ADDIS ABABA UNIVERSITY

INSTITUTE OF TECHNOLOGY

SCHOOL OF CHEMICAL AND BIO ENGINEERING

INVESTIGATIONS OF FILTRATE CLARIFICATION AND OPTIMIZATIONS OF FLOCCULANT DOSAGES: A CASE STUDY AT WONJI SHOA SUGAR FACTORY

A Thesis submitted to the Addis Ababa Institute of Technology, in Partial Fulfillment of the Requirements for the Degree of Master of Science in School of Chemical and Bio Engineering (Process Engineering Stream)

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The undersigned have examined the thesis entitled '**Investigations of filtrate clarification and optimizations of flocculant dosages: A case study at Wonji Shoa Sugar Factory**' presented by Agezegn Gezahegn, a candidate for the degree of **Master of Science in chemical Engineering (process stream)** and hereby certify that it is worthy of acceptance.

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ABSTRACT

This study was found out the installed filtrate clarification plant by flotation system in the Wonji Shoa Sugar Factory was inapplicable and ineffective. The average juices Brix to carry out by floatation system are 65^0Brix and above while the average Brix for the filtrate juice measured were 8.94. The only possible method investigated was by decanting the filtrate juice in a separate stream. This type of method of production system requires a number of processing parameters like flocculant dosages, phosphate and lime additions. The method as well accounts of optimum processing conditions for better juice purification system. Then, the only identified factors in this filtrate clarification system were lime additions and flocculant dosages because of the amounts of phosphate levels already found in the filtrate juice were 350 ppm which were sufficient for the next clarification process to be carried on. So the initial steps were the filtrated juice samples taken from the filtrate juice tank and then measurements of the parameters such as purity, pH, phosphate contents and calcium contents were analyzed before the clarification system carried out. Next flocculants were measured by weighing balance and they were prepared in solutions form at a temperature of 50-60^0C. Then, the juices passed through a heating medium until the temperature reaches 70-75^0C. When the temperatures reached at the required range, the filtered juices treated with limes at different pH levels. Then, the heating processes were also continued until the temperature reaches 95-100^0C. Next the prepared samples were taken and poured into the 1000 ml graduated cylinders, and then different concentrations of flocculants (Separan) were added. Immediately, the parameters such as the settling rate, the height of muds formed in the cylinder, the colors, the turbidity, the percentage purity, the purity rise, calcium and magnesium contents of the clear juice were measured. Finally, the effect of milk of limes starting 6.8-7.4 pH and flocculants 4-8ppm within clear juices were optimized by using Design Expert software version of 6.0.8. By using this software the responses such as pH of clear juices, the turbidities and the colors were the significant while the calcium contents did not show any significant variations at 5% significance level. The optimal solutions obtained for flocculants dosages in the range of 4.05- 4.67 and limed filtrated juice pH at 6.84-7.01 combined respectively.

Key words: **Filtrate, Lime, Flocculant, Phosphate, Color, Turbidity and pH**

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DECLARATION

I declare that this thesis **'Investigations of filtrate clarification and optimizations of flocculant dosages: A case study at Wonji Shoa Sugar Factory'** is my own work and that it has not been submitted in any form for another degree at any university or other institute. Information derived from the work has been duly acknowledged in the text and a list of reference is given.

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Acronym	Nomenclature
A	Flocculant dosage
APF	Anionic Polyacrylamide Flocculant
B	Limed filtrate juice pH
BC	Before Clarification
°Be’	Degree Baume
CCD	Central Composite Design
CJ	Clear Juice
DS	Dry solids
ESCRDC	Ethiopian Sugar Corporation Research and Development Center
EDTA	Ethylene Diamine Tetra Acetic acid
H.V.A	Handlers Vereenining Amsterdam
ICUMSA	International Commission for Uniform Methods of Sugar Analysis
IU	ICUMSA Color Unit
LFJ	Limed Filtrate Juice
MDJ	Muddy Juice
MJ	Mixed Juice
MOL	Milk of Lime
PC	Prepared cane
Pol	Polarization
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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CHAPTER ONE

