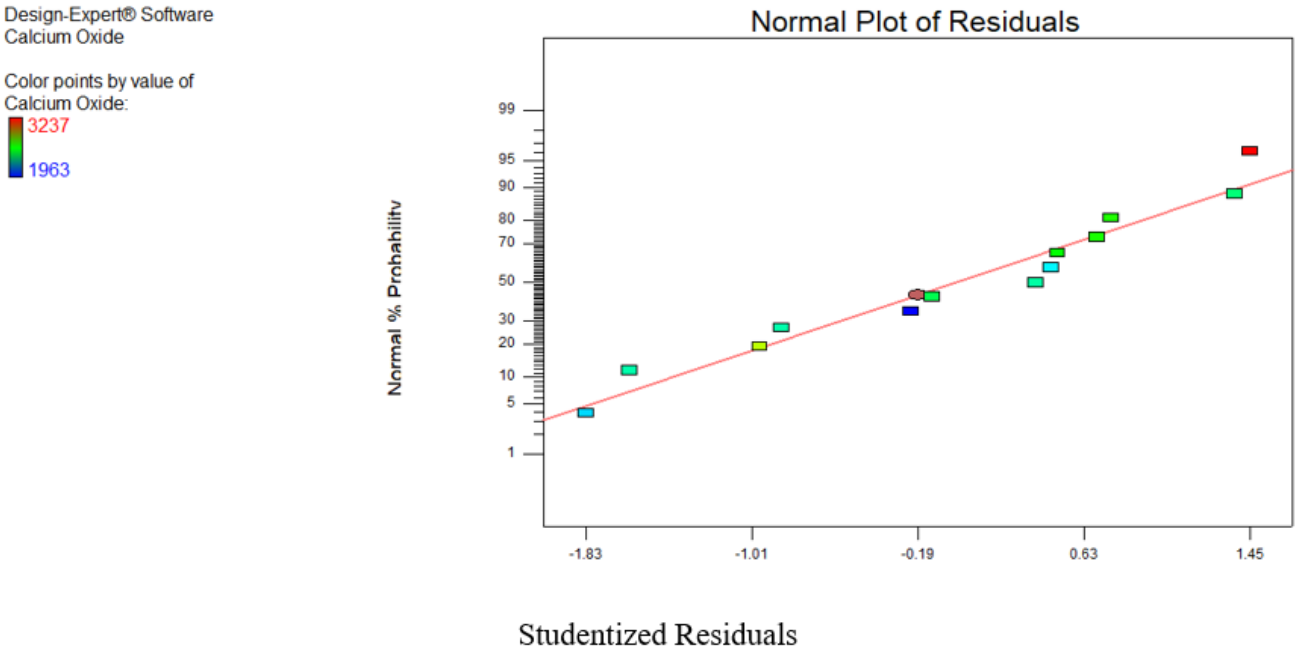


Figure 4. 11: Normal probability plot

As shown in figure 4.11 above, the normal probability plot indicates the residuals following by the normal % probability distribution, in the case of this experimental data the points in the plots show fitted to the straight line in the figure, this shows that the quadratic polynomial model satisfies the assumptions analysis of variance (ANOVA) i.e., the error distribution is approximately normal.

4.6.2. Interaction graph of calcium oxide content of clear juice

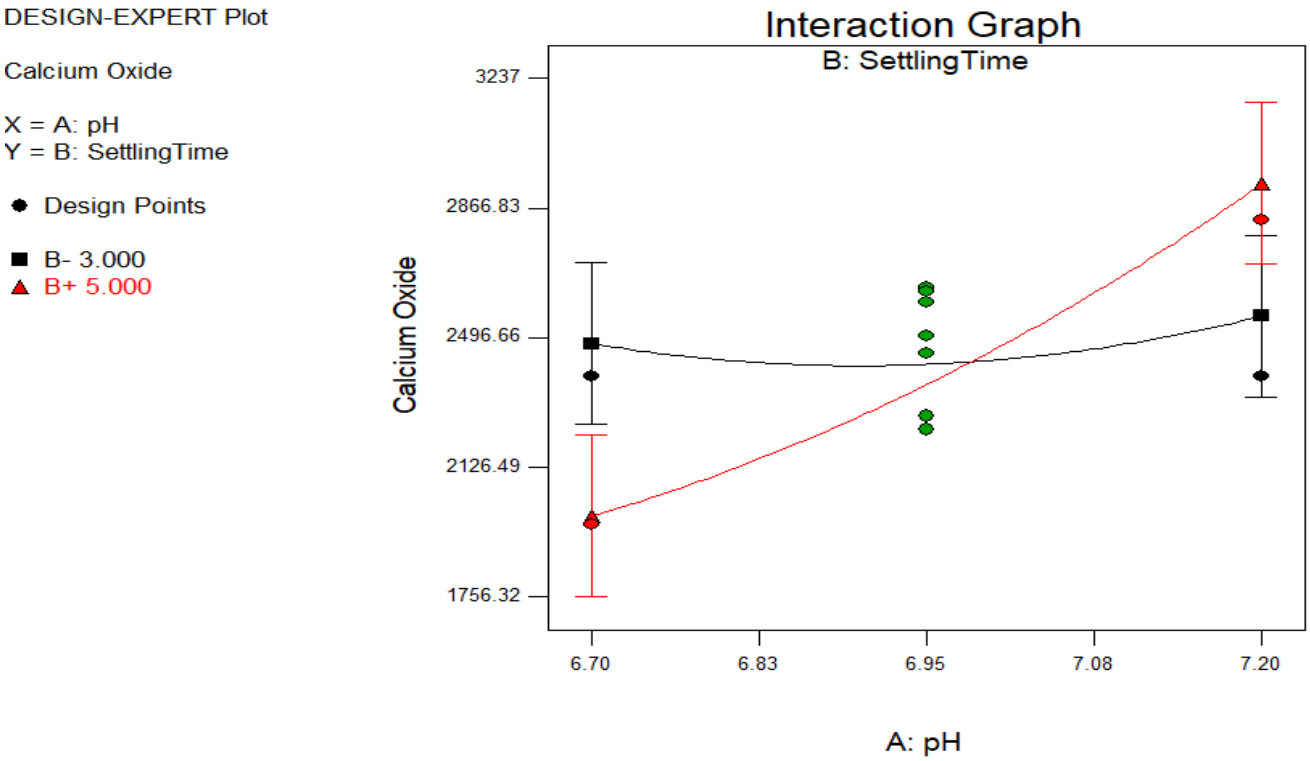


Figure 4. 12: Interaction graph for calcium oxide contents of clear juice

The figure above shows the interaction effects of pH and settling time on calcium oxide content. This type of interaction is called crossover interaction. Crossover interactions often indicate that a factor has one kind of effect in one condition and the opposite kind of effect in another condition. Since the pH of 7.0, the lowest level of settling time favor over the higher level by giving a maximum calcium oxide content of 3237 ppm this is obtained at a pH of 7.3 and a settling time of 4 hrs. The interaction graph of Figure 4.12 shown above is significant. The point at which the fixed amount of calcium oxide levels were obtained from the intersection of high and low levels of settling time at between 6.95 and 7.08, which is pH values of 7.0. For the values of settling time below, the intersection point the calcium oxide content increased when it subjected with high level of settling time and for the interval of 6.7-7.0 pH at the same time calcium oxide content decreased slightly for low level of settling time and for the same interval of pH.

