Investigation and Improvement of Sugar Clarification Process: A Case of Wonji/Shoa Sugar Factory

By Wendowesen Kassaye Gebramariam

A Thesis Submitted to Chemical Engineering Department,

School of Mechanical, Chemical and Materials Engineering

Presented to Partial Fulfillment of the Requirement for the Degree of Masters of Science in Chemical Engineering (Specialization in Process Engineering)

Office of Graduate Studies

Adama Science and Technology University

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Adama, Ethiopia

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DECLARATION

I hereby declare that this Master Thesis entitled **"Investigation and Improvement of Sugar Clarification Process: A Case of Wonji/Shoa Sugar Factory"** is my original work and has not been submitted for the award of any academic degree, diploma or certificate in any other university. All sources of materials used for this thesis have been duly acknowledged.

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Name of student	Signature	Date

RECOMMENDATION

We, the advisor(s) of this thesis, hereby certify that we have read the revised version of the thesis entitled "**Investigation and Improvement of Sugar Clarification Process: A Case of Wonji/Shoa Sugar Factory"** prepared under my/our guidance by Wendowesen Kassaye submitted in partial fulfillment of the requirements for the degree of Mater's of Science in Chemical Engineering (Specialization Process Engineering). Therefore, I/we recommend the submission of revised version of the thesis to the department following the applicable procedures.

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<u>Dr. P. Selvakumar</u>	_________________	__________________
Co-advisor	Signature	Date

APPROVAL PAGE

We, the advisors of the thesis entitled "**Investigation and Improvement of Sugar Clarification Process: A Case of Wonji/Shoa Sugar Factory"** and developed by Wendowesen Kassaye, hereby certify that the recommendation and suggestions made by the board of examiners are appropriately incorporated into the final version of the thesis.

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We, the undersigned, members of the Board of Examiners of the thesis by Wendowesen Kassaye have read and evaluated the thesis entitled "**Investigation and Improvement of Sugar Clarification Process: A Case of Wonji/Shoa Sugar Factory"** and examined the candidate during the open defense. This is, therefore, to certify that the thesis is accepted for partial fulfillment of the requirement of the degree of Master of Science in Chemical Engineering

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LIST OF ACRONYMS

Acronym	Nomenclature
As	Absorbance
ANN	Artificial neural network
CCD	Central composite design
CJ	Clear juice
°Be’	Degree Baume
DS	Dry solid
GA	Genetic algorithm
ICUMSA	International commission for uniform methods of sugar analysis
MOL	Milk of lime
MJ	Mixed juice
PWS	Plantation White Sugar
POL	Polarization
pH	Quantitative measure of the acidity/basicity of liquid
RDS	Refractometric dry substance
RSM	Response surface methodology
RVF	Rotary Vacuum Filtrate
SASTA	South African Sugar Technologist Association
TCD	Tons of cane crushed per Day
WSSF	Wonji/Shoa Sugar Factory

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ABSTRACT

Juice clarification is an important stage in cane sugar production. In this context, this study was conducted aiming to minimize color and turbidity, maximize purity change and settling rate.

Hence, analysis, modeling and optimization to examine the impact of lime, SO_2 and juice heating temperature on juice clarification in the cause of Wonji/shoa sugar factory. Purity change, color, mud settling, and Turbidity were taken as response variables to measure juice clarification efficiency of clarification plant: towards reduction of sucrose through filter cake and final molasses.

particularly, modeling and optimization on the dose of main clarification inputs, first and second heating temperature were done for efficiency improvement of clarification plant by varying the liming pH (8.1, 9.0 and 9.9); sulphited juice pH (6.2, 7.0 and 7.8); first heating temperature (60 ^{0}C, 70 ^{0}C and 80 ^{0}C) and second heating temperature (90 ^{0}C, 105 ^{0}C and 120 ^{o}C). The data obtained from the experiments were statistically validated using one-way ANOVA. Process parameters such as liming, sulphitation pH, and temperatures (first and second heating) were statistically optimized by response surface methodology (RSM) using central composite design Moreover artificial neural network (ANN) was used for prediction purpose. As a result, the optimum points that keeps all the parameters to a reasonable degree of importance and the solution with reasonable desirability were, liming pH of 9.6, sulphitation pH of 7.2, first heating of 71 ^{o}C, and second heating of 101 ^{o}C The optimal results of purity change, color, mud settling, and turbidity were 1.64%, 6,943.15 IU, 0.057 cm/s, and 836.98 IU respectively. The sugar quality obtained adequately meet international standards (i.e., ICUMSA). Also, by applying the above optimum conditions can increase productivity and quality of sugar and minimize the sucrose loss with filter cake and final molasses. Consequently, productivity can be improved.

Keywords: *Artificial neural network, Clarification parameters, performance indicators, Response surface methodology*

CHAPTER ONE

1. INTRODUCTION

1.1 Background

Sugarcane is a grass grown in tropical and subtropical countries. It is a complex hybrid of various species, derived largely from saccharum officinarum and other Saccharum species.

It is propagated vegetative by planting pieces of cane stalk(Eggleston et al., 2014). The major reason for processing cane is to recover sugar, which in its pure state referred by its chemical name sucrose. It is a combination of the two monosaccharide sugars, fructose and glucose. A sugarcane plant is comprised of juice and fiber. The juice includes a sucrose solution and other soluble inorganic and organic substances, whilst fiber constitute all the insoluble substances in the cane(Eggleston et al., 2014).

In Ethiopia, the modern sugar industry was started at Wonji with 1,400 tons/day production capacity. It was established as a share company between the Ethiopian government and Netherland' H.V.A. Company in 1951. After ten years Shoa sugar factory was built with a production capacity of 1,700 tons/day. The two factories known by the name Wonji-Shoa sugar factory (WSSF) altogether had a production capacity of 3,100 tons/day. Wonji and Shoa sugar factories were closed in 2012 and 2013, respectively because of low production capacity. Due to low capacity and oldness of these two factory government decided to build new factory so the new factory was built at Asela region place called Dodota which is around 20km away from wonji and started full capacity operation in 2013. In Ethiopia, domestic consumption of sugar increases from time to time due to the growth of population, growth of urbanization, rise of living standards, growth of sugar requiring industries, etc. Then, the demand for sugar does not meet the supply of sugar produced domestically. For this reason, the country is forced to import sugar annually to fill the gap between demand and supply. To solve this problem the government is undertaking new projects and expansion of existing factories. Nowadays, there are fourteen sugar factories and projects with the production of white and refinery sugar from sugar cane. Some of them started production like Kesem, Tana beles and Omo kurz 2 and 3 sugar factories.

In juice clarification, many chemical and process steps are utilized. First, the treatment of juice with phosphoric acid is undertaken to eliminate the phosphate deficiency in juice

(Eggleston et al., 2014). Juice is heated to 75 ºC to coagulate proteins, destroy micro-organisms and enzymes, and accelerates the reaction rate between juice acids and lime. In heated juice, lime ($Ca(OH)_2$) is added to neutralize the acidity of the juice and precipitates phosphate. Immediately, SO_2 gas is added to sulfite the juice to neutralize the excess quantity of lime and to minimize color (P. W. Rein, 2009). And, about 3 mg/L of flocculent (anionic polyacrylamide with trade name Separan) is added to decrease sugar loss with filter cake, improve the clarity of juice, increase settling rate, reduce mud volume by increasing mud density, etc. And then, the juice is heated at 102 ºC to flash out air bubbles. Finally, the clear juice is separated from mud by filtration (Asfaw, 2015a).

The main goal of the sugar industry is to maximize production capacities while simultaneously improving the product quality by improving sugar/sucrose recovery or reducing sucrose losses. To maximize production capacities, juice clarification parameters should be kept at the standard. The degree of juice clarification has a great influence on boiling house operations, profitability, and quality of the final product; therefore juice clarification efficiency has to be maximized to remove impurities that can affect any subsequent processes (Asfaw, 2015a). The goal of juice clarification is to remove impurities from juice as much as possible. Raw juice contains about 85% water, 13% sugar, about 3% of dissolved non-sugar solids, 0.3 to 0.5% of colloidal substances (such as waxes, proteins, gums, starch, and silica), and 0.3 to 0.5% of suspended impurities (including soil and fine particles of bagasse during milling) (P. Rein, 2016). 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 possible sucrose in clarified juice (Meng et al., 2019).

The production of sugar from sugarcane consists of a series of units of operation, which begins with the extraction of the cane juice from the cane stalks and subsequent removal of the non-sugars (Chen & Chou, 1993). Sucrose is first extracted from the cane by the addition of water through counter current milling or diffusion. Extracted juice then undergoes an extensive process of clarification, which involves screening, heating, and addition of milk of lime (calcium hydroxide) to adjust pH and enhance flocculation. The limed juice is then left to settle out suspended particles in a Dorr clarifier (decanter). Following the juice cleaning process, the clear or clarified juice is then heated and evaporated to approximately 65% dry solids (DS) in a stepwise evaporation process (Hugot, 1986). The juice, which is now concentrated syrup, is then put through a final fractionation of sugars from non-sugars by crystallization in vacuum pans via evaporation and concentration and in an open crystallizers by cooling. Centrifuging affects the final separation of the sugar from the mother liquor, drying and cooling of the sugar was done, bagged, and the cane sugar is then stored for transportation and distribution to the customers(Hugot, 1986) [7]. For the sugar process to be profitable attention must be given to the quality of sugar produced, the overall recovery of sugar, and the use of products in molasses. Processes before evaporation are critical to achieving good sugar recovery and have a serious impact on the composition of final molasses. Of interest is the clarification process, which represents the unit of operation where major juice cleaning takes place, and where major non-sugar components are precipitated (lal Mathur, 1999).

1.2. Statement of the Problem

The aim of sugar industries is to be profitable with the required sugar quality by improving sucrose recovery or reducing sucrose losses. To improve the recovery of sucrose, juice clarification efficiency must be done properly and in a good manner. The uses of effective clarification are sucrose recovery, minimization of fouling effects, improvement of quality of the final product (sugar) and operational consistency in the boiling house which will be favorable with higher capacity. Usually mixed juice is transferred from an extraction plant that contains sucrose (pol) and impurities such as; reducing sugars, colloidal substances, insoluble matter, suspended impurities, etc. During the clarification process all impurities and coloring matter should be removed. The major problems related to the clarification process at the Wonji/shoa sugar factory are;

- ✓ Meeting quality standards for the final sugar product, especially color, which currently exceeds the standard set range for plantation white sugar (<100 IU). According to factory annual performance report 500-1500IU, ICUMSA color value produced. This far exceeds the <100 IU color requirement.

- ✓ Sucrose loss through filter cake and final molasses, which is related to clarification efficiency, low neutralization pH of juice leading to sucrose conversion. Wonji/shoa sugar factory case, sucrose loss in final molasses reaches 40-42%, which exceeds the standard 30-35% and sucrose loss on average of 3-4%% in wonji in the filter cake but the standard is 1-2% this is directly related with the inefficiency of clarification.

- ✓ Therefore this study is initiated to investigate and assessing the current performance of clarification plant during the crushing season and in regulating the optimum level of liming pH, sulphitation pH, and first and second juice heating temperature.

1.3. Objectives of the Study

1.3.1. General objective

The general objective of this research thesis is to investigate the existing juice clarification process and parameters of Wonji/shoa sugar factory for improving the juice clarification process that increases the quality of the product and reduce sugar losses.

1.3.2. Specific objectives

The specific objectives of this research are to:

- ✓ Investigate the effect of juice heating, liming and sulphitation on the mud settling and color level of clear juice at different pH and temperature.
- ✓ Study the effect of liming pH, sulphitation pH, first and second juice heating on the relationship among Mud settling, Turbidity, Color, and Purity of the clarification process with chosen process parameters
- ✓ Investigate the parametric interaction effect of process parameters, model and conduct parametric optimization using RSM and ANN couple with GA.

1.4 Scope of the Study

The scope of the research was restricted to the effect of juice treatment chemicals and juice heating temperature on the clear juice quality parameters such as; purity of clear juice, color of clear juice, settling rate of mud and turbidity of clear juice to reduce sucrose losses with filter cake and final molasses and minimize scale formation inside tubes of evaporators and heat exchangers.

1.4. Significance of the Research

This thesis study may increase awareness of the preparation and optimization of the consumable chemicals and identify the best heating juice temperature. The results obtained can be used as a reference for other future research in the area of sugar juice clarification. It may also be used to improve overall final commercial sugar quality, and reduce sucrose losses from acid inversion and thermal distraction due to high-temperature heating of the juice. By utilizing of the results of this study, the sugar factory can minimize economic benefits in case of consumable chemicals (Sulphur and lime) consumption and steam consumption by controlling the juice heating temperature at different heaters.

CHAPTER TWO

2. LITERATURE REVIEW

2.1. General overview of Wonji Shoa Sugar Factory

The factory is found in Oromiya regional state near Adama city at 110 kilometers from Addis Ababa. Wonji sugar factory started sugar production in 1954; it is the oldest and the pioneer in the history of Ethiopia's sugar industry. After some years Shoa Sugar Factory was constructed in 1962 as the second oldest. Both factories have stopped production since July, 2012 and July, 2013 respectively. The two factories were constructed by the Holland Company known as H.V.A and had a capacity of producing 750,000 quintals of sugar per year. The sugarcane plantation land of these two factories was 7,000 hectares out of which 1,000 had been planted by out growers.

Since it was required to replace these two oldest factories with a new and modern technology and also to increase capacity, an expansion project had been carried out both in the cane cultivation field and the factory since 2010. And the factory plant expansion project was coming into its completion in July, 2013.Accordingly, the newly built and modern WSSF has currently a design capacity of crushing 6,250 tons of cane per day (TCD) and producing 6,250 Quintals of sugar per day. In the same way to increase its financial earning from the sugar byproduct new ethanol plant is planned to be built, with a capacity of producing 12,800 meter cube ethanol as a project. Building of the ethanol plant is underway to be finalized 2022.

The Factory has the capacity to produce 32 megawatts (MW) of electricity. Its agricultural expansion project is currently being carried out around the areas known as Wakie Tiyo, Welenchiti and North Dodota areas. The factory with the help of this agricultural expansion project will have 16,000 hectares of sugarcane plantation field in total.

The total cane cultivation field of the factory has currently reached 12,800 hectares. And, the 7,000 hectares of the factory's sugar cane field cultivated with the agricultural expansion project are owned by out grower farmers of the surrounding area. The Factory, beyond supplying the farmers with selected seeds, and supporting professional as well as technical support to them, has made irrigable land available to all.

Figure 2.1: Wonji/Shoa Sugar Factory

2.2. Sugar Cane

Sugarcane is a grass grown in tropical and subtropical countries. It is a complex hybrid of various species, derived largely from Saccharium Officinarum and other Saccharum species (Eggleston et al., 2014). It is propagated by planting pieces of cane stalk. New growth emanates from buds on the cane nodes, ensuring uniform progeny. In the process of cane breeding, new varieties are produced and tested in a constant search for new and better varieties. This has been a major factor in improving productivity in the sugarcane industry (P. W. Rein, 2009). Yields of cane vary from one area to another, depending on cane variety, climatic condition, and availability of water, cane growing practices and the length of the growing season (Asfaw, 2015a). Cane yields may vary from 50 t/ha in poor conditions to values approaching 200 t/ha under exceptional conditions with long growing periods. Yield of sugar may vary from 5 t/ha to 25 t/ha. Cane is generally not replanted every time it is harvested, but is allowed to re-grow and produce another crop, called a ratoon or stubble crop (P. Rein, 2016). The yield declines after a number of ratoons and at some point the cane has to be ploughed out and replanted. Cane is generally harvested in the winter and the length of the crushing season is dictated by weather conditions, principally rain. In some countries such as Colombia, Peru and Hawaii, cane may be processed virtually all year round (Meng et al., 2019). The major reason for processing cane is to recover sugar, which in its pure state is referred to by its chemical name sucrose and has the formula $C_{12}H_{22}O_{11}$ and is termed a disaccharide. It is formed in plant by a complex process but essentially consists of a combination of the two monosaccharide sugars, fructose and glucose (Meng et al., 2019).

The reverse of this reaction is termed inversion when under the right conditions; sucrose sugars. The sugars are in solution in water together with other soluble components: this liquid phase is referred to as juice. Other than juice, the cane stalk contains about 13 g vegetable fiber as a solid phase/100 g cane, giving the stalk its rigidity (P. Rein, 2016). Will be hydrolyzed, and together with a molecule of water, to the formation of the two component

Table 2.1: Composition of mixed juice (P. W. Rein, 2009)

Parameters	**Values**
Brix	15^0-18^0
Purity	84-87%
Reducing sugar	0.4-1.0%
Ashes	0.4-0.7%
P_2O_5	250-300 mg/L
N_2	0.03-0.04%
pH	5.0 -5.5

2.3. The Production of Raw Sugar

The production of sugar from sugarcane consists of a series of units of operation (Chen & Chou, 1993). The first step was extraction of cane juice from the prepared cane by the addition of water through counter current milling or diffusion. Extracted juice called mixed juice (MJ), MJ then undergoes an extensive process of weighing stepwise heating, clarified juice from the clarifier pass through a stepwise evaporation process to evaporate the excess amount of water in mixed juice and bring the juice concentration up to 65 % dry solids (DS). The juice, which is now concentrated is called syrup, goes to a final fractionation of sugar from non-sugars by crystallization in vacuum pans through evaporation and concentration and in crystallizers by cooling. Centrifuging effects the final separation of the sugar from the mother liquor, and the cane sugar is then stored for transportation and distribution to the customers (Hugot, 1986). A schematic drawing and overview of the process is shown in Figure **2.2**.

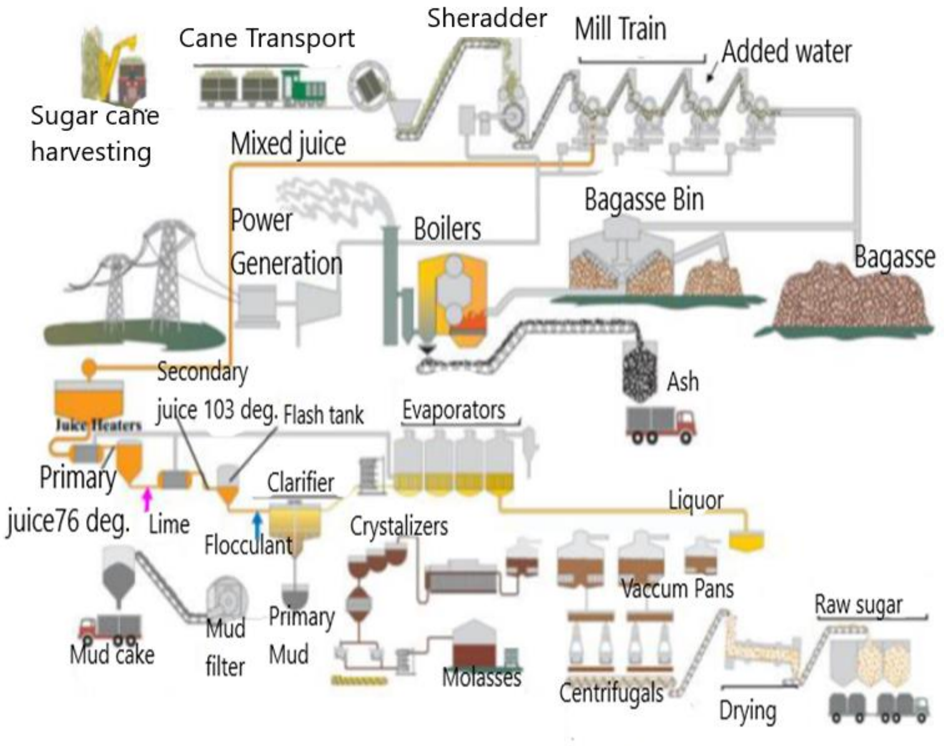

Figure 2.2: Manufacturing process for raw sugar (Hugot, 1986)**.**

2.4. Clarification as a unit operation

The sugar cane juice as expressed from the cane contains not only sucrose but also numerous other dissolved substances, as well as cellulose or woody fiber (lal Mathur, 1999). Sugar cane juice is an extremely complex liquid medium, containing many organic and inorganic constituents in soluble, suspended/decant able and suspended/colloidal form (Dionisi et al., 2008). The percentage of sugar (polarization) in the cane varies from 8 to 16% and depends on the variety of cane, its maturity, the condition of the soil, the climate and agricultural practices followed (Mathur, 1981).

Cane sugar for human consumption is produced by means of clarification of sugar cane juice. A critical step in the manufacture of any sugar (raw, plantation white, or refined) is clarification process. 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 the most important step to obtain high yields and high quality sugar (Dionisi et al., 2008).There are different clarification methods currently used, including cold liming, hot liming, fractional liming with double heating, liming while boiling, the Java method, and calcium saccharate (sucrate) treatment (Hugot, 1986).

Sulphur dioxide (SO_2) has been used for centuries to minimize color in food processing and fruit and vegetable storage. Sugarcane processors throughout the world use SO_2 to produce plantation white sugars (Andrews & Godshall, 2002). It consists of SO_2 absorption by the hot limed juice, reducing its basic pH to about 7.0. To improve floc formation, soluble phosphate is added to the juice. The soluble phosphate produces an insoluble calcium phosphate primary floc that traps other suspended impurities (Rundell & Pottage, 1977). In the cane sugar factory, the major role of sulfur dioxide has been to make white sugar rather than raw sugar through inhibition of color forming reactions including enzymatic browning reactions (Andrews & Godshall, 2002). However, periodic spikes in sulfur prices and sugar quality issues have stimulated efforts to reduce or even eliminate its use. With that in mind, a carbonation process has been tested and compared with standard sulphitation and defecation. Besides eliminating the use of sulfur, carbonation would also provide a means to utilize and take away some of the excess CO_2

The term clarification is defined as a process by which all insoluble and some soluble impurities are removed from the juice (Woreta, 2020). These impurities impart dark color to the juice and contain sucrose that may be available cost-free in some sugar factories from molasses or juice fermentation(M Saska et al., 2010). Among sugar cane processors worldwide, there is mixed interest in usage of sulfitation.

In Ethiopia, most of the sugar factories employ sulphitation process for the manufacture of plantation white sugar (PWS) but the newly built sugar factory this day has carbonation process instead of sulphitation. In the United States (US), sulfitation 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 color retardant as many US factories are considering the production of high quality low color raw sugar to be sold as a food grade sugar (Andrews & Godshall, 2002). To improve decolorization without using an excessive amount of lime that would raise scale problems, phosphatation can be supported by a flocculating agent (Cartier et al., 1997). Rapid flocculation and sedimentation of suspended particles in primary cane sugar juice is achieved using a high molecular weight anionic polymer flocculent (William O.S. Doherty et al., 2003).

Flocculation is the agglomeration of fine particles suspended in solution in to a flocculent mass (Chen & Chou, 1993). Flocculation removes insoluble suspended particles and colloidal impurities by aggregating them into flocs. A typical size range for pre-flocculation impurities in mill-extracted mixed juices at Louisiana raw sugar factories is 0.1 - 22 microns (Dionisi et al., 2008). In many cane sugar producing countries, the standard sugar product has a very low color in the range 80 to 250 IU. This sugar which is used in Ethiopia is commonly referred to as plantation white sugar (PWS). On the other hand, the standard for white (refined) sugar is considerably higher, requiring a color below 45 IU for EU No 2 or even 22.5 IU for grade 1 white sugar (P. W. Rein, 2009). The methods employed to produce PWS usually involve sulphitation of the juice and/or the Syrup (Steindl & Doherty, 2005). Plantation white sugar has been produced at raw sugar mills for many years without refining or re-crystallization (P. W. Rein, 2009).

2.5. Objective of clarification

The term clarification is defined as a process by which all insoluble and some soluble impurities are removed from the juice. These impurities impart dark color to the juice and contain sucrose, reducing sugars, organic and inorganic matter in the solution. It also contains insoluble matter in suspension such as bagacillo/fine bagasse and soil (Asfaw, 2015a).

The main objective of clarification of mixed juice is as follows (P. W. Rein, 2009):

• Removal of maximum non sugars

• Elimination of suspended and colloidal impurities

• Removal of color forming compounds

• To obtain brilliant, light colored, transparent clear juice free from any suspended impurities

To obtain high quality clarified juices, 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 SiO_2), and sesquioxides can be removed by heating, while liming neutralizes acids, forms calcium phosphates, and coagulates colloidal particles. Colloids are known to be responsible for higher or lower turbidity of sugar products and to have an adverse effect on the entire process (Demsash, 2021). Cane juice is a poly dispersed irreversible colloidal suspension in which the suspended particles carry a large negative charge (Whayman & Crees, 1974). According to established theory of colloid stability, small particles are attracted one another by Van der Walls forces and therefore always tend to aggregate together but they are kept apart by electrostatic forces arising from their electrical charges. The measures of these repellent force is called Zeta potential and has been defined as a measure of net electrical potential (in millivolts) carried by particles in size ranges of 10 A° to 10 μ m. The aggregation process is aided by the addition of simple electrolytes like $Ca(OH)_2$ to the juice. The negatively charged colloidal calcium phosphate is formed by liming. This interaction results in coagulation and sedimentation of the particles (Asfaw, 2015b). Generally removal of non-sugar impurities helps in the process for better crystallization of sugar, for the production of white sugar free from complications and bringing down the final molasses purity which helps for achieving less sugar loss in final molasses.