1. INTRODUCTION

1.1 Background of the study

Sugar cane is a subtropical or tropical grass that originated in Papua, New Guinea and spread throughout South East Asia, India, the Mediterranean, the Caribbean, Hawaii and the south United State because of human migration and the slave trade. The juice from the sugar cane stalk is highly appreciated and is the source of 70 percent of the world's sugar. The cultivation of sugar cane is limited to the tropical areas within 35° North and South of the equator. Sugar cane flourishes in a wide variety of soils ranging from very heavy clays to almost pure peat to sandy soils. It is a crop which requires high temperature for its growth. Though temperatures between 20 and 38°C are favorable and optimum growth takes place at air temperatures between 30 and 33°C. Temperatures below 20°C and above 38°C are not conducive to good growth and plenty of sunlight, with large quantities of water with at least 1500 mm (as rain or irrigation) per year. The ideal climate is one with a long warm summer growing season, fairly dry, sunny and cool but frost free ripening and harvesting season (JR, 1985). This humidity and heat loving plant produces tall, edible canes and enjoys a soil pH of 5.5 to 6.5. Sugar cane also has the highest number of calories per unit area of any plant.

Sugar cane is comprised of stalks, leaves and a root system. The stalk contains the juice used to make sugar and is broken up into segments called joints. Each joint has a node (band) and an Internode (area between nodes). The leaves are attached to the node. The length and diameter of the joints and stalk vary by sugar cane species. The root system at the base of the plant anchors the grass to the soil and takes in water and nutrients from the ground. If the top part of the sugar cane is cut, but the roots remain intact or damaged, the plant will regrow from where it was cut. It can take six to twenty four months for the plant to grow to maturity.

Sugar cane is harvested when it reaches maturity and during the dry season of the particular sugar cane plantation. Harvest time can take anywhere from 2.5 months to eleven months. Harvesters (humans or machinery) chop down the stems and leave the roots to regrow for next season, collect the stems and load them onto a truck for transport to the processing facility.

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 (Mathur, 1981). 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 cleaning, which involves screening, heating, and the addition of milk of lime (calcium hydroxide) to adjust pH and enhance flocculation. The limed juice is then allowed to settle out suspended particles in a clarifier.

Following the juice cleaning process the clear or clarified juice is then evaporated to approximately 65 % dry solids (DS) in a stepwise evaporation process. 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 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 (Rein, 2008) .

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 prior to evaporation are paramount 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 (E.Hugot, 1986).

After clarification, the muds are removed from the clarifier and then send to the filters to recover the sucrose from the mud solids. Rotating vacuum filters are used to recover the trapped sucrose and which generate continuously two products: a filter cake that was being withdrawn from the process and a filtrate juice having 8 up to 9°Brix that was being continuously recirculated and mixed with the mixed juice. Recycling of this filtrate juice into the mixed juice tank is not good.
In the Wonji Shoa Sugar Factory the filtrate clarifier was installed in order to reduce overloading in clarification station. The plant is consisting along with two heaters, the main two clarifiers and having two clarified filtrate tank holders and the aim of these plants is to clarify the filtrate juice.

1.2 Statement of the problem

Suspended solids and some dissolved compounds are separated from mixed juice forming a mud that is de-sweetened in vacuum filters. Filtrate leaving from vacuum filter is very low in purity, highly turbid, rich in gums and non-sugars and a lot of impurities. This filtrate is feeding backward to the mixed juice and it contains about 15 to 20 percent of the total mixed juice before clarification. The return of this process results a lot of problems with the factory operation, including an increase of the mixed juice tank level, sugar destruction because of recirculation, decreases the quality of the filtrate and sugar recovery, formation of maximum mud volume on filtration plant, increase of sucrose inversion, increase of reducing sugars and additional overloads on plant capacity especially in clarification plant.

The another disadvantage of feeding filtrate to mixed juice appears gives that filtrate contains microbiological reduced products of sugar decomposition, and all of these extremely undesirable impurities are regularly recycled to the mixed juice tank, where they are returned through juice liming, juice heating and juice clarification and these results in additional color formation.

The expected plant which was installed to clarify the filtrate juice through the floatation method in the factory was not functional and parts of its accessories were taken for another plant maintenance activity. The plant is consisting along with two heaters, flocculant dosing tanks, reaction tanks, and lime dosing tanks which are waiting without any operation.