4.6.3. Contour plot for calcium oxide contents of clear juice

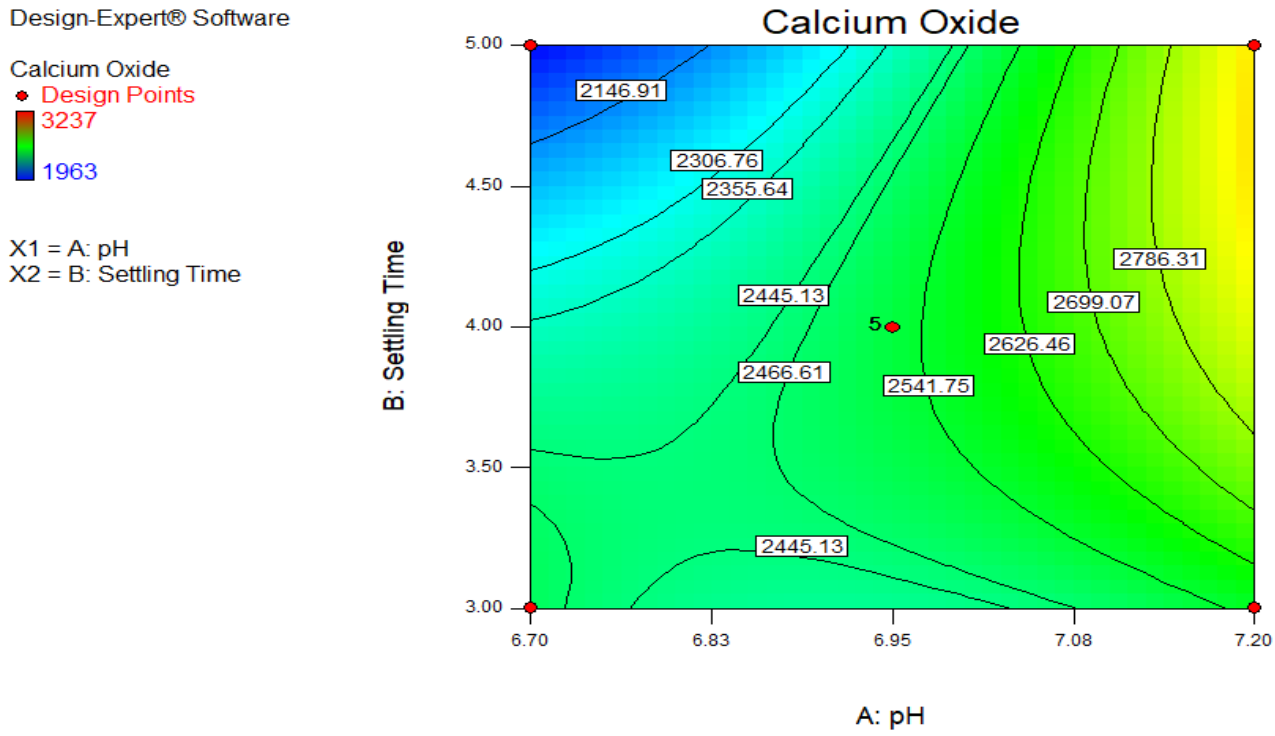


Figure 4. 13: Contour graph for calcium oxide of clear juice

In general, the calcium oxides content of the factory was greatly above the standard operations. For the settling time below of 3.5 hours and approximately of 6.7 pH, the obtained calcium oxide contents equal with for settling time of 5 hours and 7.08 of pH. This is due to fast settling time subjected with low pH and 7.08 pH at high settling time of 5 hours. The minimum calcium oxides were obtained at pH of 6.7 even though the settling time increases from 4.5 - 5 hours. Therefore, it shows that the calcium oxide content of clear juice has increase for both increasing of settling time and pH value finally the greater scale formation is formed higher pH and settling time. To minimize this problem care must be taken to come up the optimum condition in Table 4.9 above.

CHAPTER FIVE

5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

All Ethiopian sugar factories have been practiced in the production of plantation white sugar by proper application of lime and controlling the settling time in the clarification unit. The result of the study has indicated that it is mainly focused on two things: one is, to do the existing process parameters of the boiling house station (Clarification plant) and the other one is the optimization of clarification process parameters that could lead to a reduction of hardness of clear juice. After identifying the most proper response variables that lead to facilitate, for the formation of clear juice hardness, then the process optimization of parameters was carried out along the clarification station. The optimization experiment has been successfully designed, analyzed, proper data are generated and statically validated. Response surface methodology of complete composite design was used to optimize the parameters since it is an essential tool for process optimization.

This study indicates that there can be significant potential for minimizing the clear juice hardness in the clarification units so as to minimize the formation of scale in the calanderia tubes of the evaporation plant. The experiment was designed in a series of steps aiming to minimize the colour, turbidity and calcium oxide content of clear juice through by optimizing the addition of lime and proper measuring of settling time. In this study effect of hardness of clear juice were carried out with two independent variables i.e. pH and settling time of (7.2, 6.7 and 6.7), and (3, 4.66 and 4.65 hrs.) respectively and their contour and interaction effects on colour, turbidity and calcium oxide were analyzed. Response surface methodology of Complete Composite Design method was used to check the model significance and optimization of colour, turbidity and calcium oxide.

Finally, the juice clarifications systems were performed in laboratory scale by optimizing the pH and settling time of the clear juice. The optimum values are presented in Table 4.10. The required range of the constraints were 6.7, 6.7 and 7.2 pH and a settling time of 3, 4.65 and 4.66 hours. Therefore, the results obtained from this work indicated that the power to produce sugar could be improved if the optimum levels of the chemical dosages are practiced. Experiencing the

process under these, ranges of conditions of levels improve the clear juice pH, settling time, the turbidity, the colour, and calcium oxide content.

The actual change in factory performance of the optimized clarification parameters, settling time, turbidity, colour and calcium oxide of clear juice could only be seen in the full operation of the factory. The performance parameters such as boiling house recovery, loss in final molasses sucrose loss due to inversion and sugar quality are also affected by variables other than clarification parameters.

5.2. Recommendations

According to the experimental results and practical lab scale observations, the following recommendations are to be considered at MSF to reduce the formation of scale in the evaporators tubes and juice heaters through minimizing the clear juice hardness. Put in practice and strictly control the optimum process parameters obtained by this study for colour, turbidity and calcium oxide content of clear juice. The existing calcium oxide content of clear juice is high or beyond the recommended level. To minimize the calcium oxide contents the lime purity must be increased in order to minimize the calcium oxide content of the clear juice. While the colour forming compounds should controlled to minimize the colour of clear juice, on the other hand we can minimize the turbidity by the proper controlling of the impurities during juice settling time. After proper controlling of the mentioned output variables the next step would be to implement the optimum processing parameters for improvement of the clarification unit and for better recovery of sucrose by controlling losses through the inversion. The implementation does not require any additional investment cost, but it requires the processing parameters like colour turbidity and calcium oxide content of the clear juice by controlling the factors during effective clarification. Also minimize cane spillage during cane unloading process and the deteriorated cane should not be returned to the cane table, because the time it goes into the production microbial duplication and deteriorations of sucrose will increase at high temperature and low pH. In addition to this training and awareness must be given to the laborers and other workers at the cane unloading station so as to control the process parameters i.e. the proper application of pH and controlling of settling time. Finally, I recommend MSF to analyze the colour and turbidity of clear juice therefore in order to prevent clear juice hardness o the factory.

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APPENDICES

Appendix -A

1. 1: **Logical framework matrix**

	Narrative Summary	Objectively verifiable (performance) Indicator	Means of verification	Important Assumptions
Program Goal	**Development objectives** Securing internationally competent sugar industry through the adoption and improvement of sugar and co-products utilization technology that minimizes	**Impact indicator** -To minimize the poverty of the country and to attain demand satisfaction.	**Source of information** -RDC research reports	**Important events** - The commitment of the top management in improving and adopting-sugar-production technologies -Competent researchers and factory staffs are hired
Project Purpose	**Immediate objective** - To minimize the scale formation by improving the clarification process. - To asses and identify the factors affecting juice clarification process.	**Effectiveness indicator** The influence of lime on hardness of clear juice in juice clarification by defecation system.	**Source of information** Performance reports of the MSF. RDC Research reports	**Important events** - Competent researchers and appropriate facilities-are-available -Commitment of stakeholders to implement the completed research -Transport, baccommodation and financing facilities are improved.

Research Outputs	**Research results** - Reducing sugars pol %, Brix, color turbidity %, calcium and magnesium oxide content, bagasse, maximum & well planned sugar boiling house station management practice.	**Output indicators** Improved sugar yield and well-planned management practice of the plant.	**Source of information** Performance reports of the Metahara sugar factory. RDC Research reports	**Important events** - Research staff and facilities are available - Transport and other facilities are available - Released technologies are implemented
Activities	**Major activities** Studying the existing conditions of the clarification plant. Conduct laboratory work and sample analysis. Identify the operation that causes for scale formation. Optimization of process parameters.	**Progress indicators** Monthly & quarterly report -Experiments conducted -Laboratory analytical results -Progress reports	**Source of information** -Research proposals -Progress reports -Research reports	**Important events** -Sugar Cooperation factory personnel -Appropriate and skilled supporting manpower is available - If necessary inputs are available -If released recommendations are applied accordingly
Inputs	**Necessary Inputs** Appropriate research laboratory at MSF Senior researchers at RDC. School advisors at AAiT. - Laboratory technicians -Transportation facilities -Adequate-advance payments.	**Resource (ground sand services)** - Manpower - Vehicle - Lab technician	**Source of information** -Progress report -Lab test report - Documents	**Preconditions** - Equipped laboratory is available - Manpower is available -Vehicle available and financial service are improved

1. 2: Monitoring & Evaluation

Out-put/Process	Monitoring & evaluation objectives	Information to be col-lected	Methods of col-lecting infor-mation	Tools of collecting in-formation
-Optimized cal-cium oxide, color & & tur-bidity of clear juice -Well prepared juice purity and reducing sugars -Improved Heat transfer	- To assess the experi-ments wheth-er conducted properly	-Factory op-eration per-sonal & re-search result implementa-tion depart-ment.	-Laboratory exper-iments, internet sources, recent documents and other related activi-ties	- Primary data using experiments and sec-ondary data from in-ternet sources, interna-tionally published pa-pers, journal articles
Process -Sample collec-tion and labora-tory analysis.	-To asses proper meth-od of test and analytical methods used.	Pol, Brix, Color, Tur-bidity, RS CaO & Mg	-Lab-experiments and related other activities	-Primary data using experiments and sec-ondary data from inter-net sources, interna-tionally published pa-pers, journal articles
-**Conduct** la-boratory exper-iment	- To asses that, experi-ments are car-ried out ac-cording to the proposal and that the data will be col-lected proper-ly.	- Responses	-Interview factory personnel -progress reports	

Appendix- B

2. 1: Pol or sucrose factors according to refractometer solids.