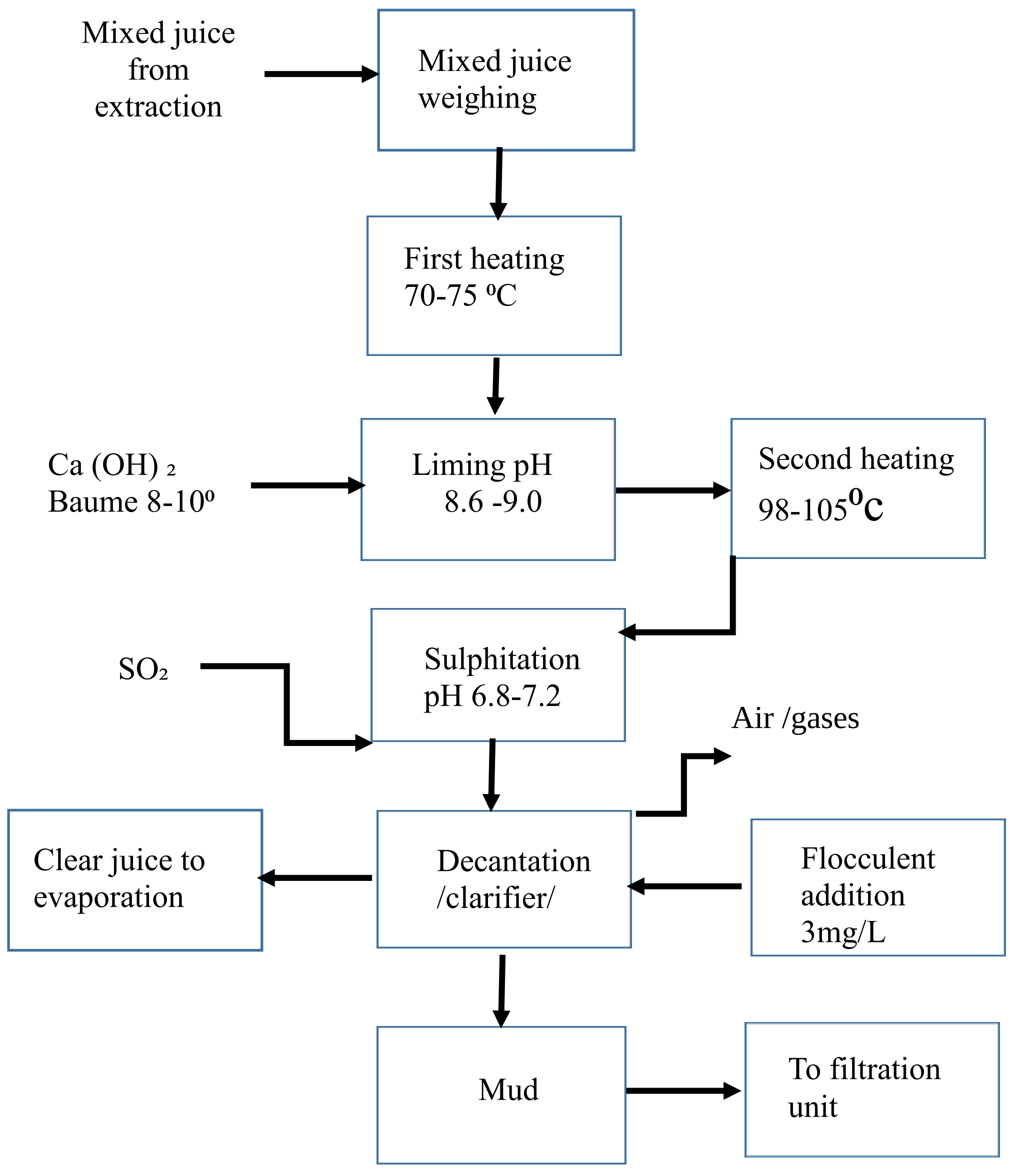

Figure 2. 3: Juice clarification process.

2.6. Juice Clarification methods

Depending on the type of final product to be produced, there are different methods of juice clarification. Such as, defecation, sulphitation and carbonation processes which are described here under accordingly (Yu et al., 2013).

2.6.1. Defecation process

Clarification by heat and lime is known as a defecation process. Sufficient lime is added to neutralize the organic acids present in the juice, after which the temperature is raised to 100 ^{0}C (Rukkumani et al., 2014). The treatment causes formation of heavy precipitate of complex composition. The flocculent precipitate carries with it most of the finely suspended material of the juice that has escaped mechanical screening. A juice clarifier does the separation of this precipitate from the juice. The defecation process is used usually in the manufacture of raw sugar (Nemera, 2020). Lime is used in the clarification process as calcium oxide (CaO). It is obtained from high grade lime stone, calcium carbonate ($CaCO_3$). On burning lime stone in kiln lime is burnt at 1200– 1300 ^{O}C and dissociated into quicklime and carbon dioxide

$$CaCO_3 \rightarrow CaO + CO_2$$

Here $CaCO_3$ dissociation will yield 56% CaO. The reaction is endothermic, so heat is absorbed being 42.5 kCal/ mole. Properly burnt lime is highly porous, light in weight and slakes quickly to form calcium hydroxide.

$$CaO + H_2O \rightarrow Ca(OH)_2 + Heat\ (15.2Kcal)$$

It is a hydroxide of divalent metal: Ionization takes place in two steps.

1st step $$Ca(OH)_2 \rightarrow Ca + OH + OH^-$$

2nd step $$Ca + OH^- \leftrightarrows Ca^{++} + OH^-$$

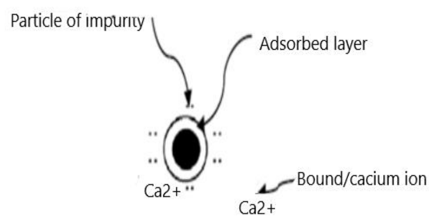

Figure 2.4: the raw juice particle (Thai, 2013).

The chemical activity of the compound depends upon the effective concentration or activity of the Ca^{2+} and the OH^- ions; Although Ca^+ OH ions may take part in the reactions also. Because the secondary ionization of calcium hydroxide is small, the concentration of calcium ion is low (Thai, 2013).

2.6.1.1. Quality of lime

Lime (CaO) is the basic chemical in the clarification processes. Lime neutralizes the acidity of the juice and also reacts with phosphoric acid in the juice to produce a precipitate. Source of calcium carbonate is mostly the lime stone quarries or the coral deposits. $CaCO_3$ content of commercial limestone varies from 85-95%, the other impurities being silica, iron alumina, magnesia, sulphates and alkalies should be less than 2%.The maximum impurities allowable are listed in **Table 2. 2**.

Table 2. 2: Composition of a good quality quick lime (Hugot, 1986).

Compositions	**Percentage (%)**
Lime (CaO)	85 – 95
Silica (SiO_2)	1.5
Iron oxide (Fe2O3)	< 2
Aluminum Oxide ($A_{l2}O_3$)	< 2
Magnesium Oxide (MgO)	1.0
Sulphate	0.5

2.6.1.2. Milk of lime preparation

Usually milk of lime is made up first by mixing (slaking) the pulverized lime or lump quick lime with water. Kulkarni (1993) describes the slaker as a mild steel drum revolving at about 6 to 8 per min with internal steel strips forming a spiral which moves the lump lime from the feed end to the discharge end of the drum. The out let of the tank must be supplied with a sieve with 1.3 mm openings in order to separate foreign matter (girts) present in the milk of lime. Milk of lime is a suspension not a solution and because of that, it has to be kept in constant motion (must has stirrer) to prevent deposition of lime in piping. In general milk of lime is pumped to different consumption points with the excess recirculate to the tank (George & Kyatanavar, 2014).

The concentration of milk of lime is measured in degree Baume (°Be). For juice clarification in sugar factories milk of lime is prepared at a concentration of 8 to 10 °be. The quality of the water used for preparation of milk of lime is important as untreated water increases risk of incrustation in evaporators it is recommended to use hot condensate (60-70 ^{0}C) from juice heaters and the evaporator (North-Coombes & Taylor, 1981).

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The following are the effect high impurity in lime on operation (P. Rein, 2016).

- 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 color of the juice. Both iron and aluminum salts cause scaling on tubes

Table 2. 3 :Concentration of milk of lime (P. Rein, 2016).

Brix	**°Be (Degree Baume')**	**Density (g/cm^3)**	**CaO (g/L)**
1.8	1	1.007	7.5
3.6	2	1.014	16.5
5.4	3	1.022	26.0
7.2	4	1.029	36.0
9.0	5	1.037	46.0
10.18	6	1.045	56.0
12.6	7	1.052	65.0
14.4	8	1.062	75.0

2.6.2. Sulphitation process

In the sulphitation system, clarification is done by heat, lime and sulphur dioxide gas. The mixed juice is heated to 75°C, more lime slurry is added to the raw juice and the excess of lime is neutralized by SO_2 gas. The treated juice is heated to 100°C and separation of flocculated non -sugars takes place in a continuous clarifier. From the settler, the clear juice continues in the process and the underflow mud is a filtered on continuous drum filter and the filtrate is recycled to the measured raw juice. Sugarcane processors throughout the world use SO_2 to produce plantation white sugars (Godshall, 1999). It consists of SO_2 absorption by the hot limed juice, reducing its basic pH. In the cane sugar factory, the major role of sulfur dioxide has been to make white sugar rather than raw sugar through inhibition of color forming reactions including enzymatic browning reactions (Godshall, 1999). However, periodic spikes in sulfur prices and sugar quality issues have stimulated efforts to reduce or even eliminate its use.

Sulphur

Sulphur is one of clarifying reagent in sugar factories including Wonji/Shoa, which apply the sulphitation process. The Sulphur is purchased in a very pure state and melted in Sulphur melter using stream of pressure of 7 kg/cm^2 and temperature of 250-270 ^{0}C then pumped the molten (liquid) to burner called furnace and burnt with the oxygen in a stream of air as shown in the following formula. The air is moisture free as much as possible must be dry.

$$S + O_2 \rightarrow SO_2 + heat(70\ kcal/32\ g)$$

Purity of sulphur should be more than 99% and free from arsenic and bituminous.

Action of SO_2 on juice clarification

SO_2 neutralizes the excess quantity of lime added and precipitates calcium sulphite.

$$Ca(OH)_2 + H_2SO_3 \rightarrow CaSO_3 + H_2O$$

If more SO_2 gas is passed and the juice is made slightly acidic, part of calcium sulphite is converted into soluble calcium bisulphite, which is undesired.

$$CaSO_3 + H_2SO_3 \rightarrow Ca(HSO_3)_2$$

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

$$2H_2SO_3 + 2Ca(OH)_2 \rightarrow CaSO_2 + 4H_2O$$

If highly acidic juice containing the soluble calcium bisulphite in solution is boiled, it again decomposes into precipitated calcium sulphate and Sulphur dioxide (P. Rein, 2016).

$$2Ca(SHO_2) \rightarrow 2CaSO_3 + SO_2$$

Sulphur dioxide bleaches juice by acting on the coloring matter. Sulphurous acid is a strong bleaching agent. It bleaches the coloring matter originally present in the cane juice. It prevents or slows down the color formation in later stages of the processing. It reduces the ferric salts which are highly colored compounds formed by the action of iron of the equipment with polyphenols into colorless ferrous compounds. Sulphur dioxide gas decreases the viscosity of the juices. The lower the viscosity of juice, the easier is crystallization (P. Rein, 2016).

Advantage of sulphitation

The advantages of sulphitation are presented as follows (Hugot, 1986):

- ✓ The mud settles more rapidly and hence improved capacity of clarifiers
- ✓ The mixture of sugar crystals and molasses before centrifuging (massecuites) are less viscous and boil faster
- ✓ Better crystallization in consequence
- ✓ There is a marked improvement in the color of the sugar, these appears to be a significantly reduced transfer of color from the mother liquor into the crystal
- ✓ There is a slight gain in the capacity of centrifugal
- ✓ Better elimination of phosphates and waxes leading to better refining quality and filterability of the sugar produced

Disadvantages of sulphitation

Kulkarni reported the demerits of sulphitation process that the use of the sulfur burner is difficult as it causes the toxic SO_2 to be released into the atmosphere, leading to serious environmental pollution. It can also create health problems for the workers handling the sulfur burners. The low color sugar and good luster obtained from syrup sulphitation is a temporary one.

Syrup sulphitation end up significant amounts of sulfur and hence it affects the fermentation of the molasses by inhibiting the growth of the yeast. Moreover, traces of the sulfur that gets into the alcohol make it unsuitable for use as a fuel supplement, liquor, and pharmaceutical industries, unless further refinements are carried out (Kahsay & Gabbiye, 2015). Thus the use of SO_2 in syrup does not provide any significant benefit. Further, whatever benefit does result lasts only for a short period of time. This realization prompted the development of an alternative technology that would avoid at least syrup sulphitation and help in producing good quality sugar. The use of appropriate biocides in proper doses limits the microbial degradation of mixed juice and the formation of degradation products like polysaccharide. Further, it decreases lime consumption, the color of juices, and residual calcium salts in clear juice (Kulkarni, 2004) . Investigations have demonstrated that syrup sulphitation can be successfully replaced by syrup clarification to achieve improvements in plantation white sugar quality.

One option to replace the second sulfitation process is to use syrup clarification based on the phosphitation process. The phosphitation process can reduce color compounds that are produced during the factory processes. Such color and color precursor groups include melanoidins, caramels and alkaline degradation products of fructose and glucose (Steindl & Doherty, 2005).

2.6.3. Carbonation

The main clarifying agents in the carbonation process are lime and CO_2 gas. The process consists of precipitating calcium carbonate (Steindl & Doherty, 2005).

$$Ca(OH)_2 + CO_3 \rightarrow CaCO_3 + H_2O$$

The precipitate (Calcium Carbonate) adsorbs coloring matter and gums. It is easily filterable. The raw juice is heated to a temperature 50–55 °C and pumped into the carbonation tank where lime is added & mixed thoroughly. The juice is neutralized with CO_2 gas. The quantity of lime used in carbonation process is approximately ten times more than the quantity used in sulphitiation process. Consequently, its working cost, labor employed etc. is higher (Kulkarni, 2004).

The soluble impurities that are formed during the process depend on the harvesting practice, transport conditions, environmental factors and the chemicals/technology used during the process (W O S Doherty et al., 2002). These soluble and insoluble impurities must be removed by the technique called clarification. Separation of impurities from the mixed juice by clarification avoids hindrance of sucrose crystallization and minimizes sugar loss in final molasses. Hundreds of materials have been tried for juice purification.

The advantages of carbonation process over sulphitation are (Kulkarni, 2004);

- Superior, lighter colored clear juice with less viscosity is obtained
- More non-sugars are removed in which recovery of sugar is higher by about 2%
- CO_2 gas is much less corrosive than SO_2 gas and the damage done to the plant is light
- The CaO content in clarified juice is much less and consequently the formation of scales in the heating and evaporators tubes is much less

2.6.4. Phosphatation process

The successful development of the phosphatation process, leads to the addition of phosphoric acid and lime to hot juice leads to the production of flocculated calcium phosphate. In sugarcane, the natural phosphates are occurring in two principal forms;

inorganic (soluble) and organic (insoluble) phosphates. Only the soluble phosphate will react with the lime to form a precipitate. It has been shown that the greater the amount of phosphoric acid in the juice, the easier is the clarification (Hugot, 1986). The accumulated evidence regarding phosphate in juice indicates that the phosphate content is the most important factor in efficient clarification (Mohammed et al., 2016). In sugarcane, the phosphates are occurring in two principal forms; inorganic (soluble) and organic phosphates. The organic phosphate exists in the form of phospholipids, phosphor proteins, nucleotide phosphates and hexose phosphates. Only the inorganic (soluble) phosphate takes part in juice clarification. Therefore, juices with adequate quantity of inorganic phosphate are desirable (Chen & Chou, 1993). Juice of lower natural P_2O_5 content (about 200 mg/L) is difficult to clarify; phosphate may have to be added externally before liming (Mathur, 1981). The presence of phosphates in cane juice is essential for good clarification. The phosphatation process is not only removes the colloidal and other impurities but also absorbs much of the coloring matter and diminishes the calcium content of the clear juice. It has been found that the recommended amount of P_2O_5 is 300 mg/L of mixed juice. If the cane is grossly deficient in natural phosphate, it is very difficult to clarify and hence external addition of phosphate in the form of soluble phosphate is needed to compensate the deficiency (Mathur, 1981). It is preferable to add the phosphate to the juice in a suitable vessel, such as stirred tank, where it can be retained with mixing for about 2 minutes for complete precipitation

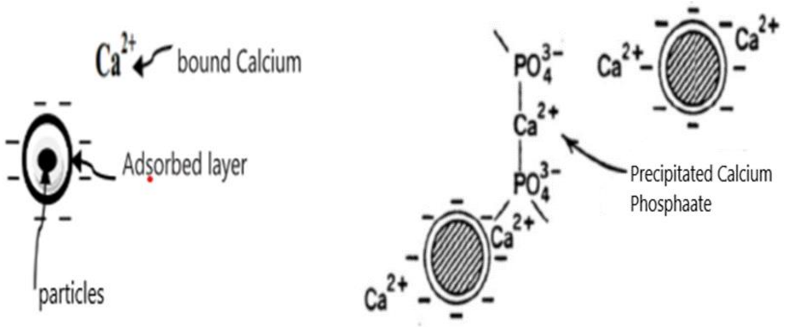


Figure 2.5: Calcium phosphate bridging (Thai, 2013)**.**

2.7. Chemical Reactions of Clarification

The chemical reactions that occur during cane juice clarification are not fully understood. It is fundamental however that those reactions with lower free energy content would take place preferentially, with reactions requiring lower energy completing first. 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 25 °C, but it is greatly increased in a solution of sucrose. The solubility however decreases with an increase in temperature. Calcium hydroxide being a relatively strong base of a divalent metal ionizes in solution to give $CaOH^+$ and OH^-. The secondary ionization is low, with less than 10 % of calcium as Ca^{++} in sucrose solution containing 0.3-0.5% calcium oxide (Honig, 1963). The reaction of lime and phosphate in juice precipitate 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 has been shown to be very low and the precipitate formed in two forms, tri-calcium phosphate $Ca_3(PO_4)_2$ and calcium hydrogen phosphate $CaHPO_4$. The formation of calcium hydrogen phosphate has been shown to have a faster reaction rate, and as it approaches completion the rate of the formation of tri-calcium phosphate increases. The rates of both reactions are greatly increased at high temperatures and result in an increase in the hydrogen ion (H^+) concentration, which has been suggested as a contributor to the drop in the pH observed when juice is heated. Approximately 80-90% of the phosphate present in juice is precipitated with lime. It was shown that at a pH of about 8.5, about 99% of the phosphate present in juice is precipitated (Simpson, 1996). Other calcium salts are precipitated in clarification; these may include sulfate, aconitate, and oxalate, tartrate, and citrate salts. These salts are normally precipitated in small quantities, but variations do occur depending on the quality of the juice. The sulfate content of cane juice is highly variable and approximately 20% is removed by liming to pH in the range 7-8 (Honig, 1963). The organic acids in juice are responsible for the natural pH of the juice (5.2-5.4) as well as the buffering capacity of the juice, which is mainly due to the juice aconitic acid content. The presence of other organic acids such as lactic, formic, acetic, propionic, and butyric acids are generally a result of microbial or chemical decomposition of sugars (M Saska et al., 2010). Another component of cane juice is silica. Silica is present in the juice in the form of dissolved silica, colloidal or suspended silica or as silicates. On average it is 0.42% of solids in mixed juice. Most of the suspended silica is removed by liming (Martin Saska et al., 2010).

2.8. Effects of Time in Clarification

The retention time of juice in the clarifiers has a great effect on the juice and its components. 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 a longer period of time. However excessive capacity clarifiers 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 juice or the reactions of the defecant in the juice may be slower and require more time than other methods (Nemera, 2020).

2.9. Effects of Temperature in Clarification

The effect of temperature in juice clarification is multifaceted as it not only prohibits microbial activity but also accelerates 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 although intermediate liming is sometimes practiced (liming at juice temperature of 72-76 °C). Cold liming is the continuous addition of lime to lime tanks as water slurry at ambient temperature. The limed juice is then heated to about 105 °C where it is flashed and then goes into the clarifier for settling. In hot juice liming the lime slurry is added when the juice is at the heated juice temperature of 103 °C – 105 ºC after which it goes on to settling in the clarifiers. Investigations carried out into the effects of cold versus hot liming done in raw sugar factories, indicated that hot liming was more favorable as it removed more color, dextran, oligosaccharides and produced clarified juice with lower calcium levels, but results in higher mud levels and a slower floc settling rate (Eggleston et al., 2002). Doherty et al (2003) confirmed some of the findings by Eggleston regarding mud volumes and settling rate of juice clarified by hot liming, but points to the main advantage of hot liming as being a reduction of scale formation in juice heaters and evaporators as a result of increased calcium removal. There is still ongoing debate about which liming technique is most effective since the literature has failed to examine the underlying chemistry of the clarification process, and there are speculations about the validity of results presented on the efficacy of the different liming methods.

2.10. Role of pH in Juice Clarification

The general relationship between pH and the rate of inversion of sucrose is well known; the more acid the solution the faster sucrose decomposes. Even if it was for this reason only, it would be one of the most important duties of every chief of fabrication to keep a close watch on the pH values of all the liquors in the factory (George & Kyatanavar, 2014). The pH, however, does not only affect the rate of sucrose inversion, it is also of primary importance in all chemical reactions which together are known as the clarification of the juices, and finally it has an important bearing on the rate of decomposition of the reducing sugars. It is only by giving the fullest attention to the pH values in the factory (and incidentally also to temperatures) that a chief of fabrication can hope to attain maximum clarification effect and a minimum loss of sucrose (Panpae et al., 2008). The pH measurement, or better pH recording, at a few strategically selected points in the factory is therefore a must wherever the highest efficiency is strived for. In most cases, that pH recording is not general practice in all factories, and that the factories endeavoring to operate pH recorders frequently run into difficulties (Nemera, 2020). Electrical pH meters and recorders have not a very good name in sugar factories; the factories think that they are expensive gadgets, and the operating staffs are often extremely doubtful about the practicability of operating them satisfactorily, i.e. with little interruption and with reliability. In sulphitation factories the pH of the juice is intentionally altered in three places, i.e. in the preliming tanks, in the sulphur tower and table P^H meter for operators for controlling of them. The exact pH to which the juice has to be tempered depends on two kinds of factors. In the first place this pH should be such that a maximum clarification effect is achieved, but on the other hand we should not forget that the loss of sucrose through decomposition during boiling, crystallization, etc., depends to a great extent on the pH of the syrup (Scott et al., 1978).

In a sulphitation factory considers introducing or extending pH registration, the correct places, in order of urgency, are (P. W. Rein, 2009).

• The tempered juice as pumped to the heaters;

• Pre-limed juice as pumped into the sulphur tower;

• Sulphured juice as emerging from the sulphur tower;

• Syrup as pumped to the pan stage;

• Clarified juice as pumped into the evaporator

Rein (2002) outlined the importance of pH in clarification, indicating that a pH of about 7 is necessary to neutralize the charge on the fine suspended particle 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 processes. Studies conducted by Saska (2002) also indicated that sucrose loss as a percentage of total sucrose per hour in clarifiers increase as the pH increases, and range from 3% at a pH of 5 to 0.1% at a pH of 6.5.

2.11. The Flocculants

A number of synthetic flocculating agents have come into use in the last two decades. These flocculants are synthetic high molecular weight polyacrylamides which are partially hydrolyzed (Chen & Chou, 1993). Flocculants form a bridge between two or more particles uniting the solid particles into a loose porous state. A high molecular weight flocculants are used at clarification, resulting in a fast bridging of very small particles present in the juice, whilst lower molecular weight flocculants are recommended at the filtration of mud where flocks are small and have a higher resistance to shear (Hugot, 1986). Their efficacy as settling aids depends on the molecular weight, which ranges between 7 to 10 million and the degree of hydrolysis. These compounds can be anionic, cationic or nonionic, and serve as bridges among particles of precipitate and thus bring about the formation of bigger aggregates of flocs. They are added at 0.05 to 0.1% solutions in water at the rate of 1 to 3 parts per million parts of juice, to the treated juice in the pipe connecting flash tank to the clarifier (Ansari et al., 2013).

A number of these polyacrylamides have come into the market and their use has become absolutely essential when dealing with refractory juices or during periods of difficult settling. Moreover, their regular use in the process with the installation of tray less clarifier has reduced the time of juice retention to 15 to 60 minutes. For improving the settling rate and increasing the clarifier throughput, the addition of synthetic flocculent is essential. Thus, the introduction of synthetic polyacrylamide as settling aids has been a significant advance in the process of clarification (Mohammed et al., 2016).

- Flocculants are materials of high molecular weight such as polyelectrolytes.
- They are synthetic water soluble, polyacrylamide powders.
- Most suspensions in lime treated cane juice contain negatively charged particles. Therefore, cationic flocculants are expected to be the most suitable.

Good flocculants improves (Hugot, 1986; Nemera, 2020):

• Flocculation by producing secondary flocculation.

• Increases mud settling rate.

• Reduces mud volume due to mud density increment.

• Decreases sugar loss in the filter cake, and improves the clarity of the clarified juice.