During plantation white sugar production by using the filtrate clarifier the consumption of flocculant dosage, addition of milk of lime and phosphoric acid as well as the juice heating temperature are the most common processing parameters. Because of the absence of standard operational parameters and dependency on variation of environmental factor and plant performance, it is desired to study about those processing parameters. The impurity loading of the filtrate has also the greatest impact on clarification performance and optimum processing conditions. In the same way the cost of the flocculant is very expensive. To compensate its cost and removal of turbidity in processing condition finding the optimum dosage is very important issue to reduce loss through mud clarifier and cost of flocculant. So trying the best option for the

process instead of feeding into mixed juice to clear juice tank is the central point of the sugar processing factory and area of study of the research work to become specific.

1.3 Objective of the study

1.3.1 General objective

The main objective of this research was to increase boiling house recovery and clarification house capacity.

1.3.2 Specific objectives

The most specific objectives of this research work were the following below:

Study of the effect of the turbidity, settling rate, mud volume and color levels after the treatment of filtrate juice at different combinations of factors.
Determine the sucrose or pol and the Brix loss or gain after the filtrate juice clarification.
Asses the contents of calcium oxide and magnesium oxide levels of clear juices after the clarification process has carried out at different flocculant dosages and limed filtrate juices.
Find out the lime purity and their corresponding impurities in relation to the recommended allowable impurities.

1.4 Significance of the Study

The main aim of this research was to provide critical information for sugar producing factory especially for WSSF with view of investigations of filtrate clarification and optimizations of process parameters, thereby decreasing the cost of production by using available resources which were already practiced in other countries. Furthermore, after implementing this technology, it can also serve as a reference in relation to transfer this technology to others Ethiopian sugar manufacturing industries. Filtrate clarification is one of the most important unit operations, which can bring to either success or failure of the income of the factory. Better work performance at the filtrate clarification plant provide a considerable benefit in terms of rise in boiling house recovery, rise filtrate juice purity, increase in clarification house capacity, improvement in performance and capacity of vacuum filter, reduction in color, increase in clear juice transmittance and reduction of scale in sulphites juice heaters.