To obtain pol multiply the saccharometer reading obtained from dry lead sub-acetate Clarification by the factor corresponding to the refractometer solids. To obtain sucrose multiply the calculated sucrose reading by the factor: $\text{Pol Factor} = \frac{26}{99.718 \times \text{ap.sp gr at } 20^0C/20^0C}$

Example Saccharometer reading = **41.5**

Refractometer solids = **11.50**

Pol = 41.5 x 0.24920 = **10.34**

Refr. solid	Pol factor									
	0.00	**0.01**	**0.02**	**0.03**	**0.04**	**0.05**	**0.06**	**0.07**	**0.08**	**0.09**
0.1	0.26064	0.26063	0.26062	0.26061	0.26060	0.26059	0.26057	0.26056	0.26055	0.26054
0.2	0.26053	0.26052	0.26051	0.26050	0.26049	0.26048	0.26047	0.26046	0.26045	0.26044
0.3	0.26043	0.26042	0.26041	0.26040	0.2603	0.26038	0.26037	0.26036	0.26035	0.26034
0.4	0.26033	0.26032	0.26031	0.26030	0.26029	0.26028	0.26027	0.26026	0.26025	0.26024
0.5	0.26023	0.26022	0.26021	0.26020	0.26019	0.26018	0.26017	0.26016	0.26015	0.26014
0.6	0.26013	0.26012	0.26011	0.26010	0.26009	0.26008	0.26007	0.26006	0.26005	0.26004
0.7	0.26003	0.26002	0.26001	0.26000	0.25999	0.25997	0.25996	0.25995	0.25994	0.25993
0.8	0.25992	0.25991	0.25990	0.25989	0.25988	0.25987	0.25986	0.25985	0.25984	0.25983
0.9	0.25982	0.25981	0.25980	0.25979	0.25978	0.25977	0.25976	0.25975	0.25974	0.25973
1.0	0.25972	0.25971	0.25970	0.25969	0.25968	0.25967	0.25966	0.25965	0.25964	0.25963
1.1	0.25962	0.25961	0.25960	0.25959	0.25958	0.25957	0.25956	0.25955	0.25954	0.25953
1.2	0.25952	0.25951	0.25950	0.25949	0.25948	0.25947	0.25946	0.25945	0.25944	0.25943
1.3	0.25942	0.25941	0.25940	0.25939	0.25938	0.25937	0.2593	0.25935	0.2593	0.25933
1.4	0.25932	0.25931	0.25930	0.25929	0.25928	0.25927	0.25926	0.25925	0.25924	0.25923
1.5	0.25922	0.25921	0.25920	0.25919	0.25918	0.25917	0.25916	0.25915	0.25914	0.25913
1.6	0.25912	0.25911	0.25910	0.25909	0.25908	0.25907	0.25906	0.25905	0.25904	0.25903
1.7	0.25902	0.25901	0.25900	0.25899	0.25898	0.25897	0.25896	0.25895	0.25894	0.25893
1.8	0.25892	0.25891	0.25890	0.25889	0.25888	0.25887	0.25886	0.25885	0.25884	0.25883
1.9	0.25882	0.25881	0.25880	0.25879	0.25878	0.25877	0.25876	0.25875	0.25874	0.25873
2.0	0.25872	0.25871	0.25870	0.25869	0.25868	0.25867	0.25866	0.25865	0.25864	0.25863
2.1	0.25862	0.25861	0.25860	0.25859	0.25858	0.25857	0.25856	0.25855	0.25854	0.25853
2.2	0.25852	0.25851	0.25850	0.25849	0.25848	0.25846	0.25845	0.25844	0.25843	0.25842

2.3	0.25841	0.25840	0.25839	0.25838	0.25837	0.25836	0.25835	0.25834	0.25833	0.25832
2.4	0.25831	0.25830	0.25829	0.25828	0.25827	0.25826	0.25825	0.25824	0.25823	0.25822
2.5	0.25821	0.25820	0.25819	0.25818	0.25817	0.25816	0.25815	0.25814	0.25813	0.25812
2.6	0.25811	0.25810	0.25809	0.25808	0.25807	0.25806	0.25805	0.25804	0.25803	0.25802
2.7	0.25801	0.25800	0.25799	0.25798	0.25797	0.25796	0.25795	0.25794	0.25793	0.25792
2.8	0.25791	0.25790	0.25789	0.25788	0.25787	0.25786	0.25785	0.25784	0.25783	0.25782
2.9	0.25781	0.25780	0.25779	0.25778	0.25777	0.25776	0.25775	0.25774	0.25773	0.25772
3.0	0.25771	0.25770	0.25769	0.25768	0.25767	0.25766	0.25765	0.25764	0.25763	0.25762
3.1	0.25761	0.25760	0.25759	0.25758	0.25757	0.25756	0.25755	0.25754	0.25753	0.25752
3.2	0.25751	0.25750	0.25749	0.25748	0.25747	0.25746	0.25745	0.25744	0.25743	0.25742
3.3	0.25741	0.25740	0.25739	0.25738	0.25737	0.25736	0.25735	0.25734	0.25733	0.25732
3.4	0.25731	0.25730	0.25729	0.25728	0.25727	0.25726	0.25725	0.25724	0.25723	0.25722
3.5	0.25721	0.25720	0.25719	0.25718	0.25717	0.25716	0.25715	0.25714	0.25713	0.25712
3.6	0.25711	0.25710	0.25709	0.25708	0.25707	0.25706	0.25705	0.25704	0.25703	0.25702
3.7	0.25701	0.25700	0.25699	0.25698	0.25697	0.25696	0.25695	0.25694	0.25693	0.25692
3.8	0.25691	0.25690	0.25689	0.25688	0.25687	0.25686	0.25685	0.25684	0.25683	0.25682
3.9	0.25681	0.25680	0.25679	0.25678	0.25677	0.25676	0.25675	0.25674	0.25673	0.25672
4.0	0.25671	0.25670	0.25669	0.25668	0.25667	0.25666	0.25665	0.25664	0.25663	0.25662
4.1	0.25661	0.25660	0.25659	0.25658	0.25657	0.25656	0.25655	0.25654	0.25653	0.25652
4.2	0.25651	0.25650	0.25649	0.25648	0.25647	0.25646	0.25645	0.25644	0.25643	0.25642
4.3	0.25641	0.25640	0.25639	0.25638	0.25637	0.25636	0.25635	0.25634	0.25633	0.25632
4.4	0.25631	0.25630	0.25629	0.25628	0.25627	0.25626	0.25625	0.25624	0.25623	0.25622
4.5	0.25621	0.25620	0.25619	0.25618	0.25617	0.25616	0.25615	0.25614	0.25613	0.25612
4.6	0.25611	0.25610	0.25609	0.25608	0.25607	0.25606	0.25605	0.25604	0.25603	0.25602
4.7	0.25601	0.25600	0.25599	0.25598	0.25597	0.25596	0.25595	0.25594	0.25593	0.25592
4.8	0.25591	0.25590	0.25589	0.25588	0.25587	0.25586	0.25585	0.25584	0.25583	0.25582
4.9	0.25581	0.25580	0.25579	0.25578	0.25577	0.25576	0.25575	0.25574	0.25573	0.25572
5.0	0.25571	0.25570	0.25569	0.25568	0.25567	0.25566	0.25565	0.25564	0.25563	0.25562
5.1	0.25561	0.25560	0.25559	0.25558	0.25557	0.25556	0.25555	0.25554	0.25553	0.25552
5.2	0.25551	0.25550	0.25549	0.25548	0.25547	0.25546	0.25545	0.25544	0.25543	0.25542
5.3	0.25541	0.25540	0.25539	0.25538	0.25537	0.25536	0.25535	0.25534	0.25533	0.25532
5.4	0.25531	0.25530	0.25529	0.25528	0.25527	0.25526	0.25525	0.25524	0.25523	0.25522
5.5	0.25521	0.25520	0.25519	0.25518	0.25517	0.25516	0.25515	0.25514	0.25513	0.25512
5.6	0.25511	0.25510	0.25509	0.25508	0.25507	0.25506	0.25505	0.25504	0.25503	0.25502
5.7	0.25501	0.25500	0.25499	0.25498	0.25497	0.25496	0.25495	0.25494	0.25493	0.25492