2.11.1. Preparation and application of flocculants

Flocculent must be free flowing powder and any agglomerate must be discarded. The flocculent is moistened with some alcohol to keep granules loose (Mohammed et al., 2016). The suspension is then added sparingly to warm water (50 to 60 ^{0}C) supplied to the preparation tank until the required volume is obtained. Continuous bubbling of hot air through the viscous solution is recommended to dissolve the granules without damaging the flocculent molecules. Two preparation tanks are essential. A metering pump is essential to control the flow rate of the solution to obtain the required dose. One tank is used for supplying the flocculent to the juice while the second is used for holding the dilute solution. The point of application of a flocculent solution of the treated juice is very important. The application point should be the 1st point that flocs once formed are not broken. This is expected to be at the inlet of the clarifier [7,31]

Separan AP 30 polymer is the type of flocculent suitable for most of Ethiopian sugar factory including Wonji-Shoa that will give consistent performance as checked by Ethiopian sugar research and development center (Kassa, 2010).

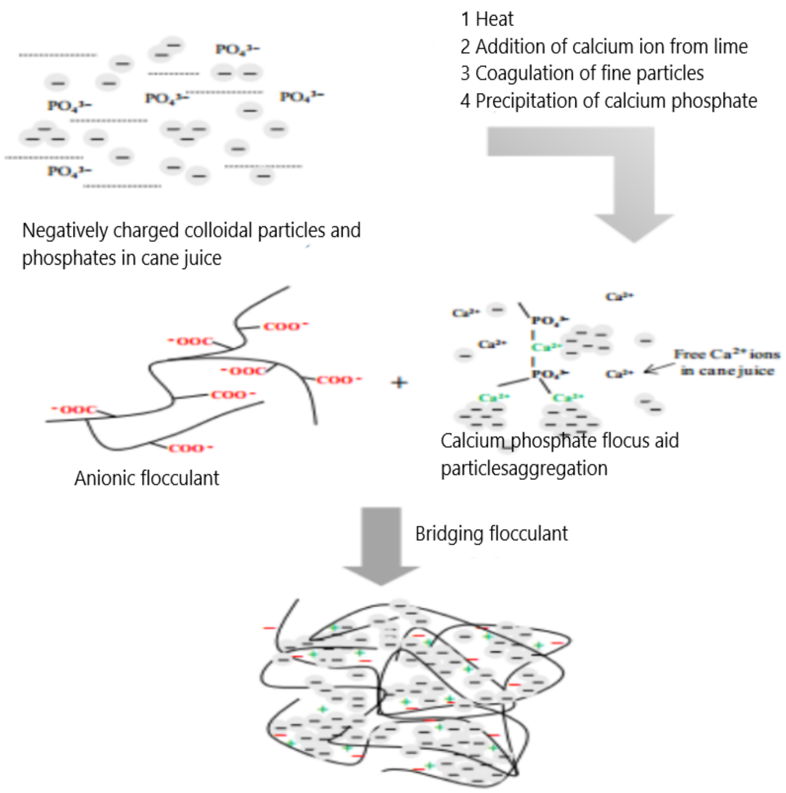


Figure 2.6: Interaction and coagulation process (Thai, 2013).

2.12. Color and Turbidity

The goal of any production process is to produce as large a quantity of product within the quality criteria. One of the most important criteria in the sugar industry is the color of the sugar. The removal of color in the recovery of sugar from cane is very important to get a high quality white sugar in addition to color removal. This color removal is expensive, complicated, and since there is loss of sucrose, understanding of the origin and chemistry of these colored compounds might contribute to their ease of removal [3, 34]

The Millard reactions occur throughout the factory and have many complex pathways (van der Poel et al., 1998). They proceed under almost all conditions, as reducing sugars and amines or amino acids are always present except in the purest of solutions. Iron also plays an important role, particularly in plant-derived colorants (Godshall, 1999). Many polyphenolic compounds found in cane juice are able to produce highly colored iron complexes. It must be noted that just as important as the colorants themselves are the compounds that are color precursors. These, often colorless, compounds can react to form highly colored species.

A lower turbidity figure is the best indicator of efficient clarification... The term turbidity is defined as the difference between the ICUMSA colors of the filtered and unfiltered solutions [42, 43] . Turbidity in solutions is caused by the presence of finely suspended matter (Goff, 2007). The most important criteria for evaluating clarification is juice clarity. Clarity is achieved by the efficient removal of turbidity (Chen & Chou, 1993). Measurement of turbidity of a solution is done usually using a turbid meter. Turbid meter is an instrument that records the loss of intensity of a light beam passed through a solution containing suspended particles (Goff, 2007).

2.13. The Juice Settling Process

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 (TESFAYE, 2020). The precipitate is falling down against rising streams if juice going to the overflow point. Obviously, for effective separation, the settling rate must be faster than all of the up-flow streams of juice so that the net direction of particle movement is downward. If juice is drawn from only one compartment of the clarifier, a very high upward velocity of juice exist which restricts the falling rate of the precipitate or particles entertainment to the overflow clear juice occurs (Hugot, 1986). The Dorr clarifier has a settling tray, which is 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 up flow velocity dividing it by the number of settling trays (Honig, 1963).The settling rate of the particles depends on the size and density of the particles as well as density and viscosity of the juice. This is expressed by stokes' Law.

$$V = \frac{D^2(d_1 - d_2)g}{18u} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2.12.1)}$$

Where; V - Falling velocity of the particle (m/sec),

D - Diameter of the particles (cm),

d_1 - Density of the solid particle (g/cm^3),

d_2 - Density of the liquid (g/cm^3),

g - Gravity constant cm/sec^2, and

u - Viscosity of the liquid (poises).

The above equation is for a particle of spherical shape; where the particles are not spherical, the constant is no longer 1/18 and the equation becomes:

$$V = \frac{KD^2(d_1 - d_2)}{u} \quad \text{……………………………… (2.12.2)}$$

Where:

K - Gravity constant as well as particle shape constant

Density of particle (d1) – Accurate density of the particle is difficult to obtain. However, approximate figures are: -

- ✓ Inorganic matter – $Ca_3(PO_4)_2$,
- ✓ Soil particles, $d_1 = 2.2 – 3.0$ g/cm^3.
- ✓ Organic matter $d_1 = 1.1$ (close to the juice)
- ✓ Fats and waxes, $d_1 < 1.0$

The average density of settling particles ≅ 1.5 g/cm^3.

2.13.1. The juice clarifier

In the clarification, precipitate is formed by the clarifying agents added. The separation of this precipitate or mud is done in a specially designed tank called the clarifier (P. Rein, 2016). The precipitate settles at the bottom and the clear juice is 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 is not properly scraped away will crust over rapidly and cool in temperature that permits bacterial action and souring, resulting sugar loss (William O.S. Doherty et al., 2003).

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 (Gravois et al., 2014).

Clarifiers (settlers or thickeners) work based on gravity sedimentation. The sedimentation (settling) process is also called clarification because it produces clarified liquid. The majority of sugar factories use continuous clarifiers to separate solids from sulphites juice. The overflow from the clarifier is called clarified juice, and the underflow is mud. Because of settling, about 75% of the sulphited juice is converted into clarified juice (clarifier's overflow), which is nearly free of suspended solids and 25% becomes mud (clarifier's underflow). The volume ratio of the mud to the slurry is called thickening ratio (Thai, 2013).

2.13.1.1. The flash tank

The treated juice fed to the clarifier must be at a constant temperature to avoid formation of convection currents and get rid of air and gas bubbles. To obtain these conditions, treated juice is heated to slightly above the boiling point (98 to 103 ^{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 the 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 the 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 thereby removed; otherwise, they would prevent the settling (TESFAYE, 2020).

2.13.1.2. The Dorr clarifier

The flashed juice is directly fed into the clarifier/decanter there are different types of clarifier of these the one found in wonji/shao sugar factory is Rapi Dorr 444 clarifier. The model consist of four compartments with separate provisions for feed, overflow take-off and mud withdrawal, allowing the unit to operate as four totally independent clarifiers enclosed in a common housing (North-Coombes & Taylor, 1981). Juice is 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 is 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 juice is taken off uniformly to eliminate stagnant pockets (Panpae et al., 2008).

The clear juice from each compartment is 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 can also be taken off by means of hydrostatic head. However, since muddy juice is too thick and heavier than clear juice Doroco pumps are provided to take off the muddy juice. Each compartment is provided with a clear juice tube, muddy juice tube, deairating tube, liquidation pipes, test cocks etc. The juice retention in this clarifier is 2 to 3 hours (Asfaw, 2015b).

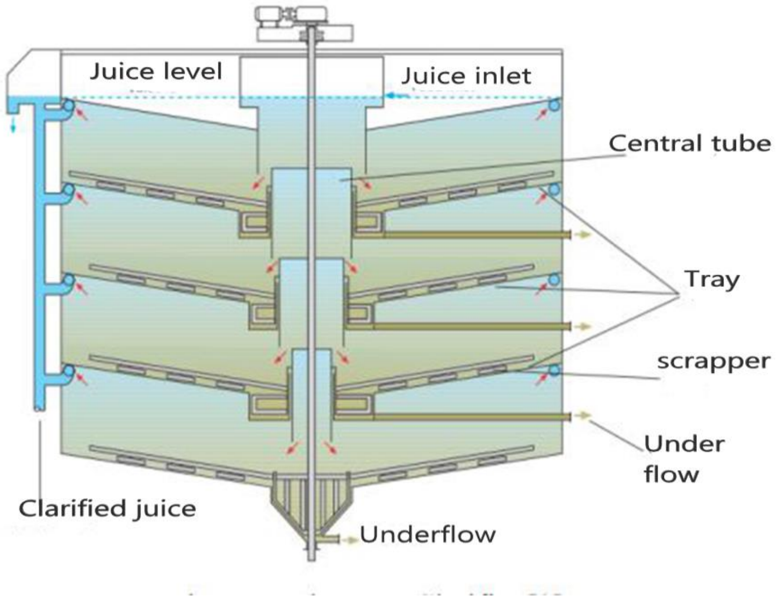


Figure 2.7: Rapi Dorr 444 clarifier (North-Coombes & Taylor, 1981)**.**

Table 2.4: Standard operational parameters of clarified juice (SOP) (Hundi, 2010)**.**

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

CHAPTER THREE

3. MATERIALS AND METHODS

3.1. Materials

The optimization of the main clarifier reagents and juice heating temperature experimental work was carried out in the clarification unit around the main sulphur header of the factory at pilot scale to provide SO_2 gas to the reaction vessel using modified pipeline where as the evaluation of the response variables was done in the factory laboratory service. **Table 3.1** Shows materials and equipment's used for experimental and laboratory work, as indicated in **Figure 3.1**.

Table 3.1: Material and equipment Used in Experiment/Laboratory Work

No	Equipment/ Instrument	Model	Supplier
1	Juice and chemical weighing scale	Sample of juice and chemicals	To carried out the experiment
2	Lab scale reactor	Stainless steel kit with stirrer with adjustable RPM	From Ethiopian metal work
3	Heaters	RCT basic	Japan
4	Screener	100-120 µm pore size screen	Germany
5	Test tubes	1000 mL, 10 mL and 50 mL	Indian company
6	Filter paper	Whatman 0.45µm pore size	Germany
7	pH meter	HANNA pH meter	To read pH
8	Spectrophotometer	CECIL CE1011	Indian company
9	Refractometer	Rudolph Automatic	Indian company
10	Saccharimeter	OIML Accuracy class 0.05	Indian
11	Measuring instruments	Different glass wares	Indian

No	Chemicals	Purity	Supplier
1	CaO	90%	Derba lime and chemicals PLC and Batu Caustic soda factory (Zeway)
2	Sulphur	99.9%	ZHEN ZE ENTERIPRIAN (HONK KONG) Limited
3	H_2SO_4	98%	Awash Melkassa Aluminium Sulphate & Sulphuric Acid S.C
4	NaOH	98%	Sharjah, United Arab Emirates
5	Water	99.9%	Made in the factory
6	Basic lead acetate	99%	ALPHA Chemika (INDIA)

3.2. Sample Preparation

After the milling operation, mixed juice was collected in a mixed juice tank. Then five liters of mixed juice was taken using a measuring flask which was then screened to remove colloidal substance and poured into a stainless steel kit followed by heating as shown in **Figure 3.1**. The kit had a stirrer and a provision of sulfur dioxide gas which had a narrow pipeline elongated from the main header of SO_2 gas from the factory. Before treatment of the juice, the phosphate content of the juice was analyzed and found for all samples within the standard from 250-300 ppm and mixed juice composition were analyzed and found with the standard like brix, pol, and pH for all samples. Then the juice was heated at 60 ^{0}C, 70 ^{0}C and 80 ^{0}C to observe the effect of first juice heating. Then milk of lime (MOL) was added at pH of 8.1, 9.0 and 9.9 to determine the effect of liming followed by injection of SO_2 gas at pH of 6.2, 7.0 and 7.8 and then heated at 90 ^{0}C, 105 ^{0}C and 120 ^{0}C to observe the effect of second heating then flocculent was added. Addition of 3 mg/L flocculants (as per the factory norm) therefore 15 mg of flocculent was added for five (5) litters. The hot treated juice was flashed to remove dissolved air. Afterwards, the juice samples were taken and placed into separate graduated cylinders of 1000 mL capacity for settling. From the graduated cylinders, clear juice samples were taken and analyzed for Brix, Pol, Purity, Color and Turbidity.

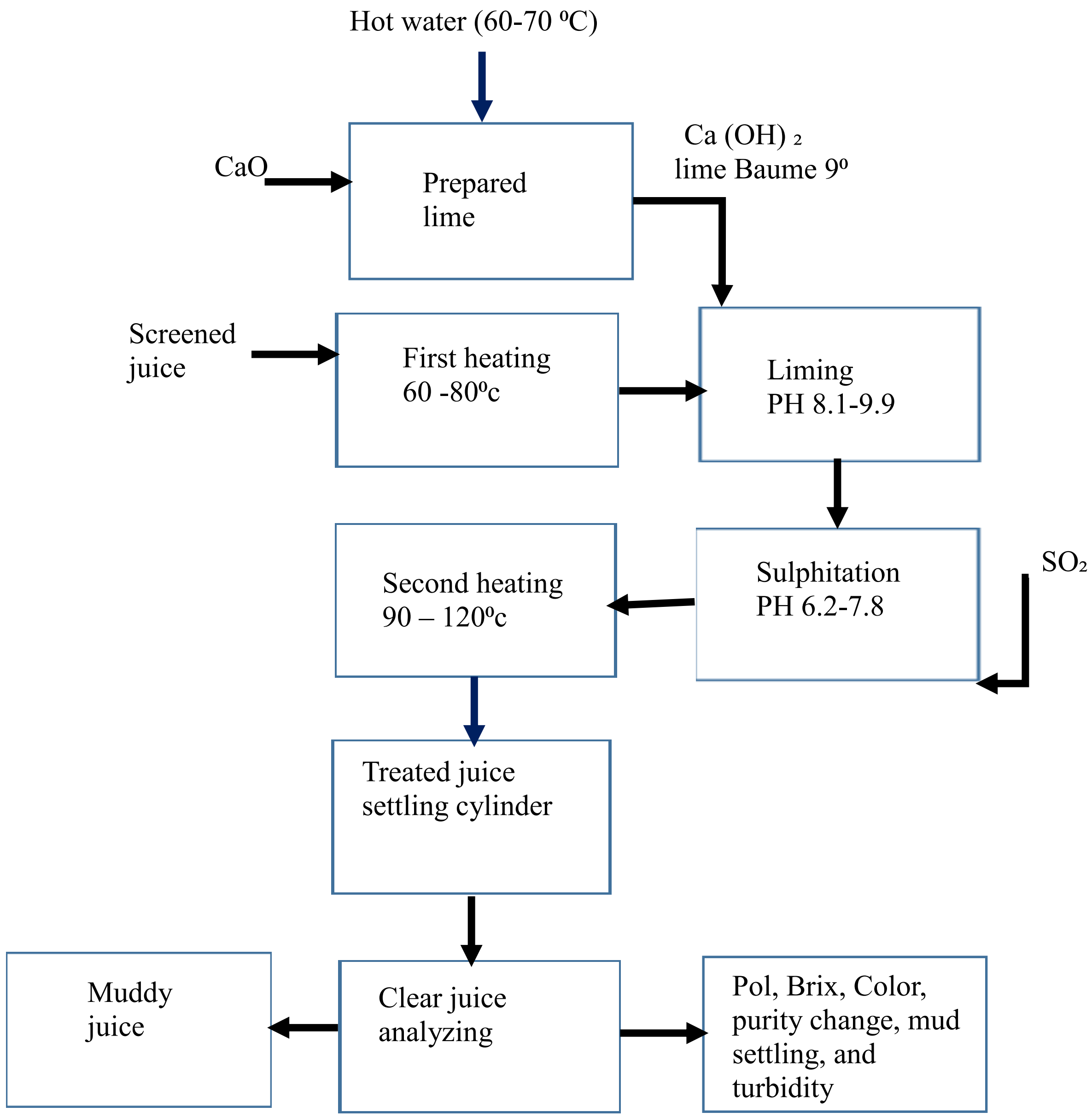


Figure 3.1: Laboratory experimental setup

3.3. Experimental Design (DOE)

The effect of four factors : Liming pH

: Sulphation pH

: First juice heating temperature

: Second juice heating temperature

Response variables: Color

: Mud settling

: Juice purity

: Turbidity

Design expert software of response surface methodology (RSM) with central composite design (CCD) was used. RSM is a collection of mathematical and statistical techniques useful for modeling and analyzing problems in which a response of interest is influenced by several variables and the objective is to optimize this response [50]. It is one effective method to optimize the effects of factors in the response variables. The main advantage of RSM is the reduced number of experimental runs needed to prove sufficient information for statistically acceptable results [51]. For four-variable with two levels (lower and higher levels) of each variable of the central composite rotatable design was employed. CCD is a very efficient design for fitting the second-order model. RSM with experiment run was conducted to investigate the effect of levels of factors on the response variables. The main response variables are color, mud setting, purity change, and turbidity. Then the optimum pH level combinations of the factors were determined. The experiment was conducted using a lower and upper real value. The lower and upper real values were coded as -1 and +1 respectively and that of the center points was coded as 0 as shown in **Table 3.2**. Then the optimum combinations of the factors were determined. Optimization was done by using Design –Expert v 7.

Table 3.2: Experimental ranges of the independent variables for RSM Optimization

Factors	**codes**	**Unit**	**Factor levels**		
			-1	0	+1
Liming	A	pH	8.1	9.0	9.9
Sulphitation pH	B	pH	6.2	7.0	7.8
First juice heating temperature	C	⁰C	60	70	80
Second juice heating temperature	D	⁰C	90	105	120

Table 3.3: RSM of Central composite design for optimization of response variables

Std	Run	liming pH	sulphitation pH	First heating (^0C)	second heating(^0C)
21	1	9.00	7.00	60.00	105.00
30	2	9.00	7.00	70.00	105.00
26	3	9.00	7.00	70.00	105.00
20	4	9.00	7.80	70.00	105.00
10	5	9.90	6.20	60.00	120.00
7	6	8.10	7.80	80.00	90.00
28	7	9.00	7.00	70.00	105.00
4	8	9.90	7.80	60.00	90.00
25	9	9.00	7.00	70.00	105.00
12	10	9.90	7.80	60.00	120.00
2	11	9.90	6.20	60.00	90.00
29	12	9.00	7.00	70.00	105.00
5	13	8.10	6.20	80.00	90.00
8	14	9.90	7.80	80.00	90.00
11	15	8.10	7.80	60.00	120.00
9	16	8.10	6.20	60.00	120.00
16	17	9.90	7.80	80.00	120.00
24	18	9.00	7.00	70.00	120.00
3	19	8.10	7.80	60.00	90.00
1	20	8.10	6.20	60.00	90.00
22	21	9.00	7.00	80.00	105.00
13	22	8.10	6.20	80.00	120.00
18	23	9.90	7.00	70.00	105.00
14	24	9.90	6.20	80.00	120.00
17	25	8.10	7.00	70.00	105.00
27	26	9.00	7.00	70.00	105.00
19	27	9.00	6.20	70.00	105.00
15	28	8.10	7.80	80.00	120.00

6	29	9.90	6.20	80.00	90.00
23	30	9.00	7.00	70.00	90.00

3.4. Artificial neural network (ANN) Optimization and Modeling

3.4.1. Artificial Neural Networks; Overview

Artificial neural networks (ANNs) are statistical models directly inspired by, and partially modeled on biological neural networks. They are capable of modeling and processing nonlinear relationships between inputs and outputs in parallel. Artificial neural networks are characterized by containing adaptive weights along paths between neurons that can be tuned by a learning algorithm that learns from observed data to improve the model(Demuth, 2000).

Architecturally, an artificial neural network is modeled using layers of artificial neurons, or computational units able to receive input and apply an activation function along with a threshold to determine if messages are passed along(Basheer & Hajmeer, 2000).In a simple model, the first layer is the input layer, followed by one hidden layer, and lastly by an output layer. Each layer can contain one or more neurons as shown in **Figure 3.2**.

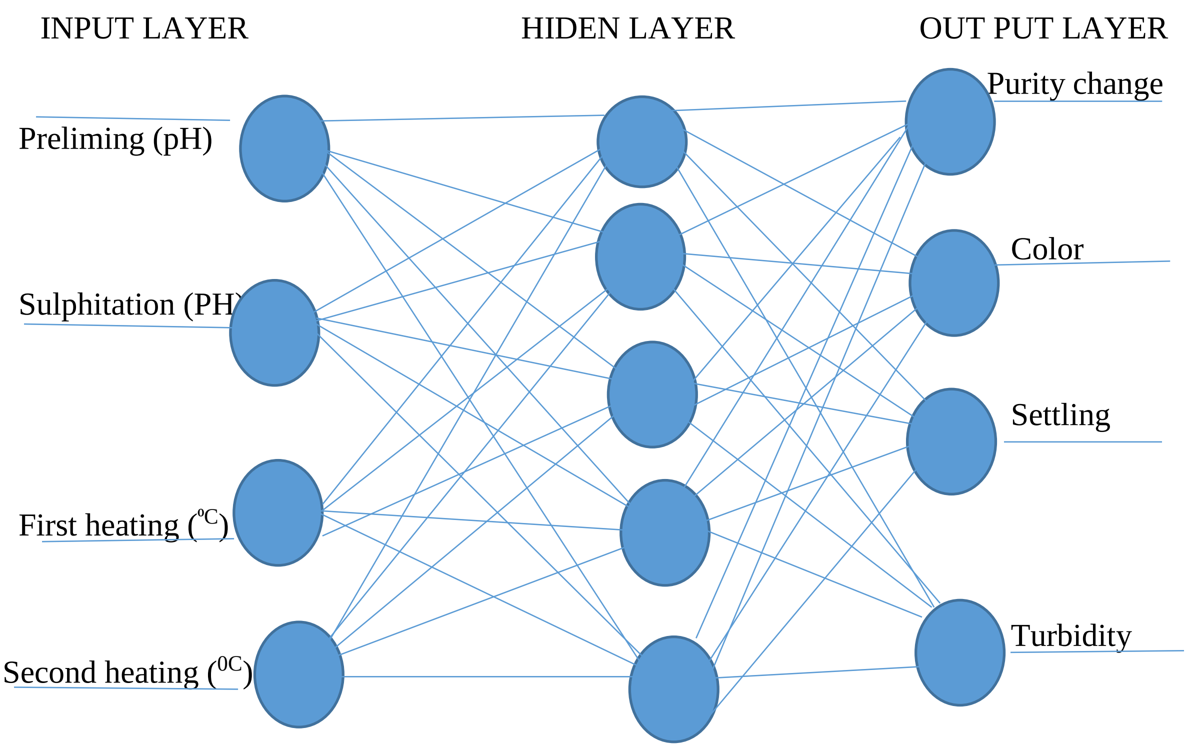


Figure 3.2: Network structure of artificial neural network of the experiment.

Workflow for using neural networks (Buscema et al., 2018)**:**

- Collect data
- Create the network
- Configure the network
- Initialize the weight and bases
- Train the network
- Validate the network
- Use the network

ANN modeling can be an admirable option for RSM in support of solving regression-based polynomial systems (Selvakumar et al., 2019). ANN structural design is made of remarkably interlinked basics which are operating in parallel called neurons and are encouraged by biological nervous systems. As in nature, the network function is determined largely by the connections between elements. One can train a neural network to perform a particular function by adjusting the values of the connections (weights) between elements. The network is adjusted, based on a comparison of the output and the target, until the network output matches the target. ANN structure is extremely linked neurons between weight matrix (w) and biases vector (b). The neurons in the ANN modeling are measured by transfer and summing function. The most used transfer functions are situated purlin (Linear Transfer Function), logsig (Log-Sigmoid Transfer Function), and tansig (Tan-Sigmoid Transfer Function) (Selvakumar et al., 2019). A multi-layer neural construction comprises an input, output, and hidden layer. Neural Network (NN) toolbox in MATLAB (R2018b) mathematical software was used for predicting the response variables such as purity change, color, mud settling, and turbidity. A multi-layer feedforward neural network approach with the tangent sigmoid transfer function (hidden layer) and a linear transfer function (output layer) was used for network training, and the experimental response was taken from Table (design expert) to train the network. The scaled data set formed after hidden layer operation act as the input for the output layer. The ultimately expected response by the ANN model was generated by the output layer (Selvakumar et al., 2019). A mean-squared error function was proven by the predicted and actual responses. The finest condition was confirmed experimentally and compared to determine the efficiency of both RSM and ANN, and then hidden layer sums up the weighted inputs with the biases.(Vrahatis et al., 2001)

3.5 Genetic Algorithms for optimization

Genetic algorithms (GAs) are based on biological principles of evolution and provide an interesting alternative to “classic” gradient-based optimization methods. They are particularly useful for highly nonlinear problems and models, whose computation time is not a primary concern. GAs approach the entire design space randomly and then improve the found design points by applying genetics-based principles and probabilistic selection criteria(Algorithm, 2006).