CHAPTER TWO

2. LITERATURE REVIEW

2.1 General overview of Wonji Shoa Sugar Factory

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

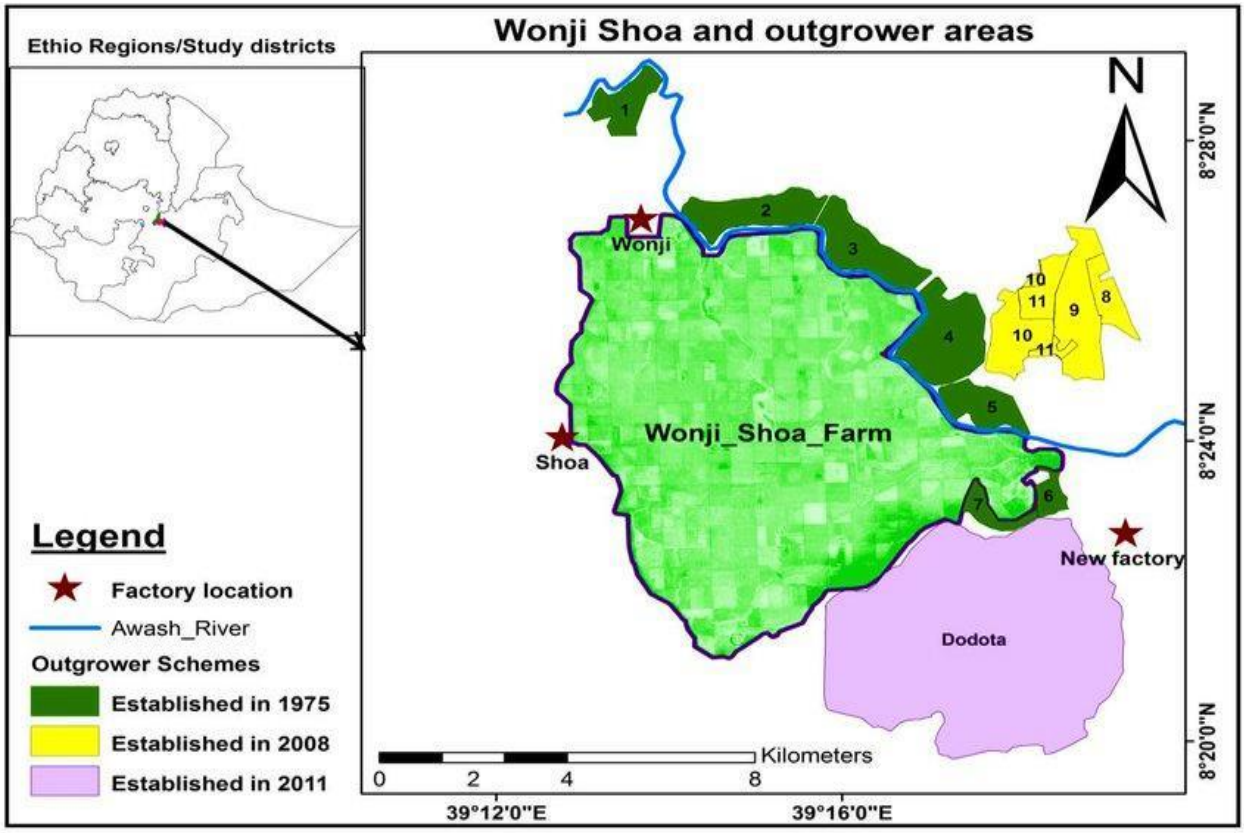

Figure 2. 1: Location of WSSF source (Mengistu, 2009)

In a bid to replace these two oldest factories with a new and modern one, 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 a day and producing over 174,000 tons of sugar per annum which with further expansion will reach up to 12,500 TCD maximizing its production to 220,700 tons of sugar a year. In the same way to rise up its economy from the sugar byproduct the new ethanol plant planned to be built, will have a capacity of producing 12,800 meter cube.

The Factory is currently contributing 20 megawatt electric powers to the national grid in addition to satisfying its own demand which is around 11 megawatts. 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. There are 32 sugar cane growers association; which has in total 9,100 member farmers. The Factory, beyond supplying the farmers with selected seeds, and rendering professional as well as technical support to them, has made irrigable land available to all (Crees, 1974).

2.2 Clarification as a unit operation

Raw cane juice contains beside sucrose more than 50 non sugar impurities, like reducing sugars, organic acids and inorganic acid, amino acids, proteins, starches, waxes, gums, minerals (such as potassium, magnesium, calcium, silica), coloring matters, and other suspended matters that cause dark color and high turbidity to the juice (Gil and Wright, Verma et al., 1994).

The main goals of a white sugar cane factory are to have an efficient, profitable operation with required sugar quality and maximum sugar recovery. One of the most factors that determine the sugar quality and recovery in both cane and sugar manufacture is clarification process. Clarification as used in the sugar industry refers to the precipitation and removal of all possible non-sugars, organic and inorganic, and the preservation of the maximum possible sucrose in clarified juice (Baikow, 1982).

In a well conducted clarification, the pol (sucrose and reducing sugars) expected to be remained intact or not damaged. But the Brix of the juice is expected to be decreased since a portion of the dissolved impurities is removed by settling. Therefore, in an efficient clarification procedure, the purity (the ratio of pol to Brix) of the clear juice will rise by 1 to 2 units to that of the mixed juice, which is known as rise in purity between the mixed juice and the clear juice (Mathur, 1981).

Separation of these impurities from the juice as early as possible should be done by clarification to avoid the problems of increasing color, sucrose inversion, high viscosity, and the generation of excess molasses (Gil and Wright, Verma et al., 1994). The quality of the product mainly depends on the efficiency of clarification (Xian et., 1998). However, troubles related to clarification, the hope to upgrade the quality of sugar, and production of new products like organic sugar have always promoted the sugar technologists to search the new separation process, which were commercially tested in other industries.

In conventional clarification process in cane mills, the mud result from the clarifier is sent to rotary vacuum filters for sugar recovery. Filtrates from the filtration station are very dirty with an apparent purity one up to five percent lowers than clarified juice and under some circumstances; this purity drop can increase too much higher value so that it is difficult to send these filtrates straight to the evaporator station (Trott, 1988). Therefore, filtrates are returned to the mixed juice tank and recirculate through the mainstream clarification system.