5.8	0.25491	0.25490	0.25489	0.25488	0.25487	0.25486	0.25485	0.25484	0.25483	0.25482
5.9	0.25481	0.25480	0.25479	0.25478	0.25477	0.25476	0.25475	0.25474	0.25473	0.25472
6.0	0.25471	0.25470	0.25469	0.25468	0.25467	0.25466	0.25465	0.25464	0.25463	0.25462
6.1	0.25461	0.25460	0.25459	0.25458	0.25457	0.25456	0.25455	0.25454	0.25453	0.25452
6.2	0.25451	0.25450	0.25449	0.25448	0.25447	0.25446	0.25445	0.25444	0.25443	0.25442
6.3	0.25441	0.25440	0.25439	0.25438	0.25437	0.25436	0.25435	0.25434	0.25433	0.25432
6.4	0.25431	0.25430	0.25429	0.25428	0.25427	0.25426	0.25425	0.25424	0.25423	0.25422
6.5	0.25421	0.25420	0.25419	0.25418	0.25417	0.25416	0.25415	0.25414	0.25413	0.25412
6.6	0.25411	0.25410	0.25409	0.25408	0.25407	0.25406	0.25405	0.25404	0.25403	0.25402
6.7	0.25401	0.25400	0.25399	0.25398	0.25397	0.25396	0.25395	0.25394	0.25393	0.25392
6.8	0.25391	0.25390	0.25389	0.25388	0.25387	0.25386	0.25385	0.25384	0.25383	0.25382
6.9	0.25381	0.25380	0.25379	0.25378	0.25377	0.25376	0.25375	0.25374	0.25373	0.25372
7.0	0.25371	0.25370	0.25369	0.25368	0.25367	0.25366	0.25365	0.25364	0.25363	0.25362
7.1	0.25360	0.25359	0.25358	0.25357	0.25356	0.25355	0.25354	0.25353	0.25352	0.25351
7.2	0.25350	0.25349	0.25348	0.25347	0.25346	0.25345	0.25344	0.25343	0.25342	0.25341
7.3	0.25340	0.25339	0.25338	0.25337	0.25336	0.25335	0.25334	0.25333	0.25332	0.25331
7.4	0.25330	0.25329	0.25328	0.25327	0.25326	0.25325	0.25324	0.25323	0.25322	0.25321
7.5	0.25320	0.25319	0.25318	0.25317	0.25316	0.25315	0.25314	0.25313	0.25312	0.25311
7.6	0.25310	0.25309	0.25308	0.25307	0.25306	0.25305	0.25304	0.25303	0.25302	0.25301
7.7	0.25299	0.25298	0.25297	0.25296	0.25295	0.25294	0.25293	0.25292	0.25291	0.25290
7.8	0.25289	0.25288	0.25287	0.25286	0.25285	0.25284	0.25283	0.25282	0.25281	0.25280
7.9	0.25279	0.25278	0.25277	0.25276	0.25275	0.25274	0.25273	0.25272	0.25271	0.25270
8.0	0.25269	0.25268	0.25267	0.25266	0.25265	0.25264	0.25263	0.25262	0.25261	0.25260
8.1	0.25260	0.25259	0.25258	0.25257	0.25256	0.25255	0.25254	0.25253	0.25252	0.25251
8.2	0.25250	0.25249	0.25248	0.25247	0.25246	0.25245	0.25244	0.25243	0.25242	0.25241
8.3	0.25240	0.25239	0.25238	0.25237	0.25236	0.25235	0.25234	0.25233	0.25232	0.25231
8.4	0.25230	0.25229	0.25228	0.25227	0.25226	0.25225	0.25224	0.25223	0.25222	0.25221
8.5	0.25220	0.25219	0.25218	0.25217	0.25216	0.25217	0.25215	0.25214	0.25213	0.25212
8.6	0.25210	0.25209	0.25208	0.25207	0.25206	0.25205	0.25204	0.25203	0.25202	0.25201
8.7	0.25200	0.25199	0.25198	0.25197	0.25196	0.25195	0.25194	0.25193	0.25192	0.25191
8.8	0.25190	0.25189	0.25188	0.25187	0.25186	0.25185	0.25184	0.25183	0.25182	0.25181
8.9	0.25180	0.25179	0.25178	0.25177	0.25176	0.25175	0.25174	0.25173	0.25172	0.25171
9.0	0.25170	0.25169	0.25168	0.25167	0.25166	0.25165	0.25164	0.25163	0.25162	0.25161
9.1	0.25160	0.25159	0.25158	0.25157	0.25156	0.25155	0.25154	0.25153	0.25152	0.25151
9.2	0.25150	0.25149	0.25148	0.25147	0.25146	0.25145	0.25144	0.25143	0.25142	0.25141

9.3	0.25140	0.25139	0.25138	0.25137	0.25136	0.25135	0.25134	0.25133	0.25132	0.25131
9.4	0.25130	0.25129	0.25128	0.25127	0.25126	0.25125	0.25124	0.25123	0.25122	0.25121
9.5	0.25120	0.25119	0.25118	0.25117	0.25116	0.25115	0.25114	0.25113	0.25112	0.25111
9.6	0.25110	0.25109	0.25108	0.25107	0.25106	0.25105	0.25104	0.25103	0.25102	0.25101
9.7	0.25100	0.25099	0.25098	0.25097	0.25096	0.25095	0.25094	0.25093	0.25092	0.25091
9.8	0.25090	0.25089	0.25088	0.25087	0.25086	0.25085	0.25084	0.25083	0.25082	0.25081
9.9	0.25080	0.25079	0.25078	0.25077	0.25076	0.25075	0.25074	0.25073	0.25072	0.25071
10.0	0.25070	0.25069	0.25068	0.25067	0.25066	0.25065	0.25064	0.25063	0.25062	0.25061
10.1	0.25060	0.25059	0.25058	0.25057	0.25056	0.25055	0.25054	0.25053	0.25052	0.25051
10.2	0.25050	0.25049	0.25048	0.25047	0.25046	0.25045	0.25044	0.25043	0.25042	0.25041
10.3	0.25040	0.25039	0.25038	0.25037	0.25036	0.25035	0.25034	0.25033	0.25032	0.25031
10.4	0.25030	0.25029	0.25028	0.25027	0.25026	0.25025	0.25024	0.25023	0.25022	0.25021
10.5	0.25020	0.25019	0.25018	0.25017	0.25016	0.25015	0.25014	0.25013	0.25012	0.25011
10.6	0.25010	0.25009	0.25008	0.25007	0.25006	0.25005	0.25004	0.25003	0.25002	0.25001
10.7	0.25000	0.24999	0.24998	0.24997	0.24996	0.24995	0.24994	0.24993	0.24992	0.24991
10.8	0.24990	0.24989	0.24988	0.24987	0.24986	0.24985	0.24984	0.24983	0.24982	0.24981
10.9	0.24980	0.24979	0.24978	0.24977	0.24976	0.24975	0.24974	0.24973	0.24972	0.24971
11.0	0.24970	0.24969	0.24968	0.24967	0.24966	0.24965	0.24964	0.24963	0.24962	0.24961
11.1	0.24960	0.24959	0.24958	0.24957	0.24956	0.24955	0.24954	0.24953	0.24952	0.24951
11.2	0.24950	0.24949	0.24948	0.24947	0.24946	0.24945	0.24944	0.24943	0.24942	0.24941
11.3	0.24940	0.24939	0.24938	0.24937	0.24936	0.24935	0.24934	0.24933	0.24932	0.24931
11.4	0.24930	0.24929	0.24928	0.24927	0.24926	0.24925	0.24924	0.24923	0.24922	0.24921
11.5	0.24920	0.24919	0.24918	0.24917	0.24916	0.24915	0.24914	0.24913	0.24912	0.24911
11.6	0.24910	0.24909	0.24908	0.24907	0.24906	0.24905	0.24904	0.24903	0.24902	0.24901
11.7	0.24900	0.24899	0.24898	0.24897	0.24896	0.24895	0.24894	0.24893	0.24892	0.24891
11.8	0.24890	0.24889	0.24888	0.24887	0.24886	0.24885	0.24884	0.24883	0.24882	0.24881
11.9	0.24880	0.24879	0.24878	0.24877	0.24876	0.24875	0.24874	0.24873	0.24872	0.24871
12.0	0.24870	0.24869	0.24868	0.24867	0.24866	0.24865	0.24864	0.24863	0.24862	0.24861
12.1	0.24860	0.24859	0.24858	0.24857	0.24856	0.24855	0.24854	0.24853	0.24852	0.24851
12.2	0.24850	0.24849	0.24848	0.24847	0.24846	0.24845	0.24844	0.24843	0.24842	0.24841
12.3	0.24840	0.24839	0.24838	0.24837	0.24836	0.24835	0.24834	0.24833	0.24832	0.24831
12.4	0.24830	0.24829	0.24828	0.24827	0.24826	0.24825	0.24824	0.24823	0.24822	0.24821
12.5	0.24820	0.24819	0.24818	0.24817	0.24816	0.24815	0.24814	0.24813	0.24812	0.24811
12.6	0.24810	0.24809	0.24808	0.24807	0.24806	0.24805	0.24804	0.24803	0.24802	0.24801
12.7	0.24800	0.24799	0.24798	0.24797	0.24796	0.24795	0.24794	0.24793	0.24792	0.24791