The main advantages of GAs are (Algorithm, 2006):

- ✓ The nature of the optimization model does not need to be known. This makes GAs very interesting for complex problems or for users inexperienced in gradient-based optimization techniques
- ✓ The optimization model and its constraints do not have to be continuous or even real values. No simplification of a problem is necessary to accommodate it to a particular algorithm
- ✓ They are readily available and easily implemented.

The main disadvantages are:

- ✓ A large number of parameters need to be set.
- ✓ Due to the comparatively very large number of function calls, GAs require significant computational resources. This makes them unattractive for optimization problems with computationally demanding analyses.

3.6. Statistical Analysis

In this study, all the experiments were conducted in triplicates, and the data expressed were as an average of three values. The clarification efficiency was statistically analyzed by the one-way ANOVA technique to determine the statistical difference between the means of independent groups. P-value < 0.05 is deemed to be statistically significant. CCD was used to design the experiments for optimizing experimental parameters of the response variables using RSM (Design Expert 7.0., Stat-Ease Inc and ANN (MATLAB (R2018b), Math Works, USA).

3.7. Analysis Methods

All analysis methods were based on Kassa's Handbook of Laboratory Methods and Chemical Control for Ethiopian Sugar Factories [49]; and SASTA (South African Sugar Technologist Association).

3.7.1. pH. Measurement of mixed and clear juice samples

The pH meter was standardized with buffer solutions of pH 4.0, 7.0, 9.2, & 10.Then, hot samples were cooled to room temperature before measuring the pH. Apart from cooling, no other treatment of the samples was necessary. The juice sample was placed in a clean beaker and after rinsing the electrodes with distilled water it was immersed in the sample. Then, the corresponding reading values were taken directly from the digital instrument.

3.7.2. Brix measurement of mixed and clear juice samples

The Brix of the mixed and clear juices were determined according to determination of brix in juices using refractometric method [44]. The refractometer has a circulatory water bath to maintain the juice temperature at 20 °C and a few drops of the sample were placed on the refractometer prism. Then, the Brix value was directly taken from the refractometer reading.

3.7.3. Determination of pol in factory products

The polarization of the mixed and clear juices was determined according to method using determination of pol in juices method [44]. 100mL of the juice sample was poured into the beaker provided with a stopper. One gram of basic lead acetate was added and shaken thoroughly, then allowed to stand for 30 seconds to permit flocculation of the precipitate. Then it was filtered through a fluted filter paper held in the funnel, which rests directly in the beaker. The first 25 mL of the filtrate were discarded and the saccharometer tube was rinsed twice with a portion of the filtrate. The tube was filled with the remaining filtrate and then the saccharometer reading was taken. Finally, Pol% of the juice sample was calculated based on the refractometer Brix reading of the juice sample and saccharimeter reading from a table on appendix 5. The purity of the mixed and clear juices was determined using the following ratio.

$Pol\% = \text{Pol reading} \times \text{pol factor}$

$\text{Purity}(\%) = \frac{\text{Pol}}{\text{Brix}} * 100$……………………………………..3.7.1

3.7.4. Determination of color in clarifying juice

The ICUMSA color method using a wavelength of 420nm is applicable for testing the color of light-colored products, usually made on clarifying the juice to measure the effectiveness of clarification. The color index of the clear juice was determined according to method 3.4 of the South African Sugar Technologists' Association [44]. A clarified juice sample was filtered through a membrane filter (0.45 µm) to remove turbidity. The pH of the solution was adjusted to 7.00 ± 0.02 using basic or acidic solutions. The Brix of the solution was measured and converted to total dissolved solids (g/mL) and absorbance of the filtered solution was measured at a wavelength of 420 nm and the ICUMSA color of the solution was calculated as the ratio of absorbance (A_S to the product of the dissolved concentration, c (g/mL), and the cell width, b (cm). The value of the absorbance index multiplied by 1,000 was reported as the ICUMSA color of the solution and the resulting value was expressed in ICUMSA units (IU). Since the wavelength at which the determination of color in solution is set at 420 nm, the value is designated as being the ICUMSA 420 color [44]. The pH meter was calibrated using fresh buffer solutions of pH 7.00 and 9.00 while stirring at a constant rate. The clear juice was allowed to cool to room temperature and was mixed thoroughly to determine the approximate Brix of the unfiltered solution and the mass of juice needed to prepare a 2° Brix solution was calculated using the formula below.

$$\text{Mass of juice(gm)} = \frac{final\ brix}{Original\ brix} \quad \text{…………………………3.7.2}$$

This mass of the well-mixed sample was diluted to 100 gm using distilled water and the solution was filtered through a 0.45µm using cellulose acetate membrane under vacuum into a clean dry Buchner flask. The pH of the solution was adjusted to 7.00 by allowing 1minute for stabilization using a pH meter. Then the absorbance of the solution was measured in a 1 cm cell using the spectrophotometer at 420 nm against distilled water as a reference.

$$\text{ICUMSA COLOR (IU)} = \frac{A_s \times 100}{b \times C} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.7.3$$

Where As =absorbance obtain at 420nm from spectrophotometer

C g/mL = dissolved concentration

b (cm)=cell width /cuvette

$$Concentration\ of\ total\ solid\ \left(\frac{g}{ml}\right) = brix \times true\ density/100$$

Density found from table of handbook of laboratory methods and chemical control for Ethiopian sugar factories/ **on Appendix 6.**

3.7.5. Determination of the settling rate

The settling rate of the mud was determined by measuring the height of the mud settling in a graduated test tube with time. First, treated juice was added to 1000 mL of the graduated test tube, and immediately stopwatch was started. And then, every five minutes the height of mud settling down was taken until the settling became constant at which the stopwatch stopped. The settling rate of mud was calculated by the height of mud settling down over time and the average settling rates were taken (cm/sec).

$$\text{settling rate(v)} = \text{heightofmud(h)} \div \text{time(t)}$$

$$v\ \left(\frac{cm}{s}\right) = h \div t \ldots\ldots\ldots\ldots\ldots\ldots 3.7.4$$

3.7.6. Measurement of turbidity in clear juice

The turbidity of the clear juice was determined according to the method 3.5 of (Analysis, 2007). The ICUMSA (IU) color of unfiltered and filtered samples of the clarified juice is measured at 420 nm and the difference between the readings is the turbidity of the unfiltered juice. The result gives a useful measure of the effectiveness of the clarification process(Hundi, 2010) . A 2° Brix solution was prepared from the clear juice (CJ) by first determining the Brix of the Clear juice. Then a known gram of the original CJ had been weighed followed by dilution with distilled water until the weight became 100 g and then divided in to two equal portions. One portion was treated with 0.5 g celite filter aid and then filtered through a Buchner funnel using a 0.45 µm Whatman nitrate membrane filter with the aid of vacuum. The pH was adjusted to 7.0 ± 0.01 using either 0.1M HCl or NaOH solution. Then the absorbance of filter (A1) and unfiltered (A2) samples were measured in a 1cm cell using spectrophotometer at 420 nm light wave. The difference between the readings is the turbidity of unfiltered juice as the results were expressed in the following formula.

$$\text{ICUMSA COLOR (IU)C1} = \frac{\text{A1} \times 1000}{\text{1cm} \times \text{concentrationof } total\ solids\left(\frac{g}{mL}\right)}$$

$$= A1 \times 1000 \div \left(\text{1cm} \times 2 \times \frac{1.00602}{100}\right)$$

$$= (\text{A1} \times 1000) \div 0.0201$$

$$\text{ICUMSA COLOR (IU)C2} = \frac{\text{A2} \times 1000}{\text{1cm} \times \text{concentrationof } total\ solids\left(\frac{g}{mL}\right)}$$

$$= A2 \times \frac{1000}{0.0201}$$

$$Turbidity\ of\ clarified\ juice = C2 - C1 = (A2 - A1) * \frac{1000}{0.0201} \qquad \text{3.7.5}$$

CHAPTER FOUR

4. RESULTS AND DISCUSION

4.1. Clarification Performance

4.1.1. Laboratory result analysis obtained from WSSF laboratory

The analysis methods that have been used in this study for each sample have been listed in **Table 4.4**: Experimental Design Layout and Laboratory Analysis the determination steps of each parameter followed the Ethiopian Sugar Factory, laboratory manual for sugar production [41]. Brix and pol content of the juice samples were determined by using a digital refractometer and polarimetry respectively from the clear juice-receiving tank. The juices were analyzed as soon as possible. The most important parameters that have conducted on clear juice laboratory analysis results; Brix, pol, color, turbidity, and mud settling for each sample.

4.1.1.1. Quality of mixed juice

Table 4.1: shows the Analysis result of mixed juice of WSSF which is based on the daily analysis result of WSSF laboratory. The average value of mixed juice pH and purity (%) obtained during the assessment was good (5.29 and 86.66 respectively) which is in the range of standard operations parameter (SOP). Whereas the mixed juices Brix are above the range of standard operation. The major problem of WSSF is higher Brix obtained. This was because of the absence or failure of auto control imbibition water at the extraction plant and presence of high amount and not easily tolerable colloidal matters the mixed juice average result of daily analysis off WSSF is shown in **Table 4.1**.

Table 4.1: The analysis result of mixed juice of WSSF

Day average	Bx (deg.baume)	Pol (%)	Pty (%)	Suc (%)	Glu (%)	P_2O_5 (ppm)	P^H	CaO (ppm)
1	16.85	14.60	86.6	12.52	0.50	300	5.4	359
2	17.00	14.83	87.3	13.00	0.43	350	5.2	397
3	17.16	15.01	87.5	12.79	0.46	350	5.3	518
4	15.85	13.94	87.9	13.18	0.43	300	5.3	337
5	16.32	14.26	87.4	12.89	0.42	350	5.2	402
6	17.47	15.26	87.5	14.71	0.42	300		413
7	17.19	14.94	86.9		0.43		5.3	
8	17.55	15.39	87.7	12.66	0.46	250	5.3	402
9	16.66	14.33	86.0	13.02	0.44	300	5.4	396
10	16.93	14.63	86.4			200	5.3	370
11	17.85	15.23	85.3	13.19	0.69	300	5.3	402
12	18.20	15.54	85.4			300	5.2	375
13	16.64	14.14	84.9	12.71	0.62		5.2	
14	16.46	14.33	87.1	12.33	0.59	300	5.3	337
15	16.05	13.80	86.0	12.83	0.45	350	5.3	359
Average	**16.95**	**14.68**	**86.66**	**12.99**	**0.49**	**303.85**	**5.29**	**389.77**
Standard	**13-14**	**14-15**	**85-86**	**12-13**	**0.5-0.6**	**250-300**	**5.3-5.5**	**<900**

4.1.1.2. Quality of clear juice

Table 4.2: Shows laboratory analysis of clear juice at WSSF. According to the range of standard operation (SOP) the brix of the clear juice and the pH of the clear juice were out of standard this shows there is a clarification problem related to the liming, sulphation pH, first and second juice heating. There is problem of maloperation the assign operator is not controlling the parameters very well. These are;

- ✓ Unsteady flow of mixed juice
- ✓ Use of non-uniform lime
- ✓ Long time stay in the clarifier more than 3 hour cause inversion
- ✓ Intermittent of crushing etc.

Table 4.2: Laboratory analysis of clear juice at WSSF

Day average	Bx (deg.baume)	Pol (%)	Pty (%)	Glu (%)	CaO (ppm)	P^H
1	19.44	17.04	87.5	0.53	798	7.0
2	18.77	16.48	87.8	0.45	784	7.1
3	18.66	16.55	88.7	0.48	714	6.9
4	17.85	15.76	88.3	0.45	614	6.8
5	18.00	15.83	87.9	0.44	665	6.7
6	18.88	17.38	87.4	0.48	731	6.9
7	19.43	16.98	87.4	0.46	886	6.7
8	20.42	17.92	87.8	0.48	836	6.7
9	20.34	17.90	88.0	0.47	1025	6.7
10	21.69	18.41	86.5	0.5	729	6.8
11	20.53	17.75	86.5	0.68	869	6.6
12	19.64	17.42	86.9	0.57	1321	6.3
13	21.96	18.03	82.1	0.63	1368	6.2
14	20.31	17.20	84.7	0.61	1065	6.6
15	21.62	18.66	86.3	0.53	1065	6.6
Average	**19.85**	**17.28**	**86.92**	**0.52**	**897.97**	**6.71**

Table 4.3: Average data of WSSF quality of mixed juice and clear juice for the past five years taken from laboratory.

Mixed juice	**2015/16**	**2016/2017**	**2017/2018**	**2018/2019**	**2019/20**
Mixed juice % cane	98.2	93.47	93.52	98.92	95.96
Brix % mixed juice	16.26	16.58	15.38	15.45	15.35
Pol % mixed juice	14.53	14.43	13.31	12.98	13.78
Apparent purity of mixed juice	86.25	87.02	85.20	84.01	86.57
Clarified juice					
Brix % clarified juice	17.09	17.12	16.47	17.46	17.01
Pol % clarified juice	14.97	14.92	14.29	15.15	14.86
Purity rise/drop from mixed juice to clarified juice	1.37	0.09	2.05	3.24	0.79

Table 4.2: Laboratory analysis of clear juice at WSSF. The average pH of clear juice is 6.71 the standard is 7.0 which was less than three units from the standard. It is necessary to keep

the pH in the neutral for the production to be maximized. Lower values of pH less than7.0 should be avoided because of sucrose inversion and higher values are undesirable, since chemical reactions leading to alkaline degradation may occur at the processing temperature. Therefore the factory faces the above problem because of many reasons like; there is no steady flow of mixed juice and keep juice for a long time within the clarifier cause fermentation of the juice which lead to inversion of sucrose. In addition, using a non-uniform concentration of milk of lime, the optimum reaction time and dose of each treatment were not kept, and mainly the failure of the lime auto control system makes it difficult to keep the standard pH. Liming is controlled to obtain a limed juice pH, which can vary between 8.6 to about 9.0 and fall to control juice heating temperature. The set point is chosen to provide a clarified juice pH between 7.0 and 7.2.

Brix: estimation of Brix in the factory is one of the quality operational parameters. The average result obtained was 19.85, which shows that Brix was not in the range of the standard value during the operation (the standard is 13.0 – 14.5). They were almost equal to the output of the first effect of the evaporator brix. This is because of heating the juice at higher temperature which is beyond standard and also cause thermal destruction of sucrose.

Purity and purity rise or fall: the average purity of clear juice for Wonji/shoa sugar was 0.09-3.24% that was read as the average purity of clear juice as obtained from factory report which keeps the standard value of clear juice quality. However, when we see according to clarification performance parameters especially purity rise or fall of mixed juice to clear juice the value is most of the time below the standard parameter in WSSF because of operating parameters of clarification plant. Purity rise or fall is one of the most important indicators of performance in the clarification process to identify whether the operating system is good or not. It is the difference between purity after and before the clarification process. Since some of the dissolved non-sugars were removed from the mixed juice, the pol of the clear juice exceeds that of the MJ, consequently, the purity (pol/Brix) increases.

Turbidity: Standard juice turbidity set by WSSF was 700-1500 IU.

Problems

- ✓ Poor cane quality was being processed (excess trash, maturity of cane).
- ✓ Poor control of clarification reagents (dosages flocculants, Sulphur, and lime) and
- ✓ Process parameters (working temperature, pressure, flow etc.)

Make the reaction that takes place in clarification incomplete and removal of unwanted Martials becomes less. Besides the disturbance of the chemical reaction processing of less quality lime is the addition of impurities on juice, which increases the turbidity of clear juice. If the heating temperature was not controlled can affect the turbidity.

- **Reasons that cause of poor clarification operation at WSSF**

Temperature, retention time

The clarification process of raw juice requires the juice to be heated and treated with consumable chemicals like SO_2, Ca $(OH)_2$ (milk of lime), and flocculants. Temperature control and flow condition of the juice (steady or unsteady) clarification is critical because heating the juice causes organic coagulation of proteins, destroying microorganisms and enzymes that prevent loss of sucrose by microbiological activity and accelerates the reaction rate. In addition, to flash out its air bubbles that would retard settling at the Dorr clarifier, coagulation of colloidal impurities and facilitate flocculation and precipitation of insoluble impurities in the clarifier. WSSF has poor juice heating control because of many reasons power fluctuation and failure of thermocouples repeatedly. Keeping the temperature as much as possible to the standard operation values can bring a better result. The retention time of juice in the clarifiers has a great effect on the juice. 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 a longer period. However excessive capacity clarifiers that hold juice for long periods result in higher levels of inversion which lead to fermentation. The minimum retention time (based on clarifier capacity) recommended for the Rapi-Dorr 444 clarifier is 2 hours and a maximum of 3 hours. At WSSF juice in clarifier stay longer time because of different reason like the failure of machinery and for maintenance, supply of cane was intermittent, steam supply also not continuous and electric power failure so all of the above made the juice to stay a long time at Dorr clarifier.

4.1.1.3. Available P_2O_5 in mixed juice

When mixed juice for its natural P_2O_5 content is less than 250 ppm, termed as phosphate deficient juice so that the deficient amount of P_2O_5 was satisfied by the addition of pre-calculated amount of phosphoric acid. However, in the case of WSSF the amounts of phosphate contents shown in mixed juice were above 350 ppm. They meet the required standard operation so the addition of phosphates during clarification are not required and advisable because of the formation of precipitates of tri-calcium phosphates (Kulkarni, 2004). In the other side, high level of phosphate in juice contribute to the juice pH and level of calcium added to the juice by milk of lime addition, thus, the proportion of soluble calcium and phosphate increase scaling in the evaporators, high ash in sugar and less efficient molasses exhaustion. Furthermore, Disadvantages of too much phosphate content of juice can be higher lime consumption and higher mud volume (and hence higher filter mud sugar loss). Additionally, high levels of phosphate present in clear juice adversely affect sugar Filterability.

4.2. Optimization of Main Parameters in Clarification of Juice

Optimization of juice treatment chemical is a process that optimizes liming pH, sulphiting pH, and first and second heating temperature to obtain maximum purity change, minimum color, maximum mud settling, and minimum turbidity as much as possible for efficient juice clarification. The data for response variables were obtained from laboratory experimental works based on factors combination. For each factor combination, the triplicate experiment was done and the average values were taken. Maximum purity change, minimum color, maximum mud settling, and minimum turbidity were observed for efficient juice clarification. Results obtained from experimental works for response variables are presented in **Table 4.4**.

Table 4.4: Experimental Design Layout and Laboratory Analysis.

		Factors				**Responses**			
Std	**Run**	**preliming pH**	**sulphitation pH**	**First heating (°C)**	**Second heating**	**Purity change**	**Color (IU)**	**Mud settling (cm/s)**	**Turbidity (IU)**
25	1	9.00	7.00	70.00	105.00	2.2	6700	0.058	640
18	2	9.90	7.00	70.00	105.00	1.3	6800	0.058	840
9	3	8.10	6.20	60.00	120.00	-0.6	14500	0.0089	2500
4	4	9.90	7.80	60.00	90.00	0.5	10500	0.03	1240
19	5	9.00	6.20	70.00	105.00	-0.1	11000	0.068	845
6	6	9.90	6.20	80.00	90.00	-0.3	13000	0.02	923
24	7	9.00	7.00	70.00	120.00	1.8	10900	0.018	1945
21	8	9.00	7.00	60.00	105.00	0.8	11000	0.025	1120
12	9	9.90	7.80	60.00	120.00	0.6	11000	0.02	1786
7	10	8.10	7.80	80.00	90.00	0.2	14300	0.009	1930
10	11	9.90	6.20	60.00	120.00	-0.2	15600	0.01	2000
8	12	9.90	7.80	80.00	90.00	0.6	12000	0.0079	1543
13	13	8.10	6.20	80.00	120.00	-0.5	15200	0.005	1432
2	14	9.90	6.20	60.00	90.00	-0.2	15100	0.03	1345
16	15	9.90	7.80	80.00	120.00	0.4	13000	0.013	1645
17	16	8.10	7.00	70.00	105.00	0.6	9800	0.04	1748
23	17	9.00	7.00	70.00	90.00	2	7900	0.05	900
15	18	8.10	7.80	80.00	120.00	0.1	15500	0.0079	1500
22	19	9.00	7.00	80.00	105.00	0.8	10800	0.034	1050
28	20	9.00	7.00	70.00	105.00	2	7200	0.055	860
30	21	9.00	7.00	70.00	105.00	1.9	6900	0.058	880
5	22	8.10	6.20	80.00	90.00	-0.5	13500	0.018	1456
14	23	9.90	6.20	80.00	120.00	-0.6	14900	0.012	1976
26	24	9.00	7.00	70.00	105.00	1.8	6800	0.059	640
20	25	9.00	7.80	70.00	105.00	1.1	9900	0.055	1182
11	26	8.10	7.80	60.00	120.00	0	14900	0.0098	2365

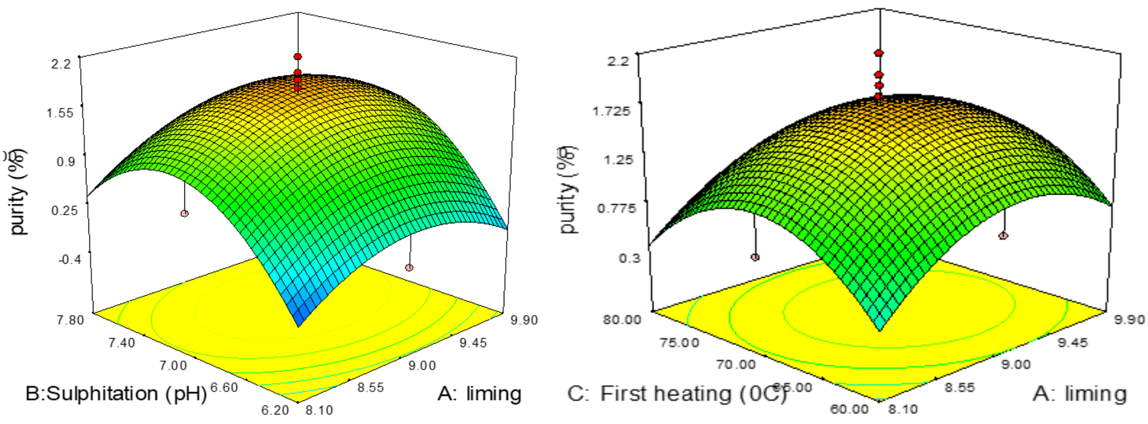
1	27	8.10	6.20	60.00	90.00	-0.4	15800	0.019	1987
29	28	9.00	7.00	70.00	105.00	1.8	6900	0.059	650
3	29	8.10	7.80	60.00	90.00	0.2	15000	0.0077	1965
27	30	9.00	7.00	70.00	105.00	2	8000	0.067	920

4.2.1. Statistical Analysis and Discussion of the Experimental Data

Experimental data were statistically analyzed by using Design-Expert software to investigate the effect of factors on the response variables. It was modeled by the statistical analysis and the statistical significance of the terms in the model was examined by analysis of variance (ANOVA) for each response. The adequacy of the regression model was checked by R^2, adjusted R^2, predicted R^2, adequate precision, and F-test (Myers et al., 2004). Adequacy of the models for all responses was tested by diagnostic plots. Variations of responses for processing variables were checked by using different model graphs such as factors plot, three-dimensional response surfaces plot, cubic plot, etc. Moreover, an artificial neural network (ANN) using Matlab software was used for prediction purpose.