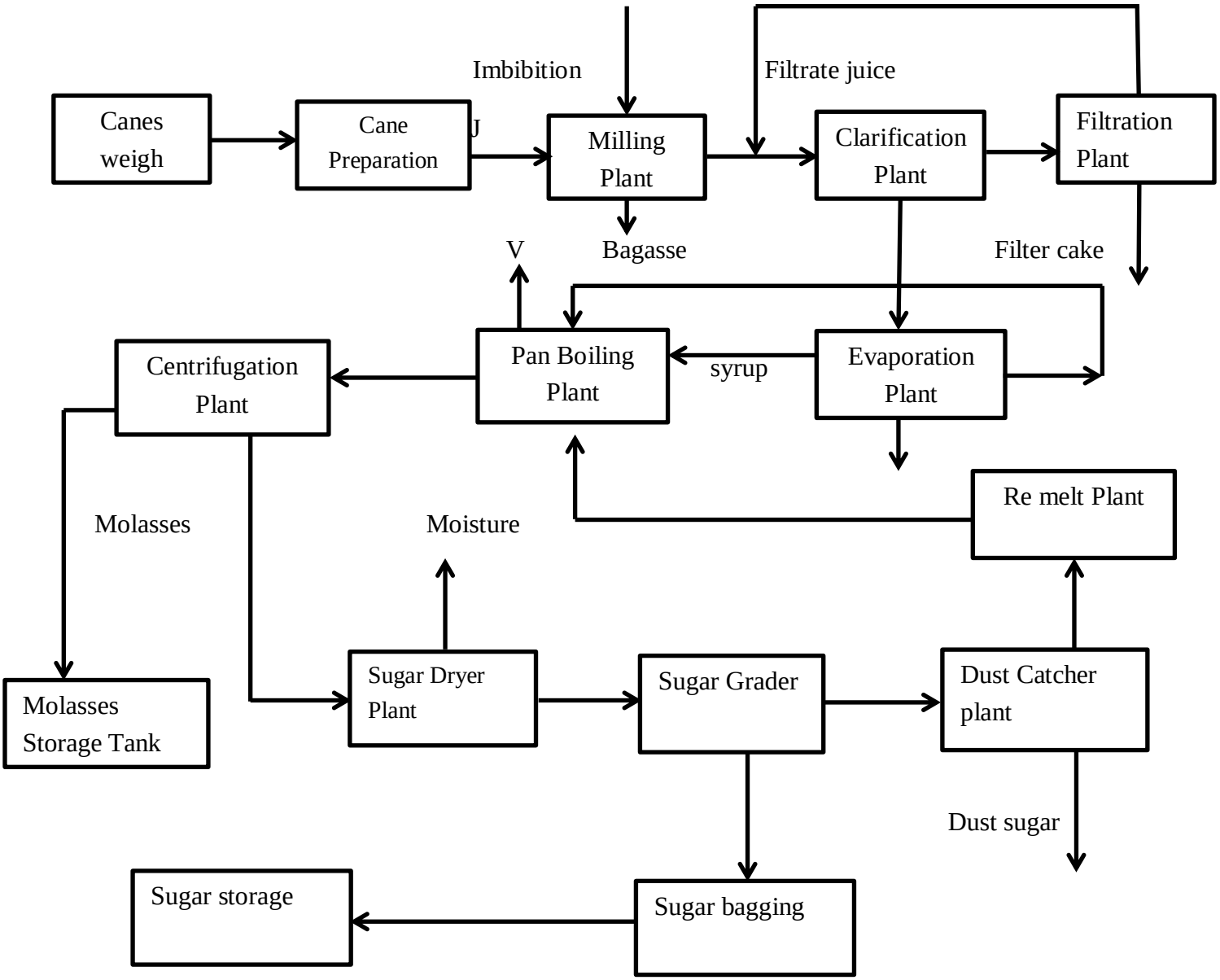

Figure 2. 2: overview of the process flow diagram of WSSF

2.3 Recycling of filtrate juice into mixed juice

Recycling the filtrates from rotary vacuum filters has many disadvantages. It makes the operation of the filters directly affects the performance of the clarifier i.e. The mud solids output of the filters must match the mud solids rate entering the factory in mixed juice. If this basic requirement is not satisfied, then the mud will accumulate in the clarifier.

During periods of high mud loadings, the buffer capacity available in the clarifier for variations in mud volume is usually very limited. The other problem of filter operations that affects significantly on the clarifier is the retention of mud solids in the filter cake and the amount of mud being recycled with the filtrate. The returned mud will exceed the mud loading problems at the clarifier, particularly during periods of high incoming mud solids loadings (Steindl, 1998).