12.8	0.24790	0.24789	0.24788	0.24787	0.24786	0.24785	0.24784	0.24783	0.24782	0.24781
12.9	0.24780	0.24779	0.24778	0.24777	0.24776	0.24775	0.24774	0.24773	0.24772	0.24771
13.0	0.24771	0.24770	0.24771	0.24769	0.24768	0.24767	0.24766	0.24765	0.24764	0.24763
13.1	0.24762	0.24761	0.24760	0.24759	0.24758	0.24757	0.24756	0.24755	0.24754	0.24753
13.2	0.24752	0.24751	0.24750	0.24749	0.24748	0.24747	0.24746	0.24745	0.24744	0.24743
13.3	0.24742	0.24741	0.24740	0.24739	0.24738	0.24737	0.24736	0.24735	0.24734	0.24733
13.4	0.24732	0.24731	0.24730	0.24729	0.24728	0.24727	0.24726	0.24725	0.24724	0.24723
13.5	0.24722	0.24721	0.24720	0.24719	0.24718	0.24717	0.24716	0.24715	0.24714	0.24713
13.6	0.24712	0.24711	0.24710	0.24709	0.24708	0.24707	0.24706	0.24705	0.24704	0.24703
13.7	0.24702	0.24701	0.24700	0.24699	0.24698	0.24697	0.24696	0.24695	0.24694	0.24693
13.8	0.24692	0.24691	0.24690	0.24659	0.24658	0.24657	0.24656	0.24655	0.24654	0.24653
13.9	0.24652	0.24651	0.24650	0.24649	0.24648	0.24647	0.24646	0.24645	0.24644	0.24643
14.0	0.24642	0.24641	0.24640	0.24639	0.24638	0.24637	0.24636	0.24635	0.24634	0.24633
14.1	0.24632	0.24631	0.24630	0.24629	0.24628	0.24627	0.24626	0.24625	0.24624	0.24623
14.2	0.24622	0.24621	0.24620	0.24619	0.24618	0.24617	0.24616	0.24615	0.24614	0.24613
14.3	0.24612	0.24611	0.24610	0.24609	0.24608	0.24607	0.24606	0.24605	0.24604	0.24603
14.4	0.24602	0.24601	0.24600	0.24599	0.24598	0.24597	0.24596	0.24595	0.24594	0.24593
14.5	0.24592	0.24591	0.24590	0.24589	0.24588	0.24587	0.24586	0.24585	0.24584	0.24583
14.6	0.24582	0.24581	0.24580	0.24579	0.24578	0.24577	0.24576	0.24575	0.24574	0.24573
14.7	0.24572	0.24571	0.24570	0.24569	0.24568	0.24567	0.24566	0.24565	0.24564	0.24563
14.8	0.24562	0.24561	0.24560	0.24559	0.24558	0.24557	0.24556	0.24555	0.24554	0.24553
14.9	0.24582	0.24581	0.24580	0.24579	0.24578	0.24577	0.24576	0.24575	0.24574	0.24573
15.0	0.24572	0.24571	0.24570	0.24569	0.24568	0.24567	0.24566	0.24565	0.24564	0.24563

Appendix C

3. Metahara Sugar Factory Standard Operational Parameter

3. 1: MSF Pre-liming parameters

s/n	Parameters	Units	Standard values
1	Milk of lime	^{o}Baume	8– 10
2	Pre-liming pH set point	pH	8.5 - 9.8
3	Retention time in the pre-liming tank	Seconds	7 – 10
4	Juice temperature	^{o}C	70 – 75
5	lime slurry temperature in the slak-er	^{0}C	98-100
6	Retention time in the slaker	hours	3 - 4
7	Baume in the slaker	^{0}Baume	15

3. 2: MSF quality standards for mixed juice

s/n	Parameter	Unit	Quality standard
1	Brix	%	13.0 - 14.5
2	Purity	%	≥ 85.0
3	pH	pH	5.2 - 5.6
4	Reducing sugars	%	<0.5-1
5	Sucrose	%	> 12.00
6	Net mixed juice, % cane	%	105-110
7	P_2O_5 % MJ	%	300 – 400
8	Suspended solid % MJ	%	< 0.5
9	Temperature	^{o}C	30 – 35

**MJ*- mixed juice

Appendix-D

4. 1: Sequential model sum of squares of colour of clear juice

Source	Sum of Squares	DF	Mean Square	F- Value	Prob > F	
Mean	7.680E+007	1	7.680E+007			
Linear	9.627E+005	2	4.813E+005	4.57	0.0390	
2FI	4.556E+005	1	4.556E+005	6.85	0.0279	suggested
Quadratic	63828.03	2	31914.02	0.42	0.6739	
Cubic	1.247E+005	2	62358.02	0.76	0.5148	aliased
Residual	4.100E+005	5	82001.44			
Total	7.881E+007	13	6.063E+006			

4. 2: Lack of fit tests for colour of clear juice

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Linear	6.886E+005	6	1.148E+005	1.26	0.4314	
2FI	2.329E+005	5	46589.21	0.51	0.7612	suggested
Quadratic	1.691E+005	3	56372.68	0.62	0.6397	
Cubic	44402.00	1	44402.00	0.49	0.5242	aliased
Pure Error	3.656E+005	4	91401.30			

4. 3: Model summary statistics for colour of clear juice

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	324.68	0.4773	0.3728	0.0481	1.920E+006	suggested
2FI	257.89	0.7032	0.6043	0.4372	1.135E+006	suggested
Quadrat-ic	276.39	0.7349	0.5455	0.1205	1.774E+006	
Cubic	286.36	0.7967	0.5121	-0.6922	3.413E+006	aliased

4. 4: Sequential model sum of squares for turbidity of clarified juice

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Mean	6.222E+007	1	6.222E+007			
Linear	1.365E+006	2	6.825E+005	5.24	0.0278	suggested
2FI	2.741E+005	1	2.741E+005	2.40	0.1559	
Quadratic	4.921E+005	2	2.461E+005	3.21	0.1024	suggested
Cubic	2.022E+005	2	1.011E+005	1.51	0.3064	aliased
Residual	3.342E+005	5	66838.64			
Total	6.489E+007	13	4.992E+006			

4. 5: Lack of fit tests for turbidity of clear juice

Source	Sum of Squares	DF	Mean Square	F -Value	Prob > F	
Linear	9.734E+005	6	1.622E+005	1.97	0.2663	suggested
2FI	6.993E+005	5	1.399E+005	1.70	0.3138	
Quadratic	2.072E+005	3	69063.50	0.84	0.5389	suggested
Cubic	5000.00	1	5000.00	0.061	0.8174	aliased
Pure Error	3.292E+005	4	82298.30			

4. 6: Model summary statistics for turbidity of clear juice

Source	Std. Dev.	R-Squared	Adjusted-R^2	Predicted-R^2	PRESS	
Linear	360.91	0.5117	0.4140	0.0563	2.517E+006	suggested
2FI	338.05	0.6144	0.4859	0.1543	2.256E+006	
Quadratic	276.81	0.7989	0.6553	0.2548	1.988E+006	suggested
Cubic	258.53	0.8747	0.6993	0.6872	8.344E+005	aliased

4. 7: Sequential model sum of squares for calcium oxide of clarified juice

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Mean	8.142E+007	1	8.142E+007			
Linear	5.363E+005	2	2.682E+005	4.38	0.0430	
2FI	1.897E+005	1	1.897E+005	4.04	0.0753	
Quadratic	2.089E+005	2	1.044E+005	3.42	0.0920	suggested
Cubic	22222.69	2	11111.34	0.29	0.7600	aliased
Residual	1.915E+005	5	38297.42			
Total	8.257E+007	13	6.351E+006			