4.2.1.1. Data Analysis of Purity Change Interpretation

As it was seen that from **Table 4.4** the minimum and the maximum purity change were -0.6 and 2.2% respectively. This wide range of responses indicated that the purity change value was significantly affected by the concentration of liming, sulphiting pH, and first and second heating. For example, if we look at the combination of liming pH 9.9, sulphiting pH of 7.8 first and second heating 80 and 90 ⁰C gave a purity change value of 0.6. Whereas, liming pH 9.9, sulphiting pH 6.20, first and second heating temperatures 80 and 90⁰C gave a purity change of -0.3. The combination of liming pH 9.0, sulphiting pH of 7.0, first and second heating temperature70 and 105 ⁰C gave a purity change value of 1.8-2.2 % whereas that of liming pH 8.10, sulphiting pH 7.0, first and second heating 70 and 105 ⁰C gave a purity change of 0.6%. High purity rise at center point locations was observed where the level of liming and sulphiting pH values at 9.0 and 7.00 and first and second heating temperature at 70 and 105 ⁰C as shown in **Figure 4. 1**. Since some of the dissolved non sugars are removed from the mixed juice (MJ), the pol of the clear juice (CJ) exceeds that of the MJ, consequently, the ratio of pol to Brix (Pol: Brix) increases(Dionisi et al., 2008). The clear juice purity was measured after maintaining the CJ temperature to reach room temperature. As invert sugar can form on cooling hot juices, purity changes based on pol changes are often considered to offer little information and maybe misleading [10]. As it was observed purity change became negative because of the inversion of sucrose to glucose and fructose in acidic media.



a b

Figure 4. 1: Response surface plots for purity rise

Table 4.5: ANOVA results for purity change of clear juice

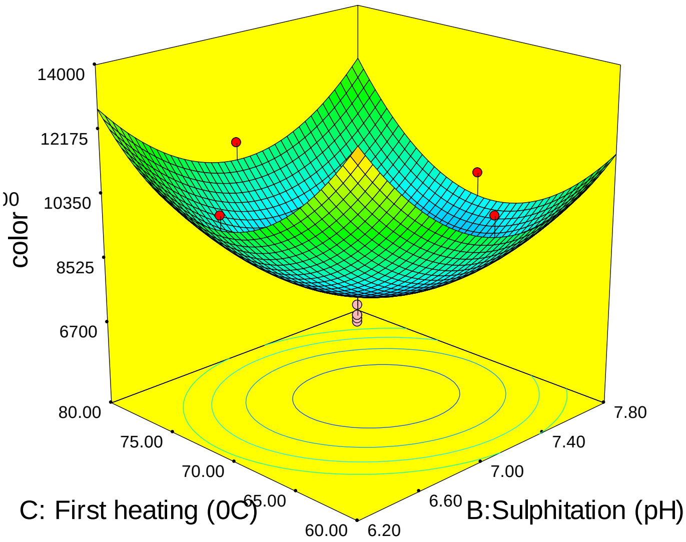
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P-value Prob > F**	
Model	23.95	14	1.71	22.83	< 0.0001	significant
A-Preliming	0.50	1	0.50	6.67	0.0208	
B-Sulphitatiom	2.80	1	2.80	37.37	< 0.0001	
C-First heating	0.014	1	0.014	0.19	0.6729	
D-Second heating	0.067	1	0.067	0.90	0.3586	
AB	0.051	1	0.051	0.68	0.4240	
AC	0.031	1	0.031	0.41	0.5323	
AD	6.250E-004	1	6.250E-004	8.341E-003	0.9284	
BC	0.016	1	0.016	0.21	0.6545	
BD	6.250E-004	1	6.250E-004	8.341E-003	0.9284	
CD	5.625E-003	1	5.625E-003	0.075	0.7878	
A^2	0.74	1	0.74	9.90	0.0067	
B^2	2.51	1	2.51	33.55	< 0.0001	

C^2	1.22	1	1.22	16.23	0.0011	
D^2	0.45	1	0.45	5.95	0.0276	
Residual	1.12	15	0.075			
Lack of Fit	1.01	10	0.10	4.39	0.0582	not significant
Pure Error	0.12	5	0.023			
Cor Total	25.07	29				

A quadratic model was taken to test for its adequacy to describe its variation with independent variables. From the ANOVA test in thejuice the Model F-value of 22.83 implies the model is significant. There is only a 0.01% chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, A^2, B^2, C^2, D^2 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 model. The "Lack of Fit F-value" of 4.39 implies there is a 5.82% chance that a "Lack of Fit F- value" this large could occur due to noise. Lack of fit is bad we want the model to fit. This relatively low probability (<10%) is troubling.

4.2.1.2. Data analysis of Color value interpretation.

The various combinations of the factors gave a clear juice color range of 6700 – 15,820IU **Table 4.4**. It was observed that the ICUMSA color increased with an increased level of liming pH and also with low sulphiting pH combinations. A high pH of liming due to the destruction of reducing sugar leads to the development of high coloring materials in the process [15]. And at higher temperatures, there is caramelization (coloration) it is the charring of sugar manifested at high-temperature operations. So forming color compound.



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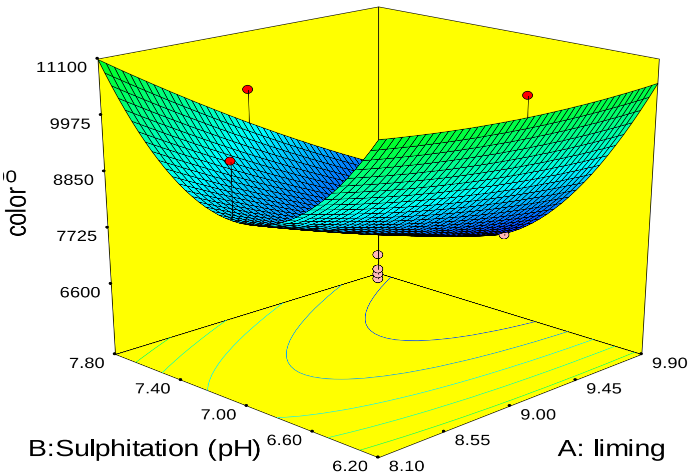


B

Figure 4. 2: 3D surface plot for color of clarified juice

Table 4.6: ANOVA results for color value of clear juice

Source	**Sums of Squares**	**df**	**Mean Square**	**F Value**	**P-value Prob > F**	
Model	2.893E+008	14	2.067E+007	28.56	< 0.0001	significant
A-Preliming	1.531E+007	1	1.531E+007	21.16	0.0003	
B-Sulphitatiom	8.681E+006	1	8.681E+006	12.00	0.0035	
C-First heating	80000.00	1	80000.00	0.11	0.7441	
D-Second heating	3.920E+006	1	3.920E+006	5.42	0.0343	

AB	1.024E+007	1	1.024E+007	14.15	0.0019	
AC	3.600E+005	1	3.600E+005	0.50	0.4914	
AD	3.600E+005	1	3.600E+005	0.50	0.4914	
BC	3.803E+006	1	3.803E+006	5.26	0.0368	
BD	2500.00	1	2500.00	3.455E-003	0.9539	
CD	2.403E+006	1	2.403E+006	3.32	0.0884	
A^2	55600.08	1	55600.08	0.077	0.7854	
B^2	1.366E+007	1	1.366E+007	18.88	0.0006	
C^2	1.954E+007	1	1.954E+007	27.01	0.0001	
D^2	4.026E+006	1	4.026E+006	5.56	0.0323	
Residual	1.085E+007	15	7.236E+005			
Lack of Fit	9.706E+006	10	9.706E+005	4.23	0.0626	not significant
Pure Error	1.148E+006	5	2.297E+005			
Cor Total	3.002E+008	29				

A quadratic model was taken to test for its adequacy to describe its variation with independent variables. From the ANOVA test in the Table 4.6, shown above, The Model F-value of 28.56 implies the model is significant. There is only a 0.01% chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, D, AB, BC, B^2, C^2, D^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 4.23 implies there is a 6.26% chance that a "Lack of Fit F- value" this large could occur due to noise. Lack of fit is bad we want the model to fit. This relatively low probability (<10%) is troubling (Myers et al., 2004).

Table 4 7: Checking of data and adequacy of model of color

Std. Dev.	850.64	R-Squared	0.9638
Mean	11480.00	Adj R-Squared	0.9301
C.V. %	7.41	Pred R-Squared	0.8422
PRESS	4.737E+007	Adeq Precision	15.298

The "Pred R-Squared" of 0.8422 is in reasonable agreement with the "Adj R-Squared" of 0.9301. "Adeq Precision" measures the signal-to-noise ratio. A ratio greater than 4 is desirable. A ratio of 15.298 indicates an adequate signal.

This model can be used to navigate the design space. 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) measures how a particular model fits each point in the design.

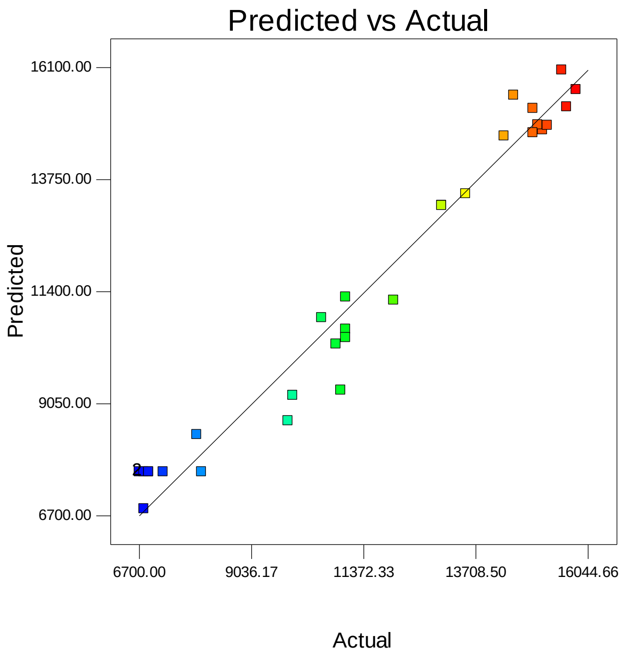


Figure 4.3: Actual by predicted plot of ICUMSA color for clear juice

Figure 4.3: indicates the relationship between the actual and predicted values of the color. A residual is a 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, tend to be close to the diagonal line.

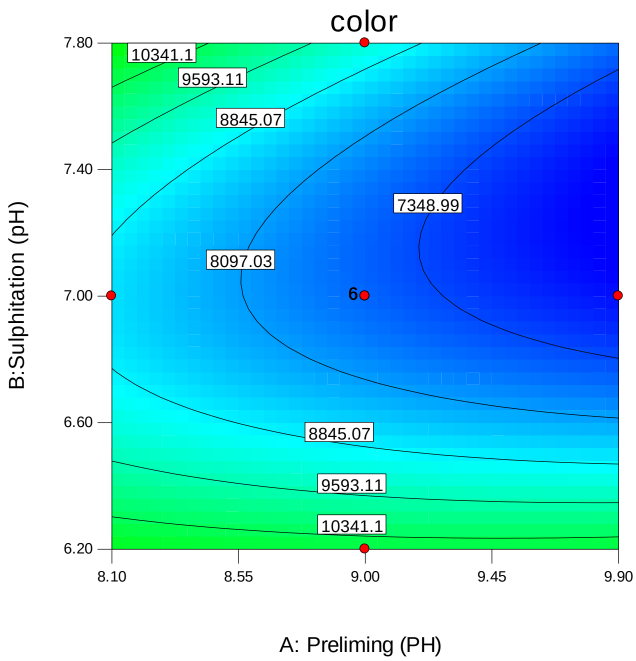


Figure 4.4: Contour Plot of color of clear juice

As was seen from figure 4.4 above as sulphitation and preliming pH increases the color of the clear juice becomes increases. This is due to an increase in the coloring matter in raw juices. At the same time for the low level of the factors (Martin Saska et al., 2010), (i.e. the sulphitation and preliming pH the observed color was minimum as they were compared with that of the high levels). If the amount of lime put into the juice is too small or the amount of sulfur dioxide is too large, the pH value in the sugarcane juice will become acid, which will affect the neutralization reaction and cause high SO_2 and calcium contents in the purified juice, and inevitably decrease the purity of juice (P. Rein, 2016). If too large an amount of lime or a too-small amount of sulphur dioxide is put into the juice, it will cause the reducing sugar to resolve and increase the color value of the purified juice, and inevitably decrease the purity of juice (Godshall, 1999). It has a vital significance to control pH value stability in the clarifying process of sugar juice, which affects directly the quality of the purified juice, and inevitably the product's quality. If the neutralized pH value is too low, this will speed up the sucrose transformation, which results in loss of sucrose and cause the juice to contain higher dissoluble calcium, and inevitably cause a lot of scales to form in the evaporation station and boiling house, thus it increases energy wastage. Contrarily, when the neutralized pH value is too high, the original sugar decomposes the new pigment so that its color becomes depth, these can increase the color value of the product sugar and affects the quality of the granulated sugar. Moreover, instability of the pH value can increase the amount of use of the clarifying agent, and increase the cost (Asfaw, 2015a).

Temperature control of juice clarification is critical because heating the juice causes organic coagulation of proteins, destroying microorganisms and enzymes that prevent loss of sucrose by microbiological activity, and accelerates the reaction rate. In addition, to flash out its air bubbles that would retard settling at the Dorr clarifier, coagulation of colloidal impurities and facilitate flocculation and precipitation of insoluble impurities in the clarifier. Too high temperature caused Brix up and form a color-forming compound that increase the color (Martin Saska et al., 2010).

4.2.1.3. Data analysis of Mud settling of clear juice

The different level combinations of preliming, sulphiting pH, and first and second heating temperature values show a settling increment ranging from 0.0079 – 0.068cm/s **Table 4.4**. High settling rates at average point locations were observed where the level of liming and sulphating pH values at 9.00 and 7.00 and first and second heating temperature values at 70 and 105 ^{0}C

Table 4.8: ANOVA results for mud settling of clear juice

Source	Sum of Squares	df	Men Square	F Value	P-value Prob > F	
Model	0.013	14	9.184E-004	18.11	< 0.0001	significant
A-Preliming	3.175E-004	1	3.175E-004	6.26	0.0244	
B-Sulphitatiom	5.202E-005	1	5.202E-005	1.03	0.3273	
C-First heating	6.272E-005	1	6.272E-005	1.24	0.2837	
D-Second heating	4.205E-004	1	4.205E-004	8.29	0.0115	
AB	1.482E-005	1	1.482E-005	0.29	0.5967	
AC	6.241E-005	1	6.241E-005	1.23	0.2848	
AD	7.290E-006	1	7.290E-006	0.14	0.7099	
BC	1.764E-005	1	1.764E-005	0.35	0.5642	
BD	1.392E-004	1	1.392E-004	2.74	0.1183	
CD	2.756E-005	1	2.756E-005	0.54	0.4724	
A^2	7.025E-005	1	7.025E-005	1.38	0.2576	
B^2	1.378E-004	1	1.378E-004	2.72	0.1201	
C^2	1.582E-003	1	1.582E-003	31.18	< 0.0001	
D^2	1.058E-003	1	1.058E-003	20.86	0.0004	
Residual	7.609E-004	15	5.073E-005			
Lack of Fit	6.796E-004	10	6.796E-005	4.18	0.0640	not significant
Pure Error	8.133E-005	5	1.627E-005			
Cor Total	0.014	29				

From the ANOVA result quadratic model was suggested.The Model F-value of 18.11 implies the model is significant. There is only a 0.01% chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, D, C^2, D^2 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. The "Lack of Fit F-value" of 4.18 implies there is a 6.40% chance that a "Lack of

Fit F- value" this large could occur due to noise. Lack of fit is bad -- we want the model to fit. This relatively low probabiity (<10%) is troubling.

Table 4.9: Fit Statistics for Settling Rate

Std. Dev.	7.122E-003	R-Squared	0.9441
Mean	0.031	Adj R-Squared	0.8920
C.V. %	22.92	Pred R-Squared	0.7535
PRESS	3.358E-003	Adeq Precision	12.497

The "Pred R-Squared" of 0.7535 is in reasonable agreement with the "Adj R-Squared" of 0.8920. as one might normally expect. This may indicate a large block effect or a possible problem with your model and/or data. Things to consider are model reduction, response transformation, outliers, etc. "Adeq Precision" measures the signal-to-noise ratio.
A ratio greater than 4 is desirable. ratio of 12.497 indicates an adequate signal. This model can be used to navigate the design space (Anderson-Cook et al., 2009).

Diagnostic Test for Settling Rate

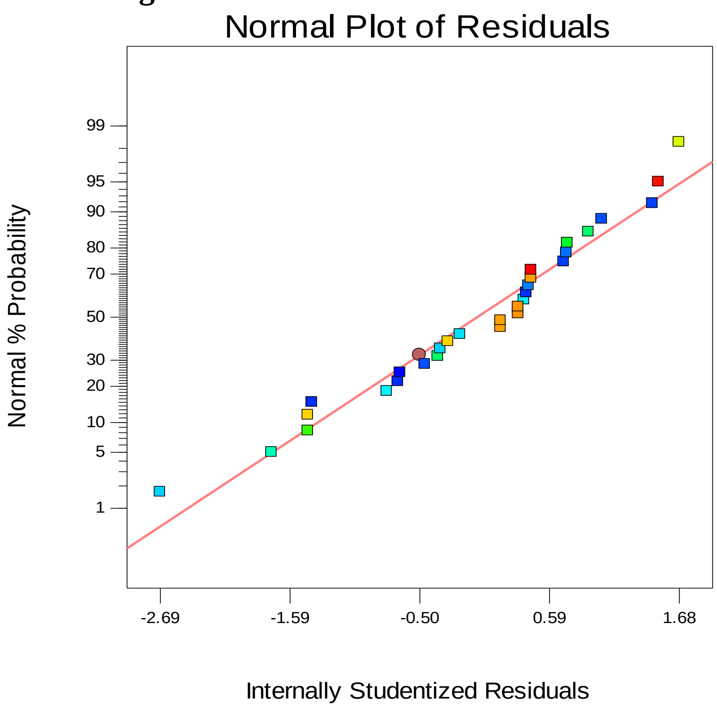


Figure 4.5: Normal probability plot for mud settling

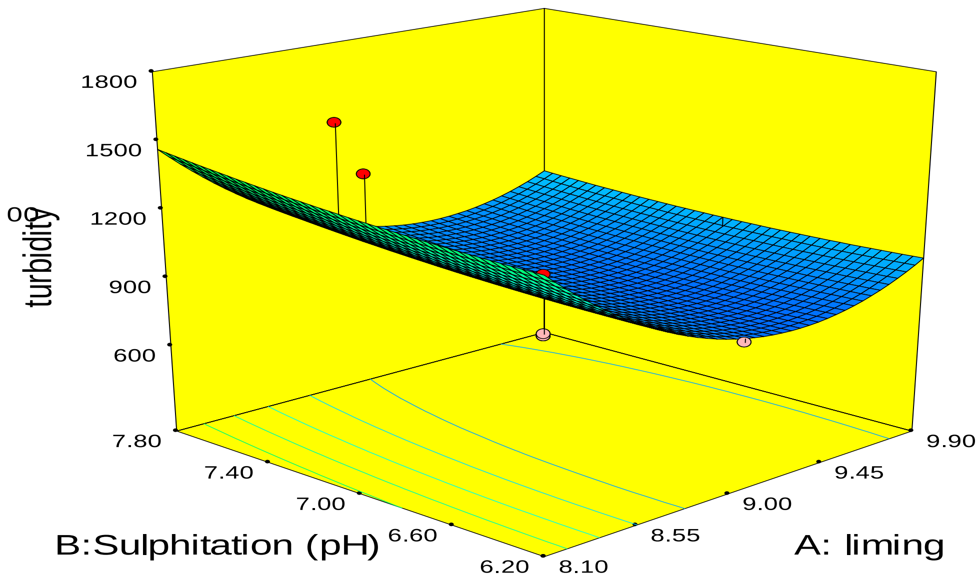
As shown in **Figure 4.5**: the normal probability plot indicates the residuals following by the normal % probability distribution, in the case of this experimental data the points in the plots show fitted to the straight line in the figure, this shows that the quadratic polynomial model satisfies the assumptions analysis of variance (ANOVA) i.e., the error distribution is approximately normal.

4.2.1.4. Data Analysis and Discussion for Turbidity

It was observed that the minimum and the maximum turbidity values were 640 IU and 2,500 IU, respectively **Table 4.4**. This wide range of responses indicated that the turbidity value was significantly affected by the concentration of liming, sulphiting pH, and first and second heating temperature.

It shows that higher levels of liming pH, sulphiting pH lower turbidity. The high liming pH in the process catches high suspended impurities in the juice by precipitation with phosphate. Turbidity is mainly caused by suspended impurities. The phosphate reacts with the calcium ions (Ca^{+2}) present in the juice to form primary flocculation of insoluble calcium phosphate, and it sticks with impurities. And also, SO2 neutralizes excess lime in juice precipitates calcium sulphite and it catches remaining impurities (Kulkarni, 2004) . At higher levels of liming pH, sulphiting pH, the juice is free from suspended impurities and which means turbidity is lower. But, higher levels of liming pH and sulphiting pH lead to a higher CaO content, which is not good. Hence, there is an optimum point to compromise all the conditions.

The effect of temperature on turbidity has also significant if the first heating temperature was low the reaction was not fast so there were more suspended impurities which means the lime was not catch up with the SO_2 gas to form $CaSO_4$ and form precipitate so that the turbidity increased. Similarly, if the second heating temperature is low the air or gases inside the juice were not vented out to the atmosphere so causing some turbulence and there were floating which hinders the settling of impurities so turbidity increases. When the temperature was higher the Brix increased the settling also decrease and there was some floating so turbidity was increased so to obtain minimum turbidity the factors should be controlled as shown in **Figure 4. 6**.



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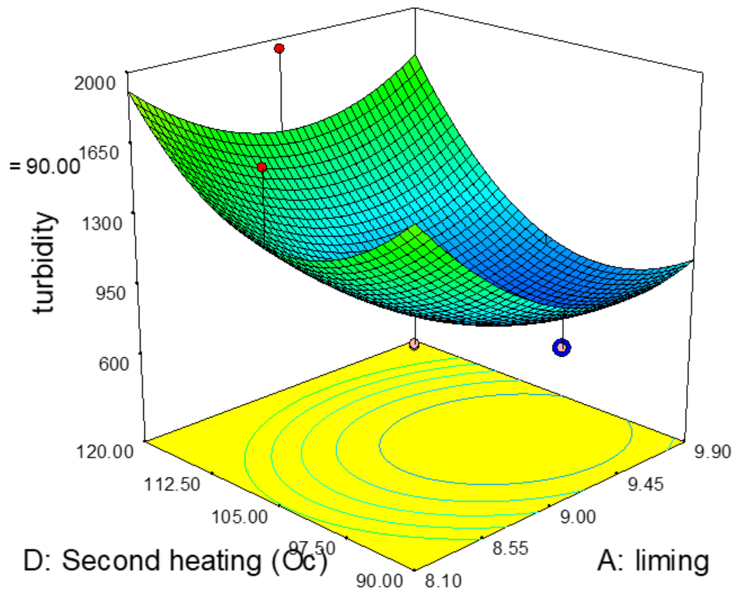


b

Figure 4. 6: 3D surface plot for turbidity

Table 4.10*: ANOVA results for turbidity of clear juice*

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	7.397E+006	14	5.284E+005	8.54	< 0.0001	significant
A-Preliming	7.140E+005	1	7.140E+005	11.55	0.0040	
B-Sulphitatiom	26603.56	1	26603.56	0.43	0.5218	

C-First heating	4.522E+005	1	4.522E+005	7.31	0.0163	
D-Second heating	8.278E+005	1	8.278E+005	13.39	0.0023	
AB	10764.06	1	10764.06	0.17	0.6824	
AC	3.066E+005	1	3.066E+005	4.96	0.0417	
AD	2.249E+005	1	2.249E+005	3.64	0.0758	
BC	1.068E+005	1	1.068E+005	1.73	0.2086	
BD	1.558E+005	1	1.558E+005	2.52	0.1333	
CD	1.248E+005	1	1.248E+005	2.02	0.1759	
A^2	2.422E+005	1	2.422E+005	3.92	0.0665	
B^2	1647.85	1	1647.85	0.027	0.8725	
C^2	24236.98	1	24236.98	0.39	0.5407	
D^2	4.885E+005	1	4.885E+005	7.90	0.0132	
Residual	9.276E+005	15	61839.59			
Lack of Fit	8.368E+005	10	83684.38	4.61	0.0527	not significant
Pure Error	90750.00	5	18150.00			
Cor Total	8.325E+006	29				

A quadratic model is suggested. The Model F-value of 8.54 implies the model is significant. There is only a 0.01% chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, C, D, AC, D^2 are significant model terms.Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 4.61 implies there is a 5.27% chance that a "Lack of Fit F-value" this large could occur due to noise. Lack of fit is bad we want the model to fit. This relatively low probability (<10%) is troubling.

Table 4.11: fit summery of turbidity

Std. Dev.	248.68	R-Squared	0.8886
Mean	1393.77	Adj R-Squared	0.7846
C.V. %	17.84	Pred R-Squared	0.4693
PRESS	4.418E+006	Adeq Precision	9.781

"Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. ratio of 9.781 indicates an adequate signal. This model can be used to navigate the design space.

Diagnostic Test for Turbidity

As shown in the Figure 4.7: Measured by predicted plot all points are closes to the straight line. This indicates that normal distribution is an appropriate model or normal distribution adequately describes the data. Therefore, this shows that the quadratic model satisfies the assumptions of analysis of variance (ANOVA).

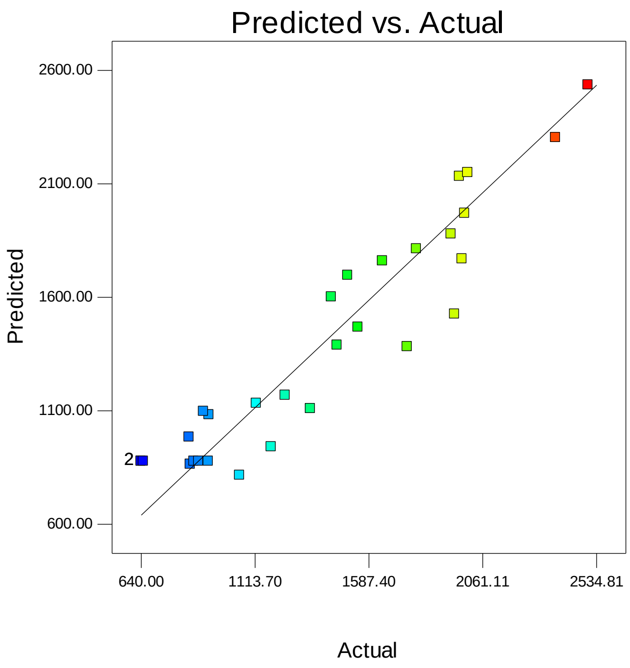


Figure 4.7: Measured by predicted plot of turbidity.

- **Searching for an optimum point**

The decision of the chemical dose and the control of the heating temperature should be made in such a way that it should not affect the other parameters. The following desirable ranges of responses need to be fulfilled to locate the common optimum point.