4. 8: Lack of fit tests for calcium oxide content of clarified juice

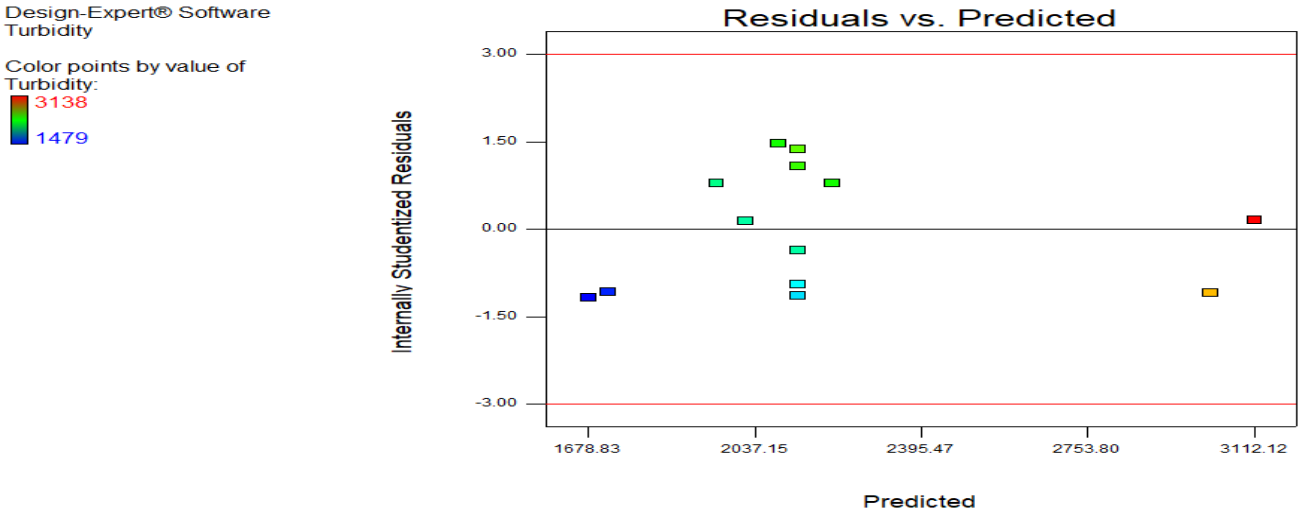
Source	Sum of Squares	DF	Mean Square	F -Value	Prob > F	
Linear	4.982E+005	6	83027.71	2.91	0.1601	
2FI	3.085E+005	5	61701.20	2.16	0.2373	
Quadratic	99643.81	3	33214.60	1.16	0.4267	suggested
Cubic	77421.12	1	77421.12	2.71	0.1748	aliased
Pure Error	1.141E+005	4	28516.50			

4. 9: Model summary statistics calcium oxide of carified juice

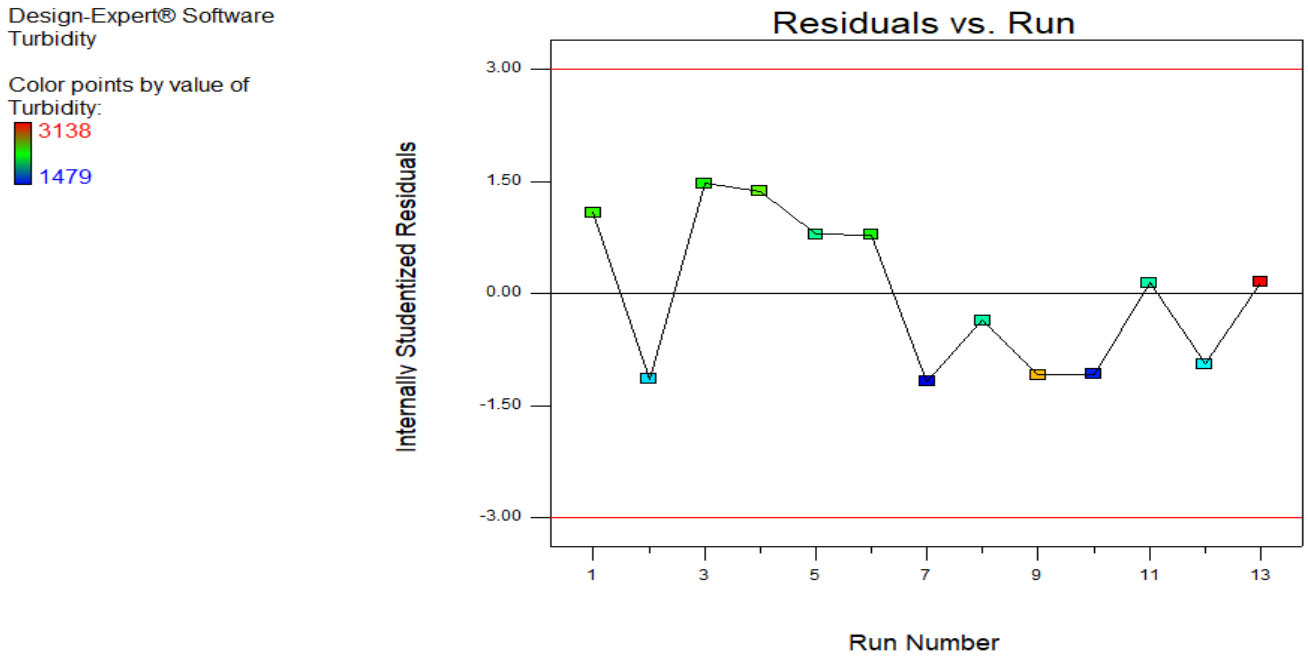
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	247.43	0.4670	0.3604	-0.0724	1.232E+006	
2FI	216.69	0.6321	0.5095	0.1173	1.014E+006	
Quadratic	174.73	0.8139	0.6810	0.2279	8.868E+005	Suggested
Cubic	195.70	0.8333	0.5999	-3.4692	5.133E+006	Aliased

Appendix-E

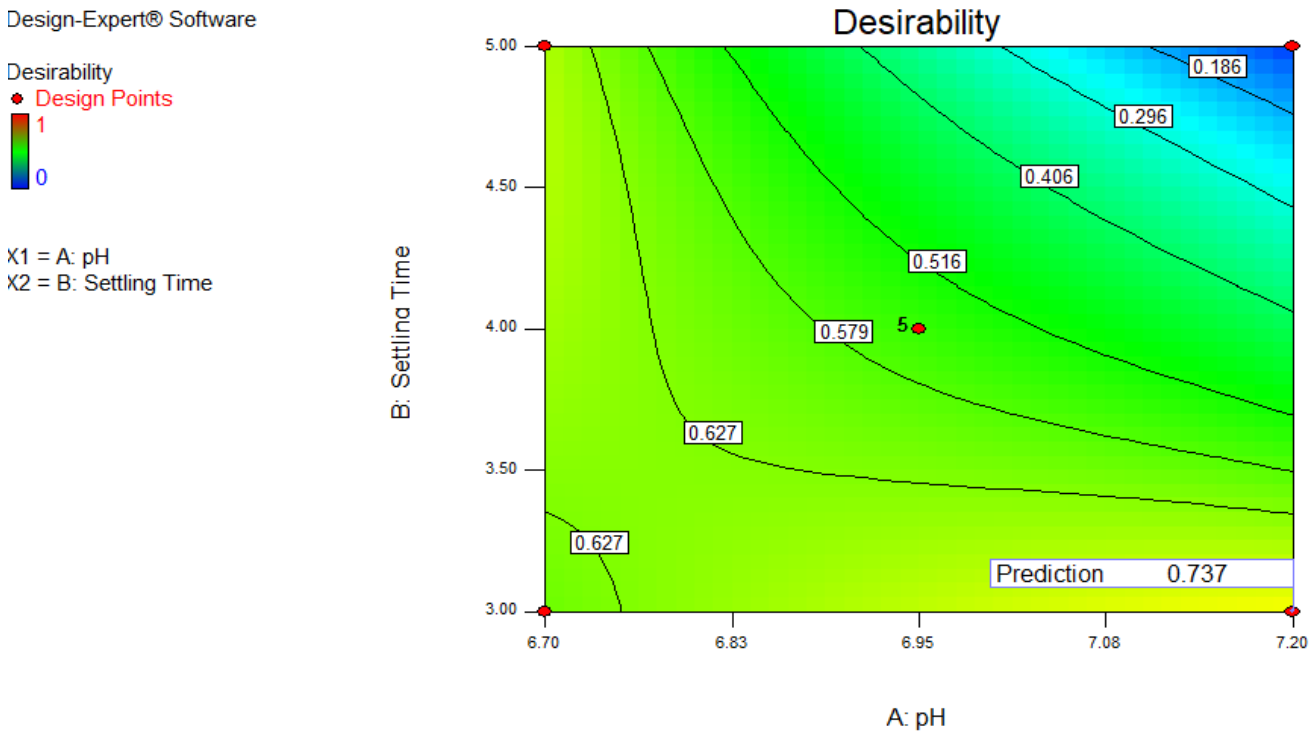
5. 1: Surface plot for residual versus predicted values for turbidity of clear juice



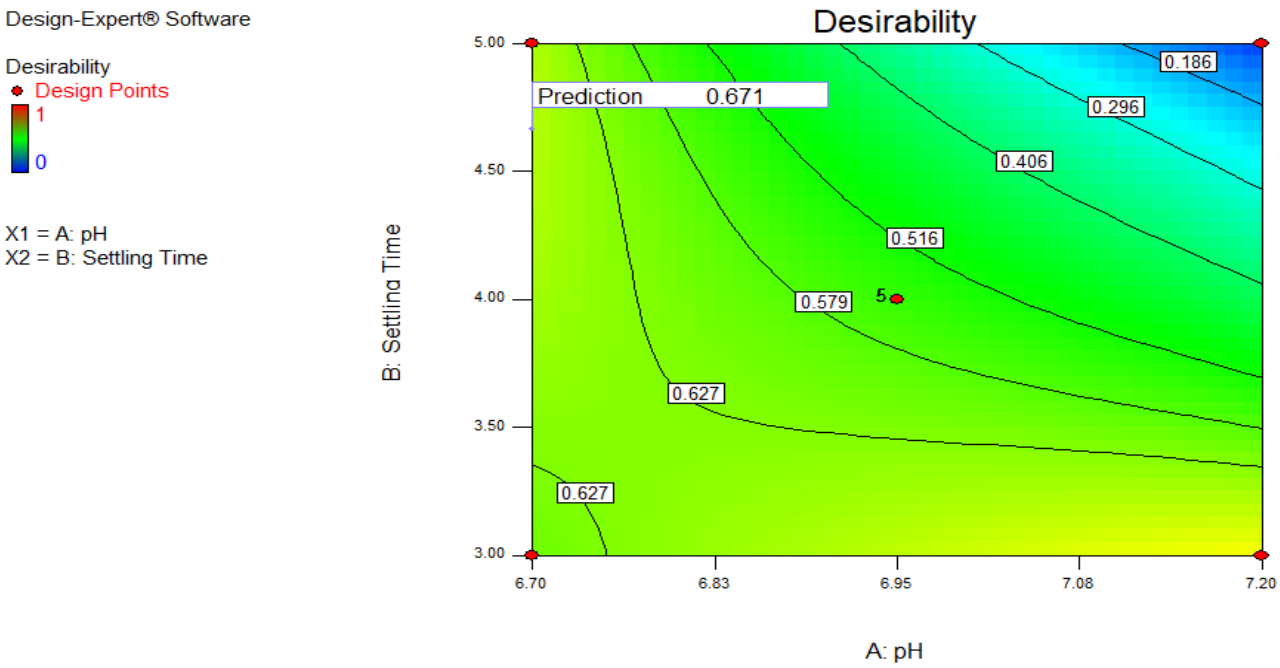
5. 2: Surface plot for residual versus run plot



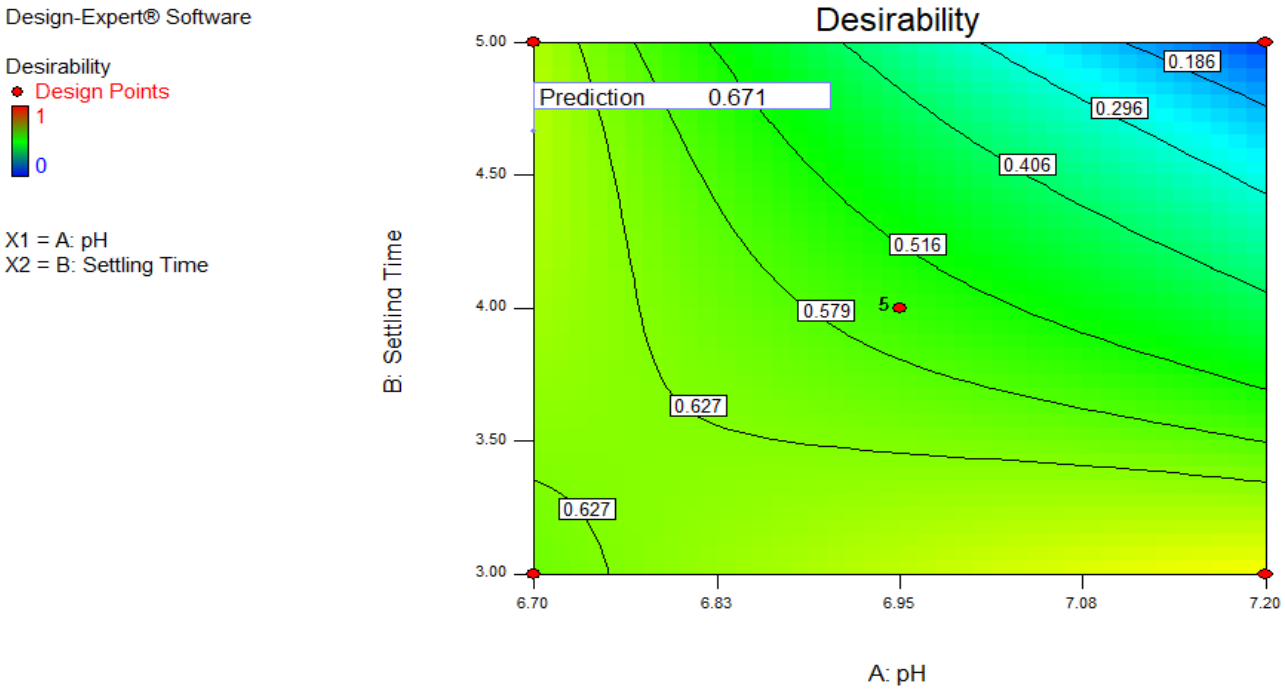
5. 3: Contour plot for turbidity of clear juice with a desirability of 73.7%



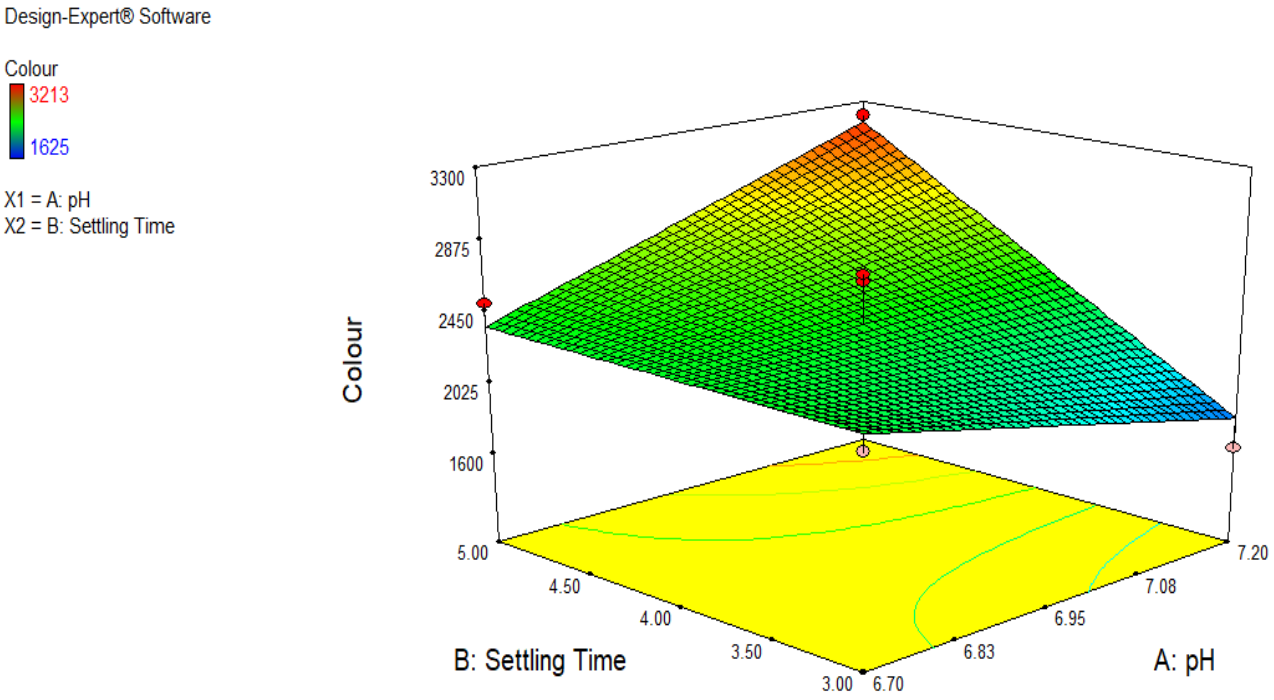
5. 4: Contour plot for colour of clear juice with a desirability of 67.1%