Purity change: High value of purity change is desirable. The 1.0 – 2.0 unit increment of mixed juice to clear juice purity rise is the indication of good clarification(Kulkarni, 2004).

Mud settling rate: high values of settling rate are desirable; hence, the region bounded by 0.064 was selected as an optimum region for settling rate.

Turbidity: Low values of turbidity are desirable. The clear juice turbidity is expected between 700-1500 IU

Color: Low values of color are desirable ICUMSA range 5000-10,000 IU.

The Design-Expert software v 7.0.0 was used to solve this multiple response optimization using the desirability function approach. Set purity change and mud settling rate to be maximum and Turbidity and Color values to a minimum. In addition, the weight and importance of individual responses are set as unity and three respectively.

Table 4.12: Numerical optimization range

Name	**Goal**	**Lower Limit**	**Upper Limit**	**Lower Weight**	**Upper Weight**	**Importance**
Preliming	is in range	8.1	9.9	1	1	2
Sulphitatiom	is in range	6.2	7.8	1	1	3
First heating	is in range	60	80	1	1	3
Second heating	is in range	90	120	1	1	3
purity	maximize	-0.6	2.2	1	1	3
color	minimize	6700	15800	1	1	3
mud settling	maximize	0.005	0.068	1	1	3
turbidity	minimize	640	2500	1	1	3

The result of optimum data analysis using design expert software indicated that six optimum region solutions were obtained by using optimum inputs (pre-liming pH, sulphiting pH, first heating, and second heating temperature) for each response as shown **Table 4.13**.

Table 4.13: Solution of optimization

No.	Preliming	Sulphitatiom	First heating	Second heating	purity	color	mud settling	turbidity	Desirability	
1	9.63	7.16	69.53	101.35	1.65841	6825.78	0.0573208	851.422	0.898	Selected
2	9.63	7.16	69.45	101.52	1.65699	6830.38	0.0573888	851.91	0.898	
3	9.63	7.15	69.72	101.31	1.65864	6824.89	0.0572598	850.378	0.898	
4	9.62	7.15	69.54	101.70	1.65555	6838.21	0.0574227	851.152	0.898	
5	9.62	7.07	70.33	101.25	1.64758	6905.54	0.0570231	839.843	0.896	
6	9.61	7.04	70.30	101.21	1.63966	6943.14	0.0570639	836.987	0.895	

The counter maps of the main responses in a single contour plot are shown in **Figure 4. 8**: The intersection point of two or more optimal counter lines defines the coordinates of the optimum level for each factor.

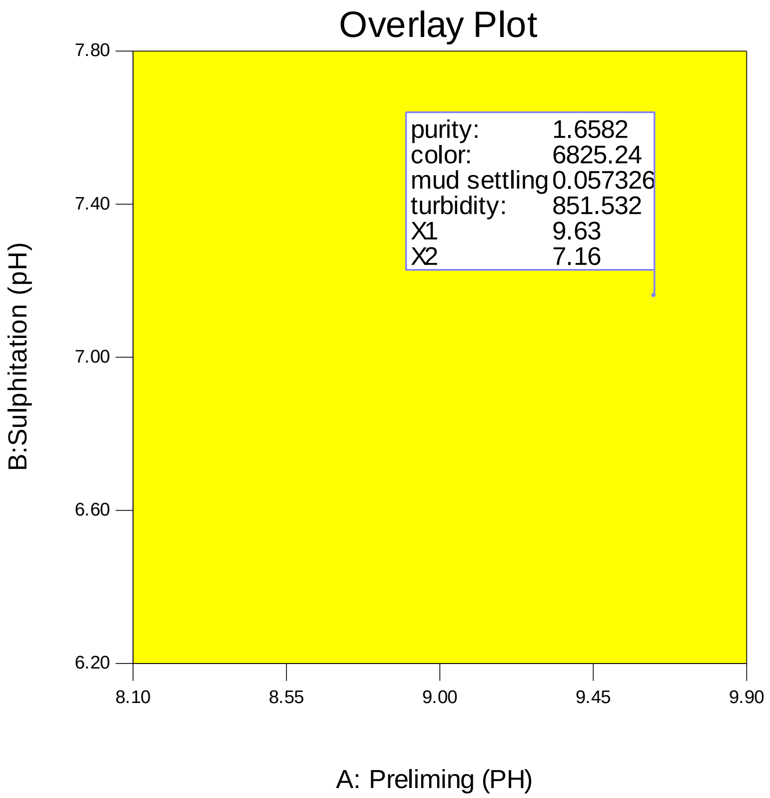


Figure 4. 8: Overlay plot for main response variables

Discussion of Optimization Solution

The optimization solution is a solution that satisfies the goals placed on the study parameters. As shown **Figure 4. 8**: the optimum solution of process chemicals is liming pH of 9.6, sulphiting pH of 7.0, first heating 70.0 ^{0}C, and second heating temperature 101.0 ^{0}C. At optimum chemical solution; purity of clear juice was 1.64%, color was 6943.15 IU, settling rate of mud was 0.057 cm/s, and turbidity of clear juice was 836.976IU.

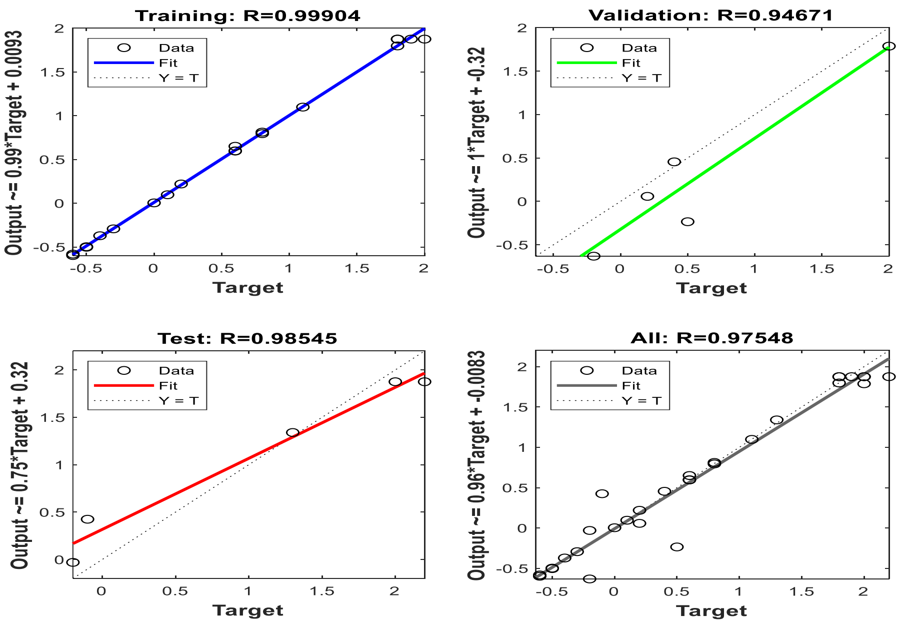
4.3. MODELING OF ANN

4.3.1. Effect of Learning Algorithm and Transfer Function

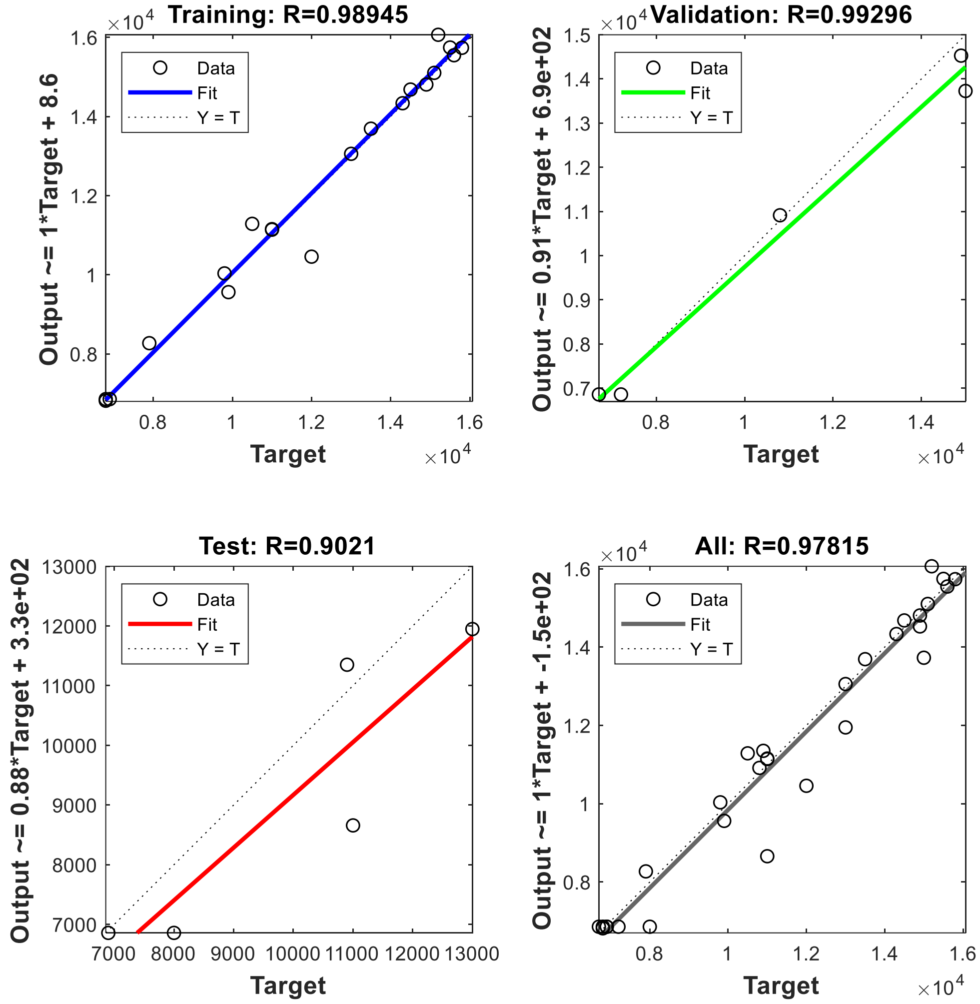
For an effective application, the selection of an optimal neural-network architecture and topology are playing critical importance. Several neural-network architectures and topologies were tested for the estimation and prediction of the clarification performance. Training a neural network model fundamentally means choosing one model from the set of permissible models that diminishes the cost measure. ANN model possesses minimized RMSE and maximized R^2 value, which indicates the suitability of learning algorithm for prediction of clarification efficiency (Selvakumar et al., 2019).

4.3.2. ANN Analysis of sugar cane clarification

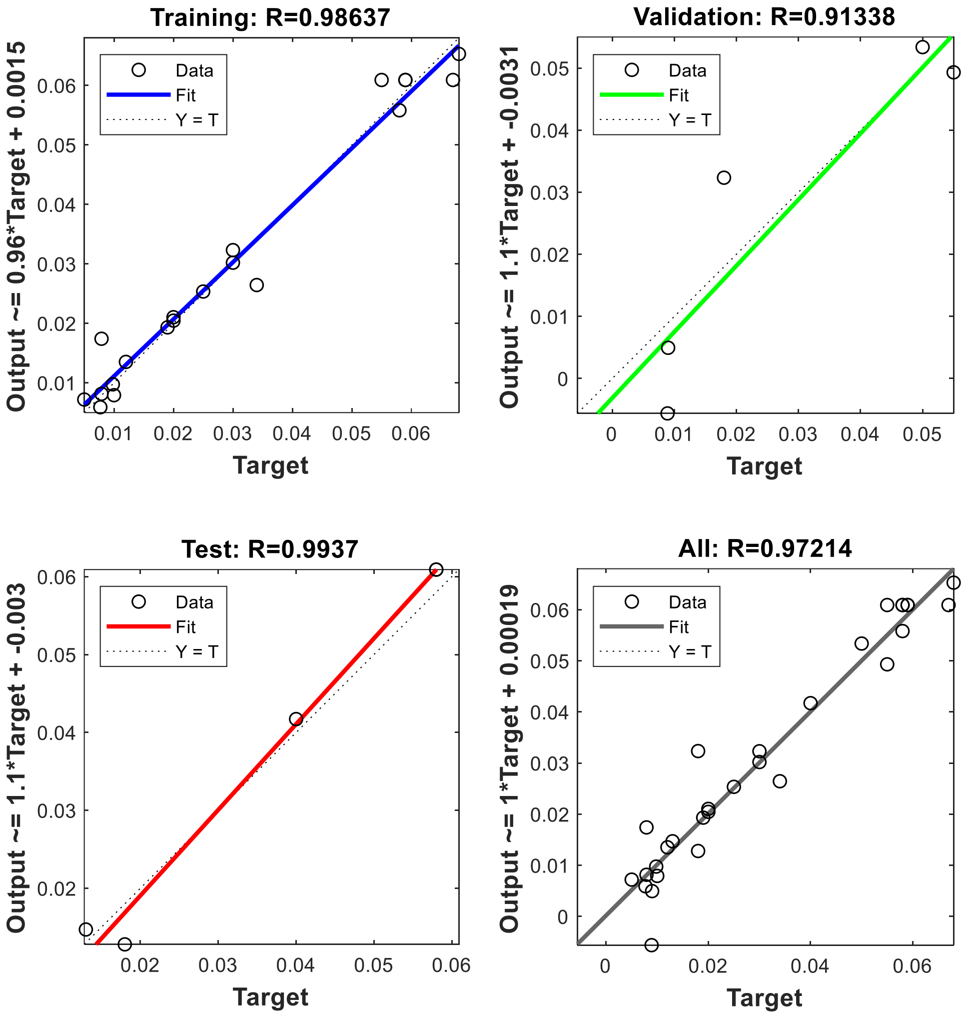
Figure 4. 9: present the evolution of the MSE of the neural network during the training phase. The final point of the training data has a lower performance function of MSE than the set objective, demonstrating an effective result of network training and the improved results used for the weights and biases. The observed correlation coefficient (R^2) of the training, validation and test data set respectively were 0.99, 0.95, and 0.98 for purity change. For the color of clarified juice, R^2 of training, validation, and test data set measured respectively were found to be 0.99, 0.99, and 0.90. For mud settling value of R^2 of training, validation, and test data set measured respectively were found to be 0.99, 0.91, and 0.99. And for turbidity of clear juice was 0.98, 0.96, and 0.58. Experimental and predicted values assessment in these data sets revealed proficiency of ANN. The R^2 value of the ANN model measured were 0.98, 0.98, 0.97, and 0.96 respectively for purity change, color, mud settling, and turbidity of clarified juice. It can be observed from the obtained results that the ANN derived models could be efficiently used to define the relationship between the input and output factors.



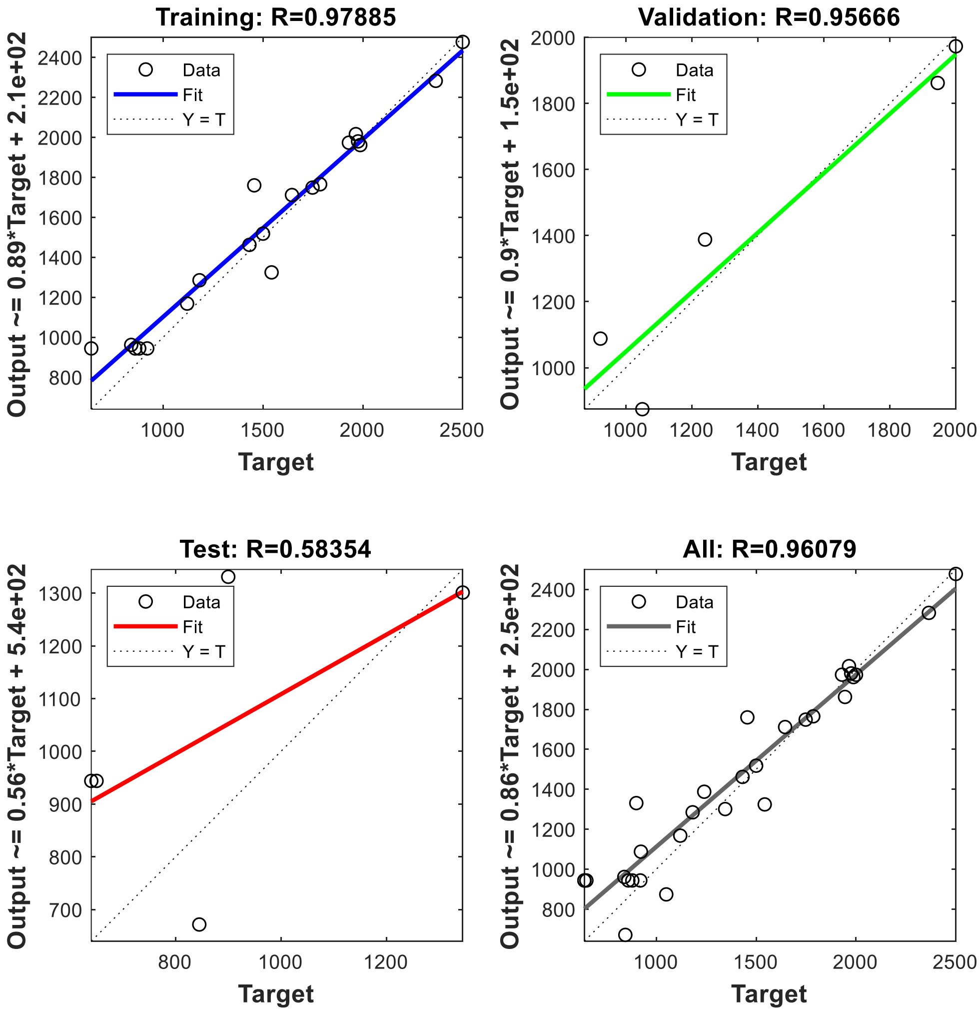
a) Regression graphs for purity change of ANN model



b) Regression graphs for color of juice of ANN model



C) Regression graphs for mud settling of juice of ANN model



d) Regression graphs for Turbidity of juice of ANN model

Figure 4. 9: Regression graphs for clarified juice of ANN model

4.3.3. Comparison of RSM and ANN Modeling

RSM is useful in getting insight information (e.g., interactions between process parameters) of the multivariable system, and similarly, ANN is also found to be an important tool in the sensitivity analysis for further investigation. The results of RSM and ANN modeling were compared to validate their predictive capabilities and optimization efficiency of process parameters. The predicted responses attained from RSM and ANN modeling were similar to the experimental values. The values of R^2, and RMSE, were used together to evaluate the optimization efficiency of RSM and ANN modeling. The comparative values of R^2, and RMSE, obtained from RSM and ANN modeling for clarification of sugar cane juice parameters are presented in **Table 4.14: comparison of RSM and ANN** It can be seen that greater R2 and, minimal RMSE were obtained from the ANN model than RSM for juice clarification. Solutions of optimal conditions from RSM and ANN indicate that the prediction error of the RSM model has more deviation than the prediction made using the ANN model. This means that the experimental data has been well fitted with greater accuracy by using the ANN model than RSM.

Table 4.14: comparison of RSM and ANN

RSM					ANN			
Parameters	Purity change	color	Mud settling	turbidity	Purity change	Color	Mud settling	Turbidity
R^2	0.95	0.96	0.94	0.88	0.98	0.98	0.97	0.96

4.4. **Optimization of parameters using Genetic Algorithm**

The optimal parameter values were searched using a GA Optimization for Excel software. The computation was run using polynomial equations developed from a regression analysis of the dependent and independent variables.

$$P = -144.946 + 12.076 \times L + 22.0419 \times S + 0.956344 \times T_1 - 0.391325 \times T_2 - 0.660603 \times L^2 - 1.5392 \times S^2 - 0.00685087 \times T_1{}^2 + 0.00184405 \times T_2{}^2$$

$$C = 406247 - 4280.04 \times L - 51103.8 \times S - 3851.75 \times T_1 - 1132.28 \times T_2 + 180.853 \times L^2 + 3588.27 \times S^2 + 27.4649 \times T_1{}^2 + 5.53997 \times T_2{}^2$$

$$M = -2.08657 + 0.120378 \times L - 0.161659 \times S + 0.0344032 \times T_1 + 0.0185377 \times T_2 - 0.0064284 \times L^2 + 0.0113953 \times S^2 - 0.00024707 \times T_1{}^2 - 0.00002899 \times T_2{}^2$$

$$T = 60659.2 - 7014.98 \times L - 503.835 \times S - 151.254 \times T_1 - 390.972 \times T2 + 377.427 \times L^2 + 39.4207 \times S^2 0.96714 \times T_1{}^2 + 1.929885 \times T_2{}^2$$

Where L is liming pH; P is purity change in %

S is sulphitation pH; C color of clear juice in IU

T_1 is first heating in ⁰C; M is mud settling in cm/s

T_2 is second heating in ⁰C; T is turbidity in IU

The objective functions were:

- Maximize Purity change (P):$1 \le p \le 2$
- Minimize Color (C): $5000 \le C \le 10{,}000$
- Maximize mud settling (M)
- Minimize Turbidity (T): $700 \le T \le 1500$

Optimum value of factors	Optimum value of Response
Liming (L) =8.5	Purity change (P) =1.93
Sulphitation (S) =6.99 (7.00)	color of clear juice(C) =8990.35 IU
First juice heating (T_1) =69 ^{0}C	Mud settling (M) = 0.037cm/s
Second juice heating (T2) =90 ^{0}C	Turbidity (T) =1381.22 IU

5. CONCLUSION AND RECOMMENDATION

5.1. Conclusion

This work mainly focused on the assessment of chemical dosage (liming and sulphitation) and juice heating temperature to reduce sucrose losses. The pH of treated juice (juice after liming, sulphitation, and heated juice) at WSSF in most cases reached between 6.5 and 7.6 which is out of standard operation due to a lack of proper control of the liming, sulphitation pH, and juice heating (first and second heating temperature) during the clarification process. These problems cause sucrose losses and reduce the quality of the final product. In this view, this thesis aimed at finding the best optimal solution for the juice treatment method.

Consequently, the optimum value for chemicals and juice heating temperature were liming pH 9.6, sulphiting of pH 7.0, the first juice heating temperature of 71.0 ^{0}C, and the second juice heating temperature of 101.0 ^{0}C. At these optimum solutions, purity of 1.64%, the color of 6,943.14 IU, mud settling of 0.057 cm/s, and turbidity of 836.97 IU. The ANN has given a good correlation between the predicted and actual values of the clarification parameter than RSM.

In conclusion, the following response has a negative effect on the clarification performance at existing WSSF:

- Crashing of different cane varieties at a time.
- Improper controlled of process parameters like the desired density of milk of lime, mixing time, stability in the flow of juice, retention time of juice in the clarifier.
- Uncontrolled addition of flocculent i.e. absence of flocculent dosing pump with juice flow.

This study clearly indicates that there can be significant technical potential for minimizing the sucrose losses across the clarification area and increase both quality and productivity of sugar if the optimum condition for each clarification reagents and juice heating temperature is practiced.

5.2. Recommendations

Based on the results obtained from this study, available resources, and practical observations, the following recommendations are suggested:

- ✓ Put in practice the optimum.

- ✓ SO_2 gas line to constant flow and minimize environmental wastage. Due to many leakages of SO_2 Currently, the SO_2 gas line in the plant does not work properly.
- ✓ Apply improved technologies in the sugar production process.
- ✓ Give training and awareness to all workers at clarification stations about how to control and follow up.
- ✓ Optimization of consumable chemical reagents for different cane varieties.
- ✓ Investigate the effect of different flocculent and dosing type on clarification performance.