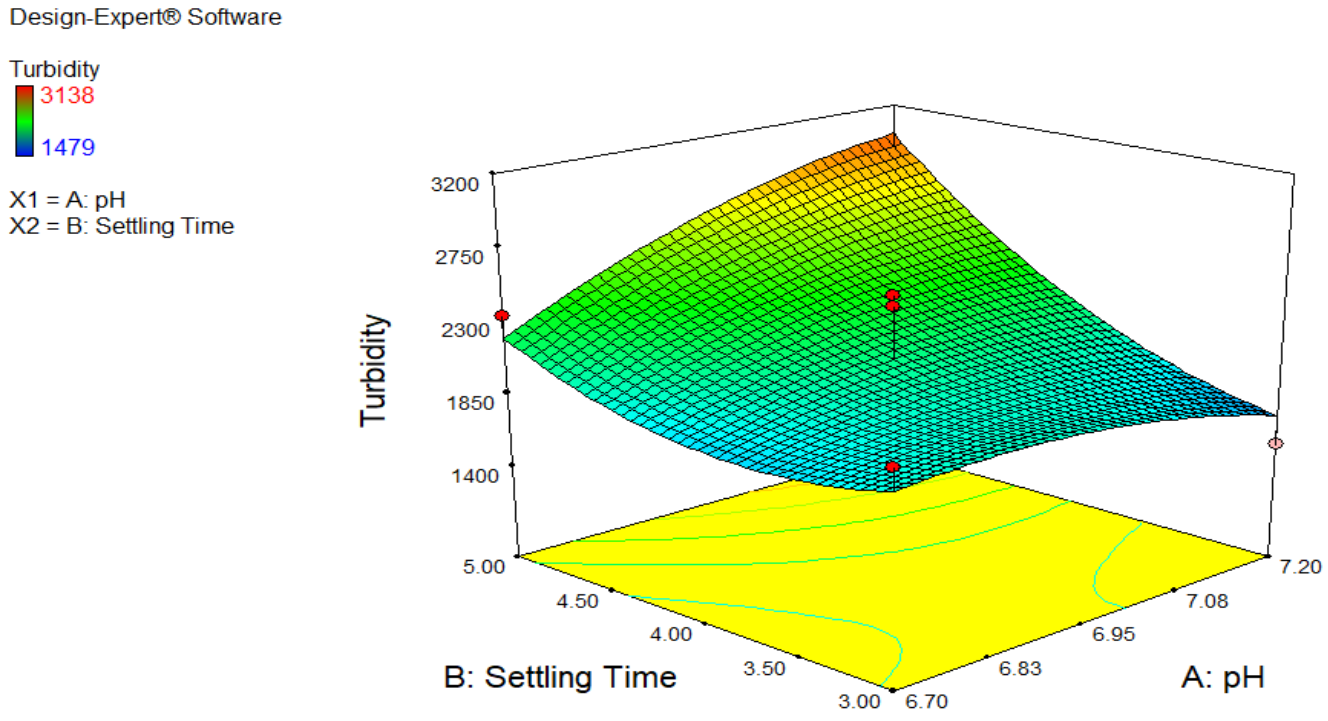
5. 5: Contour plot for calcium oxide content of clear juice with a desirability of 67.1%



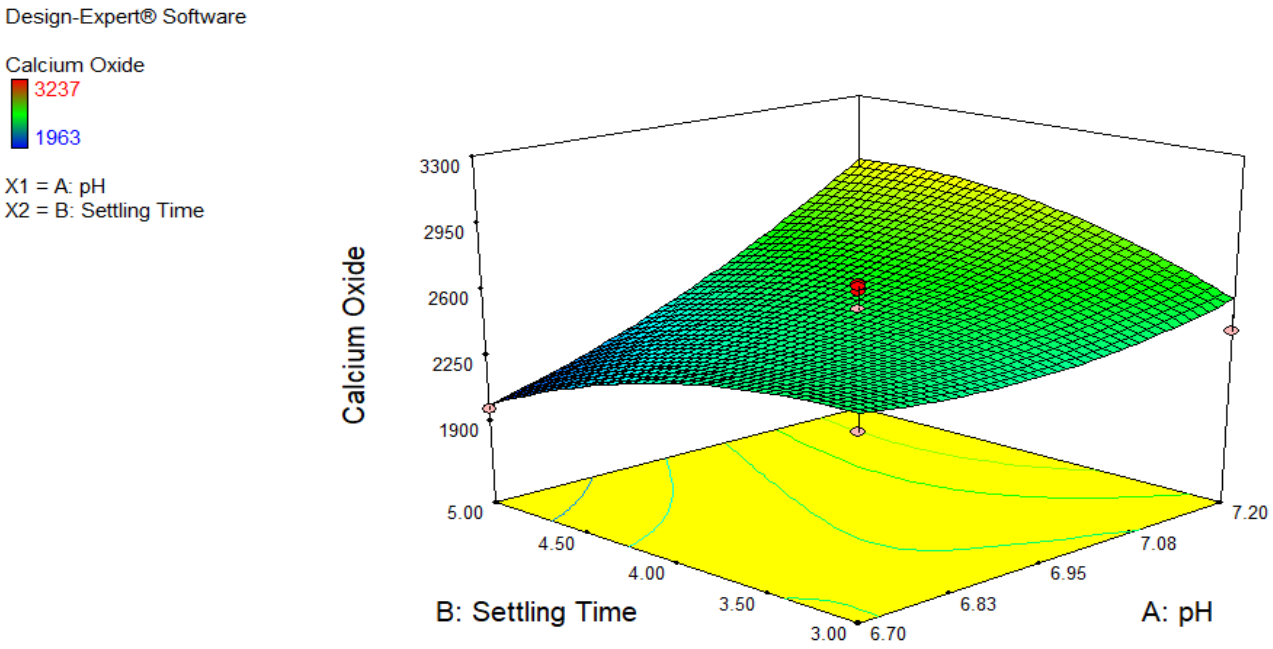
5. 6: Surface plot for colour of clear juice



5. 7: Surface plot for turbidity of clear juice



5. 8: Surface plot for calcium oxide contents of clear juice



Appendix-F

6. 1: Labratory analysis of brix % of clear juice

6. 2: Laboratory analysis of reducing sugar of clear juice

6. 3: Laboratory analysis of calcium oxide and magnesium oxide of clear juice

Appendix-G

7. 1: Conistrient solutions & their upper and lower limits of the factors and responses

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	
pH	is in range	6.7	7.2	1	1	3
ST	is in range	3	5	1	1	3
Colour	Minimize	1625	3213	1	1	3
Turbidity	Minimize	1479	3138	1	1	3
Calcium Oxide	minimize	1963	3237	1	1	3

7. 2: Optimization solutions of factors & their respective respons variables

Number	pH	ST	Colour	Turbidity	CaO	Desirability	
1	7.20	3.00	1815.52	1720.36	2559.68	0.737	
2	6.70	4.66	2369.37	2040.56	2142.06	0.671	Selected
3	6.70	4.65	2369.32	2034.65	2148.04	0.671	

Appendix-H

8. 1: Definition of Various Terminologies Used in this Paper

Pol: - The apparent sucrose content of a sugar product determined by direct or single polarization. Reading on the scale of polarometer, indicating the apparent sucrose content of the sample to be analyzed. If the sample is a pure sugar solution, the Pol equals to sucrose percentage.

Brix %: - The percent by mass of soluble solid matter (sucrose and soluble, non-sucrose) in a solution as indicated by a sugar refractometer.

Fiber %: - The dry water insoluble component of cane. Natural fiber is the fiber with chemically bound Brix-free water present in its structure.

Mixed juice: - The juice sent from the extraction plant to the boiling house.

Absolute juice: - All water contains all soluble (dissolved) solids from the cane. It thus equal to (cane – Fiber.)

Normal (undiluted) juice: - Juice pressed out by the mill tandem if no water is used for imbibition by dry crushing.

Diluted juice: - Weight of diluted juice percent weight of cane.

Saccharometer: -is simply a polarometer specially designed for measuring the polarization of sugars.

Purity: - Indicates what percentage of the solids in a sugar solution is composed of sugar. It is a percentage ratio of pol to Brix.

Reducing sugars: - The reducing substances in the cane and its products calculated as invert sugar. (Glucose, Fructose).

Sucrose: - It is the pure chemical compound disaccharide, α-D gluco-pyranosyl β- D fructo-furanoside with the formula $C_{12}H_{22}O_{11}$.

Extraneous matter: - All foreign matter delivered with a cane, or the unwanted vegetable material with a cane.

Trash content: - Materials consisting of cane leaves, root, tops, dead sticks of cane and other vegetable matter from the field in which the cane was grown.

Response Surface Methodology (RSM): - 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).

Design of experiment (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.

Dilution water: - The portion of imbibition water that goes into the diluted juice (Imbibition water minus water left in the bagasse).

Boiling House Recovery (BH Extraction) (BHR) – Mass of sucrose in sugar percent mass of sucrose in mixed juice.

Overall Recovery (OR) – Mass of sucrose in sugar percent mass of sucrose in cane.

Reduced Overall Recovery (ROR) – The actual overall recovery, reduced to what it would have been at a standard fiber content of 12.5% and a mixed juice standard gravity purity of 85.

Boiling House Efficiency (BHE) – Mass of sucrose in sugar percent mass of recoverable sucrose in mixed juice.

Factory Yield – Theoretical recoverable sugar in mixed juice and cane.

Field Yield – Theoretical recoverable sugar in cane % cane.

Plantation White Sugar or Mill White Sugar – White sugar manufactured from basic raw material in a sugar mill for direct consumption.

Refined Sugar – High purity white sugar manufactured from re-melted raw sugar.

Raw Sugar – The product of cane sugar factories which is an intermediate crystalline product resulting from the evaporation of water from sugar cane stalk juice.9