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APPENDICES

Appendix 1: Table and Figure for response variable

A: purity change

Table 1: fit summery for purity change of clear juice

Std. Dev.	850.64	R-Squared	0.9638
Mean	11480.00	Adj R-Squared	0.9301
C.V. %	7.41	Pred R-Squared	0.8422
PRESS	4.737E+007	Adeq Precision	15.298

Table 2: Lack of Fit Tests for purity change

Source	Sum of Squares	Df	Mean Square	F Value	P-value Prob > F	
Linear	2.711E+008	20	1.355E+007	59.01	0.0001	
2FI	2.539E+008	14	1.813E+007	78.96	< 0.0001	
Quadratic	9.706E+006	10	9.706E+005	4.23	0.0626	Suggested
Cubic	4.120E+006	2	2.060E+006	8.97	0.0222	Aliased
Pure Error	1.148E+006	5	2.297E+005			

Table 3: Model Summary Statistics for purity change

Source	Std Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	3299.69	0.0932	-0.0518	-0.2807	3.844E+008	
2FI	3663.70	0.1504	-0.2967	-2.0792	9.244E+008	
Quadratic	850.64	0.9638	0.9301	0.8422	4.737E+007	Suggested
Cubic	867.54	0.9824	0.9273	-0.6530	4.962E+008	Aliased

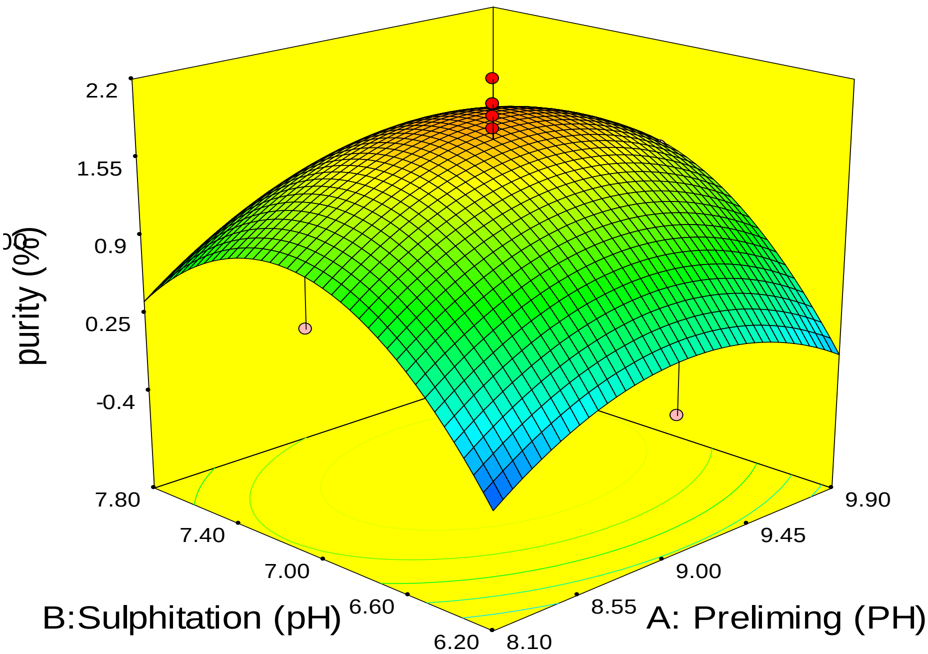

Figure 1: 3D surface graph for purity change

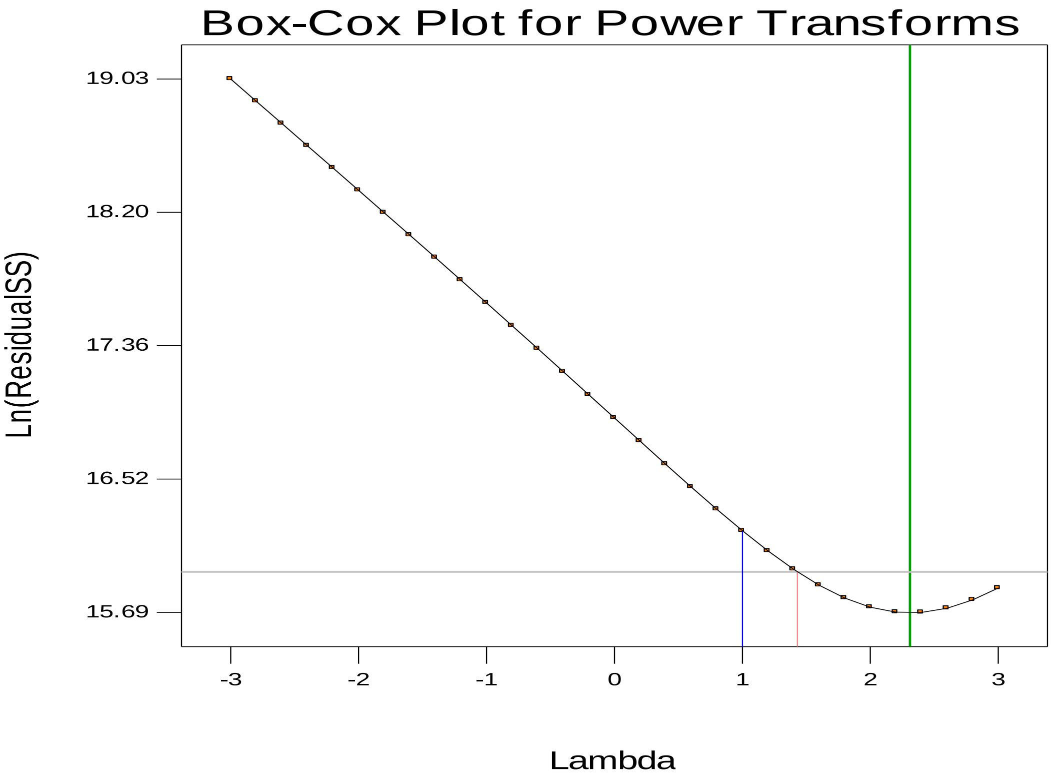

Fig 2: Box-Cox plot

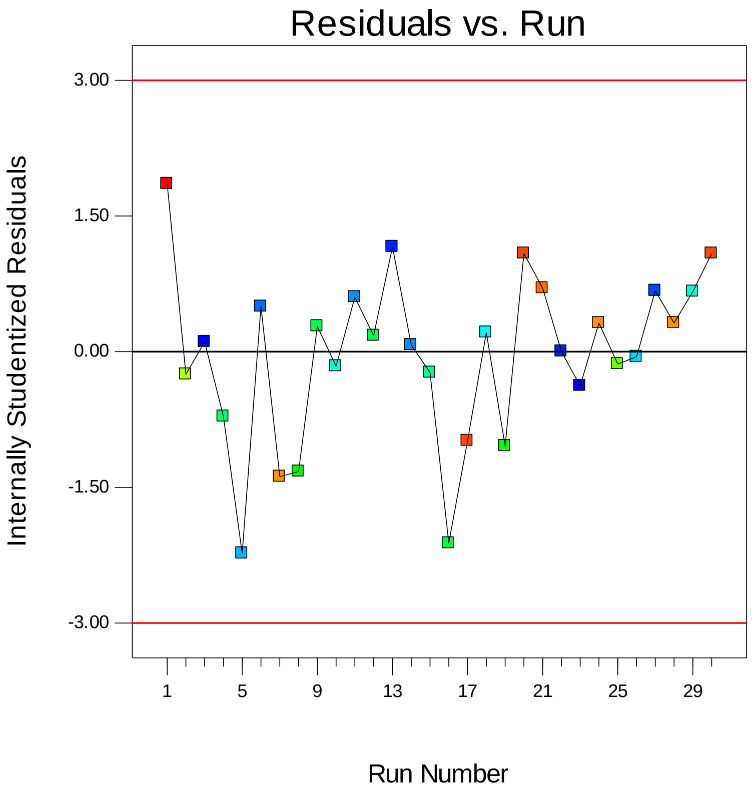

Figure 3: Residual vs run Plot for purity change

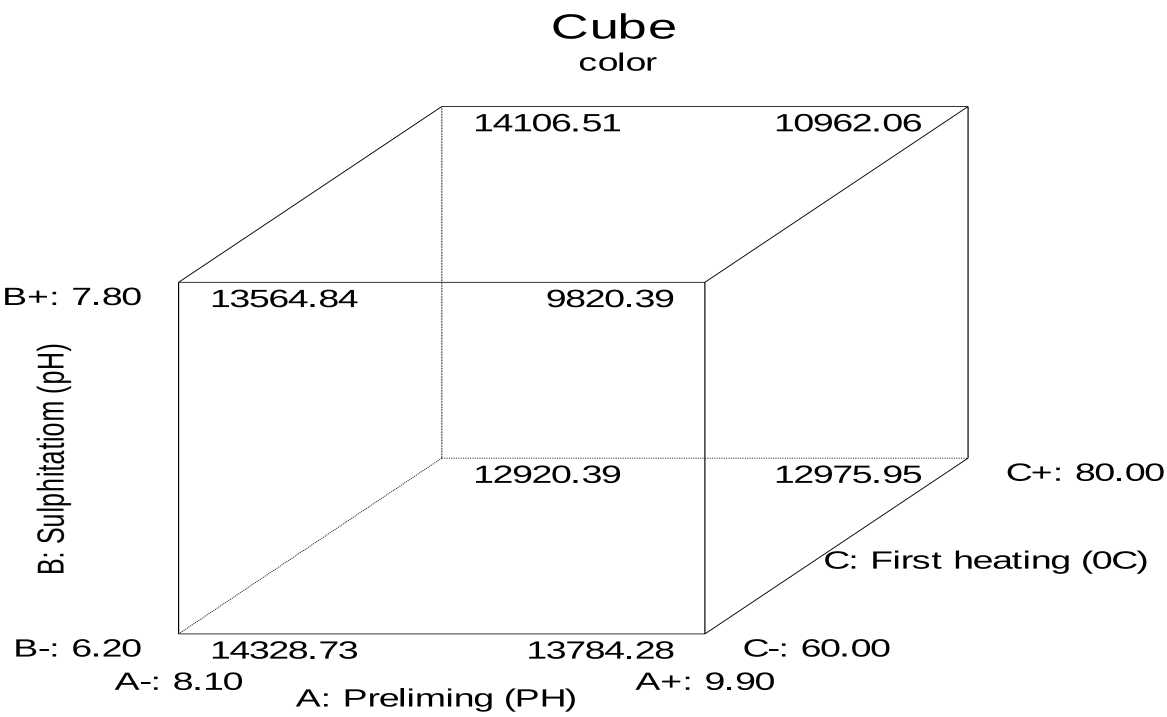

Figure 4: Cubic Plot for purity change

Appendix 2: Table and Figure for response variable

B). Color

Table 4: fit summery for color of clear juice

Source	Sum of Squares	Df	Mean Square	F Value	P-value Prob > F	
Linear	2.711E+008	20	1.355E+007	59.01	0.0001	
2FI	2.539E+008	14	1.813E+007	78.96	< 0.0001	
Quadratic	9.706E+006	10	9.706E+005	4.23	0.0626	Suggested
Cubic	4.120E+006	2	2.060E+006	8.97	0.0222	Aliased

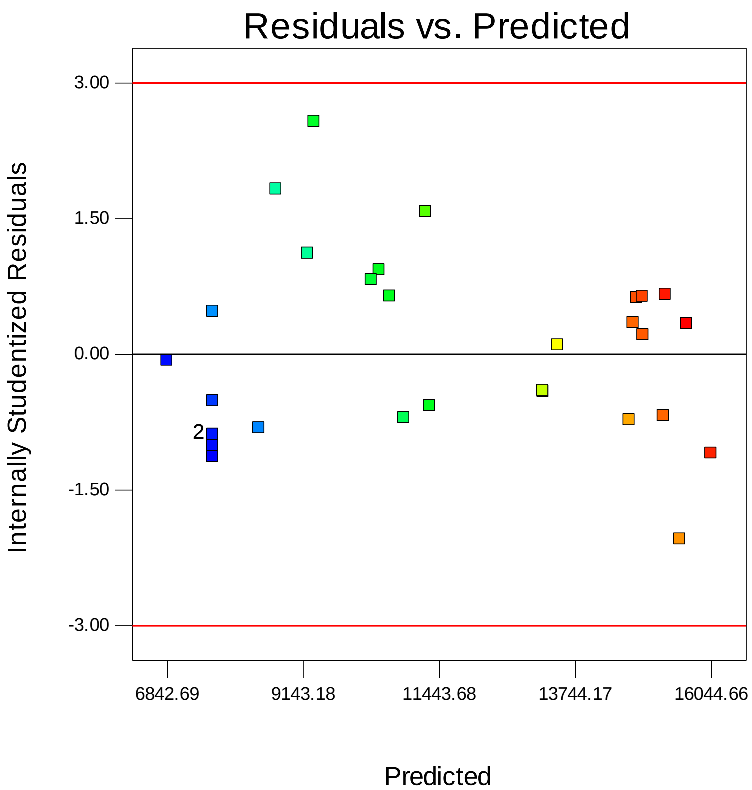

Fig 5: Residual vs .predicated plot of color of clear juice

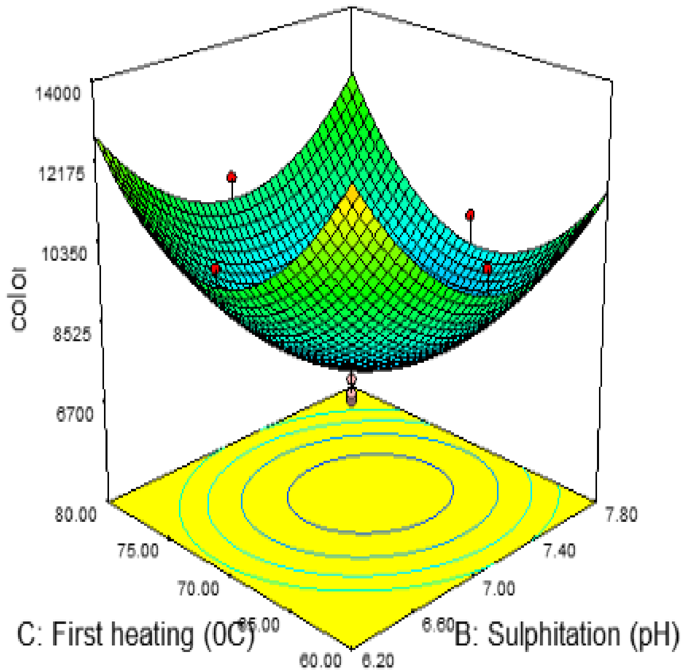

Figure 6: Three-Dimensional Response Surfaces Plot for color of clear juice

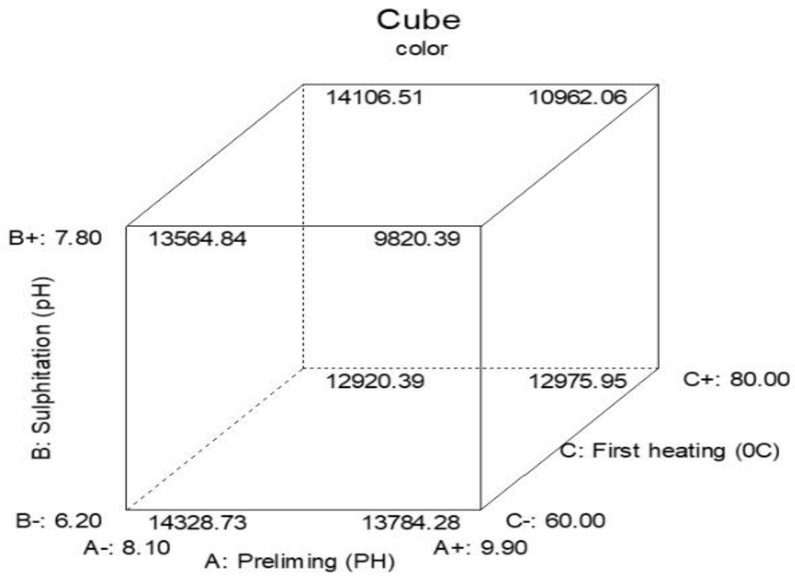

Figure 7: Cubic Plot for color

Appendix 3: Table and Figure for response variable

C) Mud settling

Table 5: Sequential Model Sum of Squares for mud settling

Source	Sum of Squares	df	Mean Square	F Value	P-value Prob > F	
Mean vs Total	0.029	1	0.029			
Linear vs Mean	8.528E-004	4	2.132E-004	0.42	0.7944	
2FI vs Linear	2.690E-004	6	4.483E-005	0.068	0.9984	
Quadratic vs 2FI	0.012	4	2.934E-003	57.84	< 0.0001	Suggested
Cubic vs Quadratic	5.852E-004	8	7.315E-005	2.91	0.0882	Aliased
Residual	1.757E-004	7	2.510E-005			
Total	0.043	30	1.420E-003			

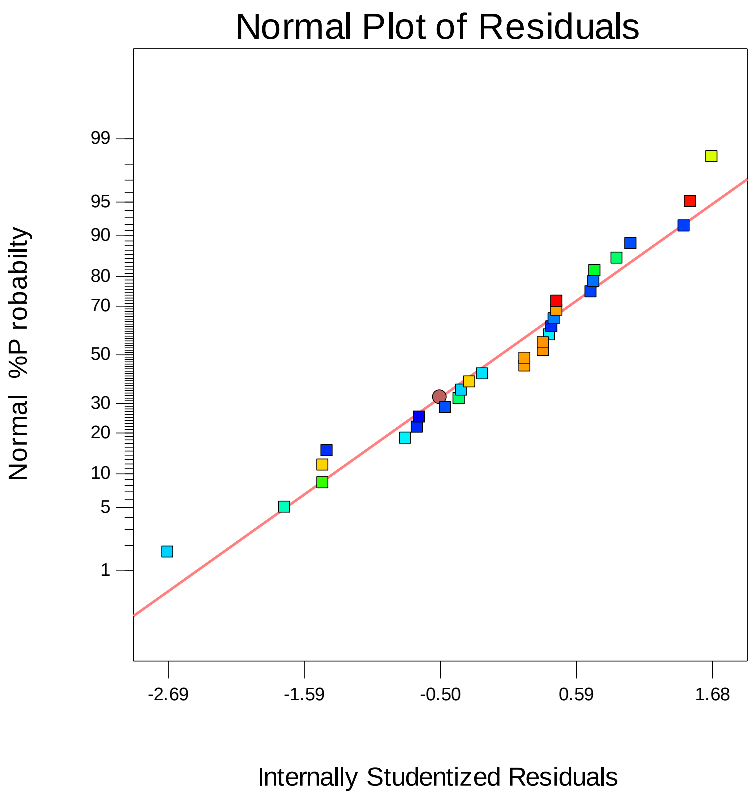

Figure 8: Normal plot of Residuals of mud settling

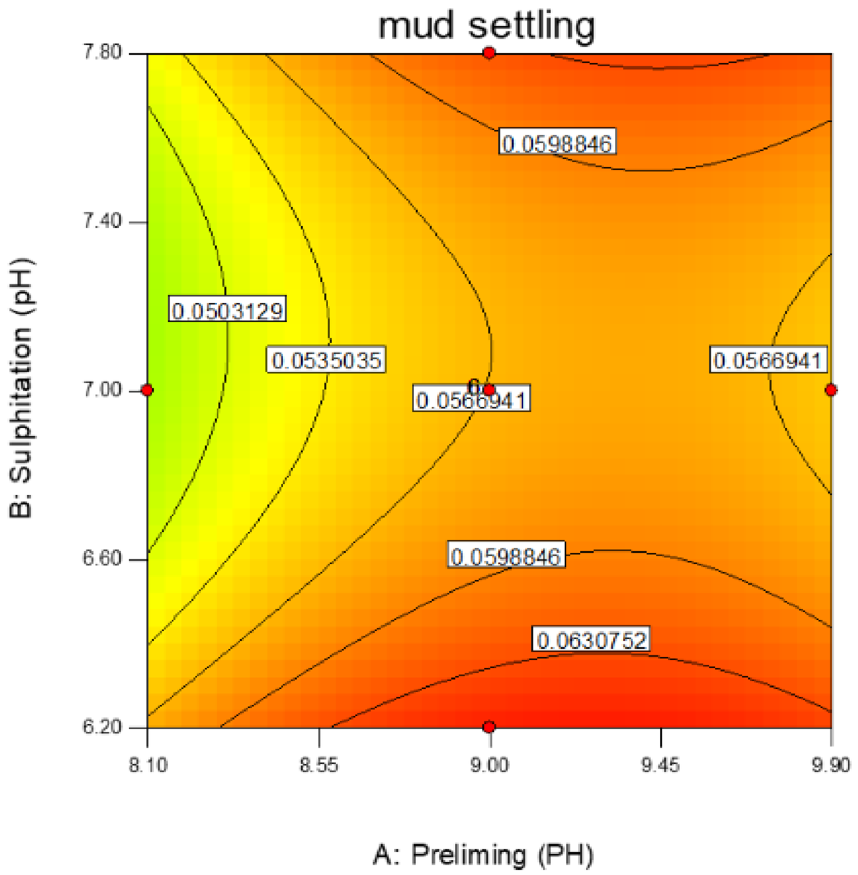

Figure 9: contour plots for mud settling

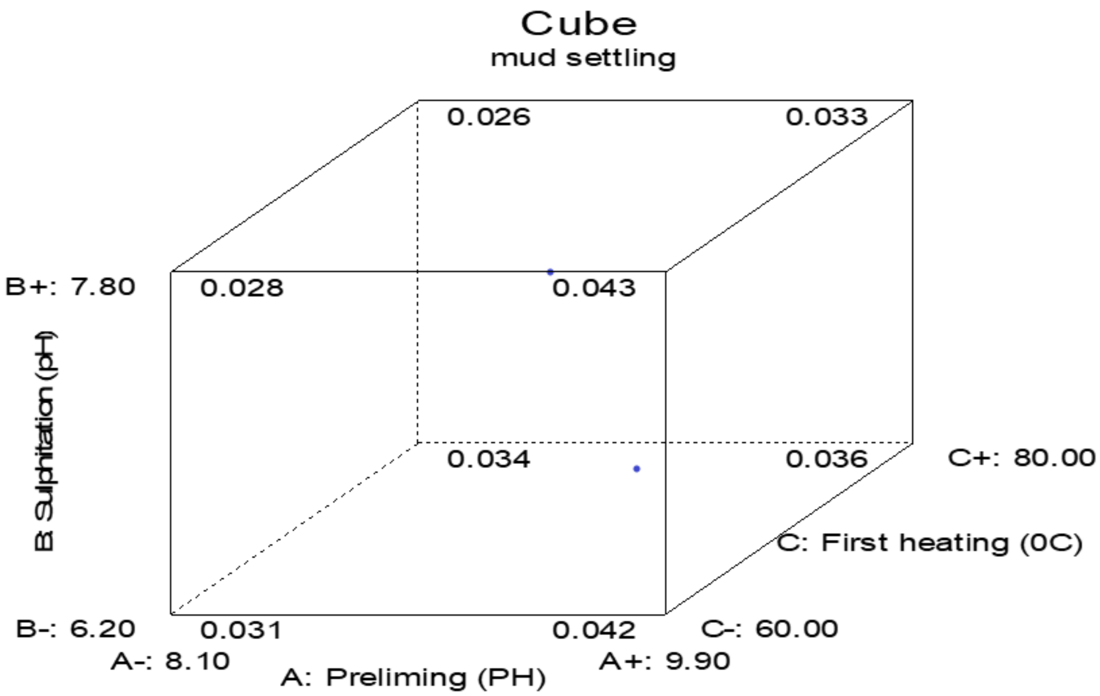

Figure 9 : Cubic Plot for mud settling

Appendix 4: Table and Figure for response variable

D) Turbidity

Table 6: Sequential Model Sum of Squares for Turbidity

Source	Sum of Squares	df	Mean Square	F Value	P-value Prob > F	
Mean vs Total	5.828E+007	1	5.828E+007			
Linear vs Mean	2.021E+006	4	5.051E+005	2.00	0.1249	
2FI vs Linear	9.297E+005	6	1.549E+005	0.55	0.7656	
Quadratic vs 2FI	4.447E+006	4	1.112E+006	17.98	< 0.0001	Suggested
Cubic vs Quadratic	6.404E+005	8	80050.05	1.95	0.1969	Aliased
Residual	2.872E+005	7	41027.63			
Total	6.660E+007	30	2.220E+006			

Table 7: Model Summary Statistics

Source	Std Dev.	R-Squared	Adjusted R-Squared	Predicated R-Squared	PRESS	
Linear	502.16	0.2427	0.1216	-0.0854	9.036E+006	
2FI	531.85	0.3544	0.0146	-1.2279	1.855E+007	
Quadratic	248.68	0.8886	0.7846	0.4693	4.418E+006	Suggested
Cubic	202.55	0.9655	0.8571	-2.1086	2.588E+007	Aliased

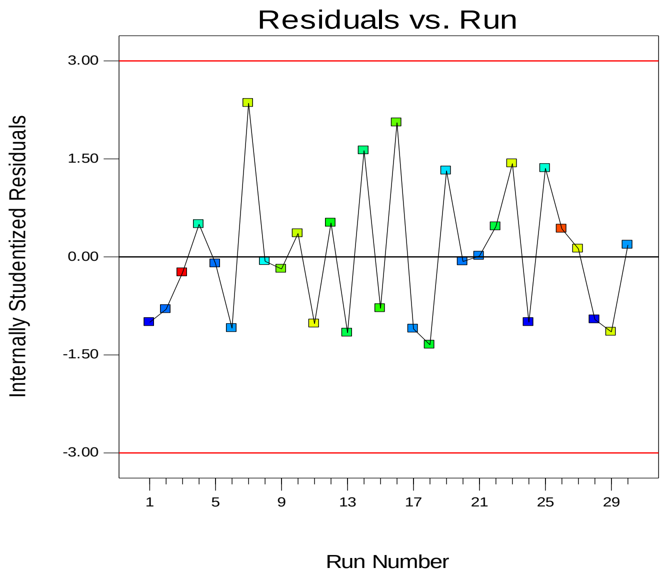

Figure 10: Residuals vs. Run plot of turbidity of clear juice

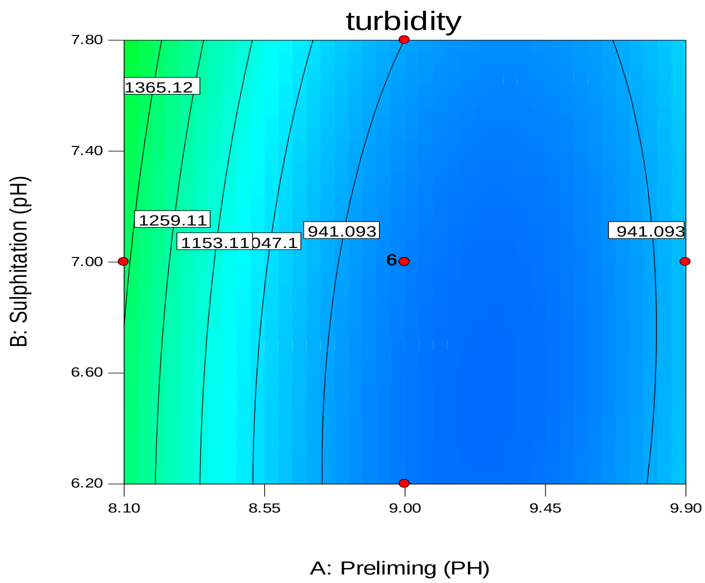

Figure 11: Response surface plot for turbidity of clear juice

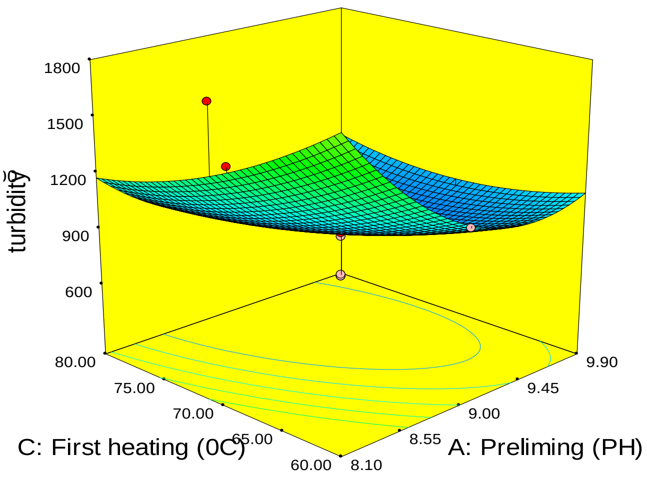

Figure 12: Three-Dimensional Response Surfaces Plot for Turbidity of clear juice

Appendix 5: pol or sucrose factors according to refractometer solids (brix values) to obtain pol multiply the saccharimeter reading obtained from dry lead subacetate clarification by the factor corresponding to the refractometer solids. To obtain sucrose multiply the calculated sucrose reading by the factor:

Pol factor = 26/ (99.718*ap sp gr at20ºC/20ºC)

Example

Saccharimeter reading = 41.5

Refractometer solids = 11.50

Pol = 41.5 x 0.24920 = 10.34

Ref	Pol factor									
Sol.	0	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	0.2606	0.26059	0.26057	0.26056	0.26055	0.26054
0.2	0.26053	0.26052	0.26051	0.2605	0.26049	0.26048	0.26047	0.26046	0.26045	0.26044
0.3	0.26043	0.26042	0.26041	0.2604	0.26039	0.26038	0.26037	0.26036	0.26035	0.26034
0.4	0.26033	0.26032	0.26031	0.2603	0.26029	0.26028	0.26027	0.26026	0.26025	0.26024
0.5	0.26023	0.26022	0.26021	0.2602	0.26019	0.26018	0.26017	0.26016	0.26015	0.26014
0.6	0.26013	0.26012	0.26011	0.2601	0.26009	0.26008	0.26007	0.26006	0.26005	0.26004
0.7	0.26003	0.26002	0.26001	0.26	0.25999	0.25997	0.25996	0.25995	0.25994	0.25993
0.8	0.25992	0.25991	0.2599	0.25989	0.25988	0.25987	0.25986	0.25985	0.25984	0.25983
0.9	0.25982	0.25981	0.2598	0.25979	0.25978	0.25977	0.25976	0.25975	0.25974	0.25973
1	0.25972	0.25971	0.2597	0.25969	0.25968	0.25967	0.25966	0.25965	0.25964	0.25963
1.1	0.25962	0.25961	0.2596	0.25959	0.25958	0.25957	0.25956	0.25955	0.25954	0.25953

1.2	0.25952	0.25951	0.2595	0.25949	0.25948	0.25947	0.25946	0.25945	0.25944	0.25943
1.3	0.25942	0.25941	0.2594	0.25939	0.25938	0.25937	0.25936	0.25935	0.25934	0.25933
1.4	0.25932	0.25931	0.2593	0.25929	0.25928	0.25927	0.25926	0.25925	0.25924	0.25923
1.5	0.25922	0.25921	0.2592	0.25919	0.25918	0.25917	0.25916	0.25915	0.25914	0.25913
1.6	0.25912	0.25911	0.2591	0.25909	0.25908	0.25907	0.25906	0.25905	0.25904	0.25903
1.7	0.25902	0.25901	0.259	0.25899	0.25898	0.25897	0.25896	0.25895	0.25894	0.25893
1.8	0.25892	0.25891	0.2589	0.25889	0.25888	0.25887	0.25886	0.25885	0.25884	0.25883
1.9	0.25882	0.25881	0.2588	0.25879	0.25878	0.25877	0.25876	0.25875	0.25874	0.25873
2	0.25872	0.25871	0.2587	0.25869	0.25868	0.25867	0.25866	0.25865	0.25864	0.25863
2.1	0.25862	0.25861	0.2586	0.25859	0.25858	0.25857	0.25856	0.25855	0.25854	0.25853
2.2	0.25852	0.25851	0.2585	0.25849	0.25848	0.25846	0.25845	0.25844	0.25843	0.25842
2.3	0.25841	0.2584	0.25839	0.25838	0.25837	0.25836	0.25835	0.25834	0.25833	0.25832
2.4	0.25831	0.2583	0.25829	0.25828	0.25827	0.25826	0.25825	0.25824	0.25823	0.25822
2.5	0.25821	0.2582	0.25819	0.25818	0.25817	0.25816	0.25815	0.25814	0.25813	0.25812
2.6	0.25811	0.2581	0.25809	0.25808	0.25807	0.25806	0.25805	0.25804	0.25803	0.25802
2.7	0.25801	0.258	0.25799	0.25798	0.25797	0.25796	0.25795	0.25794	0.25793	0.25792
2.8	0.25791	0.2579	0.25789	0.25788	0.25787	0.25786	0.25785	0.25784	0.25783	0.25782
2.9	0.25781	0.2578	0.25779	0.25778	0.25777	0.25776	0.25775	0.25774	0.25773	0.25772
3	0.25771	0.2577	0.25769	0.25768	0.25767	0.25766	0.25765	0.25764	0.25763	0.25762
3.1	0.25761	0.2576	0.25759	0.25758	0.25757	0.25756	0.25755	0.25754	0.25753	0.25752
3.2	0.25751	0.2575	0.25749	0.25748	0.25747	0.25746	0.25745	0.25744	0.25743	0.25742
3.3	0.25741	0.2574	0.25739	0.25738	0.25737	0.25736	0.25735	0.25734	0.25733	0.25732
3.4	0.25731	0.2573	0.25729	0.25728	0.25727	0.25726	0.25725	0.25724	0.25723	0.25722
3.5	0.25721	0.2572	0.25719	0.25718	0.25717	0.25716	0.25715	0.25714	0.25713	0.25712
3.6	0.25711	0.2571	0.25709	0.25708	0.25707	0.25706	0.25705	0.25704	0.25703	0.25702
3.7	0.25701	0.257	0.25699	0.25698	0.25697	0.25696	0.25695	0.25694	0.25693	0.25692
3.8	0.25691	0.2569	0.25689	0.25688	0.25687	0.25686	0.25685	0.25684	0.25683	0.25682
3.9	0.25681	0.2568	0.25679	0.25678	0.25677	0.25676	0.25675	0.25674	0.25673	0.25672
4	0.25671	0.2567	0.25669	0.25668	0.25667	0.25666	0.25665	0.25664	0.25663	0.25662
4.1	0.25661	0.2566	0.25659	0.25658	0.25657	0.25656	0.25655	0.25654	0.25653	0.25652
4.2	0.25651	0.2565	0.25649	0.25648	0.25647	0.25746	0.25645	0.25644	0.25643	0.25642
4.3	0.25641	0.2564	0.25639	0.25638	0.25637	0.25636	0.25635	0.25634	0.25633	0.25632
4.4	0.25631	0.2563	0.25629	0.25628	0.25627	0.25626	0.25625	0.25624	0.25623	0.25622
4.5	0.25621	0.2562	0.25619	0.25618	0.25617	0.25616	0.25615	0.25614	0.25613	0.25612
4.6	0.2561	0.25609	0.25608	0.25607	0.25606	0.25605	0.25604	0.25603	0.25602	0.25601
4.7	0.256	0.25599	0.25598	0.25597	0.25596	0.25595	0.25594	0.25593	0.25592	0.25591

4.8	0.2559	0.25589	0.25588	0.25587	0.25586	0.25585	0.25584	0.25583	0.25582	0.25581
4.9	0.2558	0.25579	0.25578	0.25577	0.25576	0.25575	0.25574	0.25573	0.25572	0.25571
5	0.2557	0.25569	0.25568	0.25567	0.25566	0.25565	0.25564	0.25563	0.25562	0.25561
5.1	0.2556	0.25559	0.25558	0.25557	0.25556	0.25555	0.25554	0.25553	0.25552	0.25551
5.2	0.2555	0.25549	0.25548	0.25547	0.25546	0.25545	0.25544	0.25543	0.25542	0.25541
5.3	0.2554	0.25539	0.25538	0.25537	0.25536	0.25535	0.25534	0.25533	0.25532	0.25531
5.4	0.2553	0.25529	0.25528	0.25527	0.25526	0.25525	0.25524	0.25523	0.25522	0.25521
5.5	0.2552	0.25519	0.25518	0.25517	0.25516	0.25515	0.25514	0.25513	0.25512	0.25511
5.6	0.2551	0.25509	0.25508	0.25507	0.25506	0.25505	0.25504	0.25503	0.25502	0.25501
5.7	0.255	0.25499	0.25498	0.25497	0.25496	0.25495	0.25494	0.25493	0.25492	0.25491
5.8	0.2549	0.25489	0.25488	0.25487	0.25486	0.25485	0.25484	0.25483	0.25482	0.25481
5.9	0.2548	0.25479	0.25478	0.25477	0.25476	0.25475	0.25474	0.25473	0.25472	0.25471
6	0.2547	0.25469	0.25468	0.25467	0.25466	0.25465	0.25464	0.25463	0.25462	0.25461
6.1	0.2546	0.25459	0.25458	0.25457	0.25456	0.25455	0.25454	0.25453	0.25452	0.25451
6.2	0.2545	0.25449	0.25448	0.25447	0.25446	0.25445	0.25444	0.25443	0.25442	0.25441
6.3	0.2544	0.25439	0.25438	0.25437	0.25436	0.25435	0.25434	0.25433	0.25432	0.25431
6.4	0.2543	0.25429	0.25428	0.25427	0.25426	0.25425	0.25424	0.25423	0.25422	0.25421
6.5	0.2542	0.25419	0.25418	0.25417	0.25416	0.25415	0.25414	0.25413	0.25412	0.25411
6.6	0.2541	0.25409	0.25408	0.25407	0.25406	0.25405	0.25404	0.25403	0.25402	0.25401
6.7	0.254	0.25399	0.25398	0.25397	0.25396	0.25395	0.25394	0.25393	0.25392	0.25391
6.8	0.2539	0.25389	0.25388	0.25387	0.25386	0.25385	0.25384	0.25383	0.25382	0.25381
6.9	0.2538	0.25379	0.25378	0.25377	0.25376	0.25375	0.25374	0.25373	0.25372	0.25371
7	0.2537	0.25369	0.25368	0.25367	0.25366	0.25365	0.25364	0.25363	0.25362	0.25361
7.1	0.2536	0.25359	0.25358	0.25357	0.25356	0.25355	0.25354	0.25353	0.25352	0.25351
7.2	0.2535	0.25349	0.25348	0.25347	0.25346	0.25746	0.25344	0.25343	0.25342	0.25341
7.3	0.2534	0.25339	0.25338	0.25337	0.25336	0.25335	0.25334	0.25333	0.25332	0.25331
7.4	0.2533	0.25329	0.25328	0.25327	0.25326	0.25325	0.25324	0.25323	0.25322	0.25321
7.5	0.2532	0.25319	0.25318	0.25317	0.25316	0.25315	0.25314	0.25313	0.25312	0.25311
7.6	0.2531	0.25309	0.25308	0.25307	0.25306	0.25305	0.25304	0.25303	0.25302	0.25301
7.7	0.253	0.25299	0.25298	0.25297	0.25296	0.25295	0.25294	0.25293	0.25292	0.25291
7.8	0.2529	0.25289	0.25288	0.25287	0.25286	0.25285	0.25284	0.25283	0.25282	0.25281
7.9	0.2528	0.25279	0.25278	0.25277	0.25276	0.25275	0.25274	0.25273	0.25272	0.25271
8	0.2527	0.25269	0.25268	0.25267	0.25266	0.25265	0.25264	0.25263	0.25262	0.25261
8.1	0.2526	0.25259	0.25258	0.25257	0.25256	0.25255	0.25254	0.25253	0.25252	0.25251
8.2	0.2525	0.25249	0.25248	0.25247	0.25246	0.25245	0.25244	0.25243	0.25242	0.25241

8.3	0.2524	0.25239	0.25238	0.25237	0.25236	0.25235	0.25234	0.25233	0.25232	0.25231
8.4	0.2523	0.25229	0.25228	0.25227	0.25226	0.25225	0.25224	0.25223	0.25222	0.25221
8.5	0.2522	0.25219	0.25218	0.25217	0.25216	0.25215	0.25214	0.25213	0.25212	0.25211
8.6	0.2521	0.25209	0.25208	0.25207	0.25206	0.25205	0.25204	0.25203	0.25202	0.25201
8.7	0.252	0.25199	0.25198	0.25197	0.25196	0.25195	0.25194	0.25193	0.25192	0.25191
8.8	0.2519	0.25189	0.25188	0.25187	0.25186	0.25185	0.25184	0.25183	0.25182	0.25181
8.9	0.2518	0.25179	0.25178	0.25177	0.25176	0.25175	0.25174	0.25173	0.25172	0.25171
9	0.2517	0.25169	0.25168	0.25167	0.25166	0.25165	0.25164	0.25163	0.25162	0.25161
9.1	0.2516	0.25159	0.25158	0.25157	0.25156	0.25155	0.25154	0.25153	0.25152	0.25151
9.2	0.2515	0.25149	0.25148	0.25147	0.25146	0.25145	0.25144	0.25143	0.25142	0.25141
9.3	0.2514	0.25139	0.25138	0.25137	0.25136	0.25135	0.25134	0.25133	0.25132	0.25131
9.4	0.2513	0.25129	0.25128	0.25127	0.25126	0.25125	0.25124	0.25123	0.25122	0.25121
9.5	0.2512	0.25119	0.25118	0.25117	0.25116	0.25115	0.25114	0.25113	0.25112	0.25111
9.6	0.2511	0.25109	0.25108	0.25107	0.25106	0.25105	0.25104	0.25103	0.25102	0.25101
9.7	0.251	0.25099	0.25098	0.25097	0.25096	0.25095	0.25094	0.25093	0.25092	0.25091
9.8	0.2509	0.25089	0.25088	0.25087	0.25086	0.25085	0.25084	0.25083	0.25082	0.25081
9.9	0.2508	0.25079	0.25078	0.25077	0.25076	0.25075	0.25074	0.25073	0.25072	0.25071
10	0.2507	0.25069	0.25068	0.25067	0.25066	0.25065	0.25064	0.25063	0.25062	0.25061
10.1	0.2506	0.25059	0.25058	0.25057	0.25056	0.25055	0.25054	0.25053	0.25052	0.25051
10.2	0.2505	0.25049	0.25048	0.25047	0.25046	0.25045	0.25044	0.25043	0.25042	0.25041
10.3	0.2504	0.25039	0.25038	0.25037	0.25036	0.25035	0.25034	0.25033	0.25032	0.25031
10.4	0.2503	0.25029	0.25028	0.25027	0.25026	0.25025	0.25024	0.25023	0.25022	0.25021
10.5	0.2502	0.25019	0.25018	0.25017	0.25016	0.25015	0.25014	0.25013	0.25012	0.25011
10.6	0.2501	0.25009	0.25008	0.25007	0.25006	0.25005	0.25004	0.25003	0.25002	0.25001
10.7	0.25	0.24999	0.24998	0.24997	0.24996	0.24995	0.24994	0.24993	0.24992	0.24991
10.8	0.2499	0.24989	0.24988	0.24987	0.24986	0.24985	0.24984	0.24983	0.24982	0.24981
10.9	0.2498	0.24979	0.24978	0.24977	0.24976	0.24975	0.24974	0.24973	0.24972	0.24971
11	0.2497	0.24969	0.24968	0.24967	0.24966	0.24965	0.24964	0.24963	0.24962	0.24961
11.1	0.2496	0.24959	0.24958	0.24957	0.24956	0.24955	0.24954	0.24953	0.24952	0.24951
11.2	0.2495	0.24949	0.24948	0.24947	0.24946	0.25746	0.24944	0.24943	0.24942	0.24941
11.3	0.2494	0.24939	0.24938	0.24937	0.24936	0.24935	0.24934	0.24933	0.24932	0.24931
11.4	0.2493	0.24929	0.24928	0.24927	0.24926	0.24925	0.24924	0.24923	0.24922	0.24921
11.5	0.2492	0.24919	0.24918	0.24917	0.24916	0.24915	0.24914	0.24913	0.24912	0.24911
11.6	0.2491	0.24909	0.24908	0.24907	0.24906	0.24905	0.24904	0.24903	0.24902	0.24901
11.7	0.249	0.24899	0.24898	0.24897	0.24896	0.24895	0.24894	0.24893	0.24892	0.24891
11.8	0.2489	0.24889	0.24888	0.24887	0.24886	0.24885	0.24885	0.24884	0.24883	0.24882
11.9	0.24881	0.2488	0.24879	0.24878	0.24877	0.24876	0.24875	0.24874	0.24873	0.24872

12	0.24871	0.2487	0.24869	0.24868	0.24867	0.24866	0.24865	0.24864	0.24863	0.24862
12.1	0.24861	0.2486	0.24859	0.24858	0.24857	0.24856	0.24855	0.24854	0.24853	0.24852
12.2	0.24851	0.2485	0.24849	0.24848	0.24847	0.25746	0.24845	0.24844	0.24843	0.24842
12.3	0.24841	0.2484	0.24839	0.24838	0.24837	0.24836	0.24835	0.24834	0.24833	0.24832
12.4	0.24831	0.2483	0.24829	0.24828	0.24827	0.24826	0.24825	0.24824	0.24823	0.24822
12.5	0.24821	0.2482	0.24819	0.24818	0.24817	0.24816	0.24815	0.24814	0.24813	0.24812
12.6	0.24811	0.2481	0.24809	0.24808	0.24807	0.24806	0.24805	0.24804	0.24803	0.24802
12.7	0.24801	0.248	0.24799	0.24798	0.24797	0.24796	0.24795	0.24794	0.24793	0.24792
12.8	0.24791	0.2479	0.24789	0.24788	0.24787	0.24786	0.24785	0.24784	0.24783	0.24782
12.9	0.24781	0.2478	0.24779	0.24778	0.24777	0.24776	0.24775	0.24774	0.24773	0.24772
13	0.24771	0.2477	0.24769	0.24768	0.24767	0.24766	0.24765	0.24764	0.24763	0.24762
13.1	0.24761	0.2476	0.24759	0.24758	0.24757	0.24756	0.24755	0.24754	0.24753	0.24752
13.2	0.24751	0.2475	0.24749	0.24748	0.24747	0.25746	0.24745	0.24744	0.24743	0.24742
13.3	0.24741	0.2474	0.24739	0.24738	0.24737	0.24736	0.24735	0.24734	0.24733	0.24732
13.4	0.24731	0.2473	0.24729	0.24728	0.24727	0.24726	0.24725	0.24724	0.24723	0.24722
13.5	0.24721	0.2472	0.24719	0.24718	0.24717	0.24716	0.24715	0.24714	0.24713	0.24712
13.6	0.24711	0.2471	0.24709	0.24708	0.24707	0.24706	0.24705	0.24704	0.24703	0.24702
13.7	0.24701	0.247	0.24699	0.24698	0.24697	0.24696	0.24695	0.24694	0.24693	0.24692
13.8	0.24691	0.2469	0.24689	0.24688	0.24687	0.24686	0.24685	0.24684	0.24683	0.24682
13.9	0.24681	0.2468	0.24679	0.24678	0.24677	0.24676	0.24675	0.24674	0.24673	0.24672
14	0.24671	0.2467	0.24969	0.24668	0.24667	0.24666	0.24665	0.24664	0.24663	0.24662
14.1	0.24661	0.2466	0.24959	0.24658	0.24657	0.24656	0.24655	0.24654	0.24653	0.24652
14.2	0.24651	0.2465	0.24949	0.24648	0.24647	0.25746	0.25746	0.24645	0.24644	0.24643
14.3	0.24642	0.24641	0.2464	0.24639	0.24638	0.24637	0.24636	0.24635	0.24634	0.24633
14.4	0.24632	0.24631	0.2463	0.24629	0.24628	0.24627	0.24626	0.24625	0.24624	0.24623
14.5	0.24622	0.24621	0.2462	0.24619	0.24618	0.24617	0.24616	0.24615	0.24614	0.24613
14.6	0.24612	0.24611	0.2461	0.24609	0.24608	0.24607	0.24606	0.24605	0.24604	0.24603
14.7	0.24602	0.24601	0.246	0.24599	0.24598	0.24597	0.24596	0.24595	0.24594	0.24593
14.8	0.24592	0.24591	0.2459	0.24589	0.24588	0.24587	0.24586	0.24585	0.24584	0.24583
14.9	0.24582	0.24581	0.2458	0.24579	0.24578	0.24577	0.24576	0.24575	0.24574	0.24573

Table 6 Handbook of laboratory methods and chemical control for Ethiopian sugar factories.							
Degrees Brix, True density and s.g, of sugar solutions							
Degrees Brix or percentage of sucrose by weight	**Specific gravity at $20^0/4^0C$**	**Specific gravity at$20^0/20^0C$**	**GMS of Sucrose per 100 ml weight in Vacuo**	**Degrees Brix or percentage of sucrose by weight**	**Specific gravity at $20^0/4^0C$**	**Specific gravity at$20^0/20^0C$**	**GMS of Sicrose per 100 ml weight in Vacuo**
0.0	0.99823	1.00000	0.0000	4.0	1.01388	1.01567	4.0555
0.1	0.99862	1.00039	0.0999	4.1	1.01428	1.01607	4.1585
0.2	0.99901	1.00078	0.1998	4.2	1.01467	1.01647	4.2616
0.3	0.99940	1.00117	0.2998	4.3	1.01507	1.01687	4.3648
0.4	0.99979	1.00155	0.3999	4.4	1.01547	1.01726	4.4681
0.5	1.00017	1.00194	0.5001	4.5	1.01586	1.01766	4.5714
0.6	1.00056	1.00233	0.6003	4.6	1.01626	1.01806	4.6748
0.7	1.00095	1.00272	0.7007	4.7	1.01666	1.01846	4.7783
0.8	1.00134	1.00311	0.8011	4.8	1.01706	1.01886	4.8819
0.9	1.00173	1.00350	0.9016	4.9	1.01746	1.01926	4.9856
1.0	1.00212	1.00389	1.0021	5.0	1.01785	1.01965	5.0892
1.1	1.00251	1.00428	1.1028	5.1	1.01825	1.02005	5.1931
1.2	1.00290	1.00467	1.2035	5.2	1.01865	1.02045	5.2970
1.3	1.00329	1.00506	1.3043	5.3	1.01905	1.02085	5.4010
1.4	1.00368	1.00545	1.4052	5.4	1.01945	1.02125	5.5050
1.5	1.00406	1.00584	1.5061	5.5	1.01985	1.02165	5.6092
1.6	1.00445	1.00623	1.6071	5.6	1.02025	1.02206	5.7134
1.7	1.00484	1.00662	1.7082	5.7	1.02065	1.02246	5.8177
1.8	1.00523	1.00701	1.8094	5.8	1.02105	1.02286	5.9221
1.9	1.00562	1.00740	1.9107		1.02145	1.02321	6.0266
2.0	1.00602	1.00779	2.0120	6.0	1.02186	1.02366	6.1312
2.1	1.00641	1.00818	2.1135	6.1	1.02226	1.02407	6.2358
2.2	1.00680	1.00858	2.2150	6.2	1.02266	1.02447	6.3405
2.3	1.00719	1.00897	2.3165	6.3	1.02306	1.02487	6.4453
2.4	1.00758	1.00936	2.4182	6.4	1.02346	1.02527	6.5501

2.5	1.00797	1.00976	2.5199	6.5	1.02387	1.02568	6.6552
2.6	1.00836	1.01015	2.6217	6.6	1.02427	1.02608	6.7602
2.7	1.00876	1.01054	2.7237	6.7	1.02467	1.02648	6.8653
2.8	1.00915	1.01093	2.8256	6.8	1.02508	1.02689	6.9705
2.9	1.00954	1.01133	2.9277	6.9	1.02548	1.02729	7.0758
3.0	1.00993	1.01172	3.0298	7.0	1.02588	1.02770	7.1812
3.1	1.01033	1.01211	3.1320	7.1	1.02629	1.02810	7.2867
3.2	1.01072	1.01251	3.2343	7.2	1.02669	1.02851	7.3922
3.3	1.01112	1.01290	3.3367	7.3	1.02710	1.02892	7.4978
3.4	1.01151	1.01330	3.4391	7.4	1.02750	1.02932	7.6035
3.5	1.01190	1.01369	3.5416	7.5	1.02791	1.02973	7.7093
3.6	1.01230	1.01409	3.6443	7.6	1.02832	1.03013	7.8152
3.7	1.01269	1.01448	3.7470	7.7	1.02872	1.03054	7.9211
3.8	1.01309	1.01488	3.8497	7.8	1.02913	1.03095	8.0272
3.9	1.01348	1.01528	3.9526	7.9	1.02954	1.03136	8.1334

Appendix 6: Photo taken during processing of the experiment


0.333
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