Another disadvantage of recycling filtrates appears from the fact that these filtrates contain the microbiological degradation products of sugar decomposition, and all of these extremely harmful impurities are normally returned to the mixed juice tank, where they are recycled through juice liming, juice heating, and juice clarification and this results in the additional color generation and sucrose inversion (Bennett, 1957). To overcome these problems, and for production of white sugar a separate filtrate clarification system is used (Chen, 1993).

Transfer of mud solids into the clarified juice is likely to reduce sugar filterability and other quality parameters, as well as the increasing scale formation in the evaporators and pans. The recycle of filtrate can represent an extra addition of 10% up to 15% mud solids loading on the clarifier, thereby reducing its effective capacity. The impurities recycled with filtrate (i.e. Proteins, organic acids and mineral ions) can also impact on downstream processing operations.

Large amounts of organic acids in the primary juice will not only result in the use of increased lime usage, but will hold some of the calcium in a combined complex state. The calcium, organic acid complex will slowly break down during subsequent processing resulting in the formation of scale and other impurities. The complex that is not broken will mostly end up in molasses and may impact on exhaustion.

2.4 The benefits of the filtrated juice clarification

Eliminating the recycling of impurities through juice clarification station has a dramatic effect on clarifying juice and hence thick clear juice quality. Besides this highly beneficial effect on purity, the process would save energy and chemicals at the clarification station as well as increases the capacity of the juice clarifier station by the proportion of Rotary Vacuum Filtrate (RVF) filtrate.

2.5 The juice clarification

The purpose of clarification process is to remove maximum possible impurities from the juice as early as possible in the process. The mixed juice from the extraction plant reaches the clarification station as a complex mixture of the integral components of cane plant. The juice contains considerable colloidal matter, fine suspended matter and other soluble constituents. Thus the mixed juice is turbid and viscous in nature and is not fit to be worked up without suitable chemical treatment.

2.5.1 Objectives of the juice clarification

The primary object of juice clarification is the removed of the maximum quantity of non-sugars that have harmful effects on recovery of commercial sugar at the earliest possible stage. The main objects of cane juice clarification are as follows:

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

2.5.2 The importance of effective clarification

- Sucrose recovery is raised as the loss through final molasses is reduced.
- Fouling effect of heat exchangers is minimized as the hardness of the clarified juice is minimized.
- The quality of the final product (sugar) is improved.

- Operational consistency in the boiling house will be favorable with higher capacity.

To obtain high quality clarified juices or syrups, 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. The colloids are known to be responsible for higher or lower turbidity of sugar products and to have an adverse effect on the entire process (Rodriguez et al., 1977).

Cane juice is a poly dispersed irreversible colloidal suspension in which the suspended particles carry a large negative charge. According to established theory of colloid stability, small particles have attracted one another by Van der Wall's forces and therefore always tend to aggregate together, but they are kept apart by electrostatic forces arising from their electrical charges. The measure of these repellent forces 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 μm. 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 (Rein, 2008).

2.5.3 Reaction of clarification

The chemical reactions that occur during cane juice or filtrated juice clarification are not fully understood. It is, however, fundamental that those reactions to lower free energy content would take place preferentially, with reactions requiring lower energy completing first. Defecation, as an example 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^- (Saska, 2010).

2.5.4 Lime and its behavior in clarification

Lime slurry is a solution consists of mainly a coarse suspension and colloidal solution of Ca $(OH)_2$. The solubility of Ca $(OH)_2$ in water is very low (0.2% at 25 ^{0}C). The solubility of lime is greatly increased in a sucrose solution. So that 10% of sucrose solution dissolves 1.5% CaO. In the same way, the solubility of lime is also increased by some non-sugar substances present in the juice. The solubility of Ca $(OH)_2$ decreases with the increase in temperature. Thus, a solution saturated at room temperature with precipitate out on heating. Ca $(OH)_2$ is a relatively strong base. Ca $(OH)_2$ is a hydroxide of divalent metal; ionization takes place in two steps.

1^{st} step $Ca(OH)_2 \rightleftharpoons Ca^{+}OH + OH^{-}$

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

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 (Ca^{+2}) is low.

By the combination of Ca ions with anions of inorganic and organic acids slightly soluble calcium salts are produced such as:

$$Ca^{++} + PO_4^{-3} \rightarrow Ca_3(PO_4)_2$$

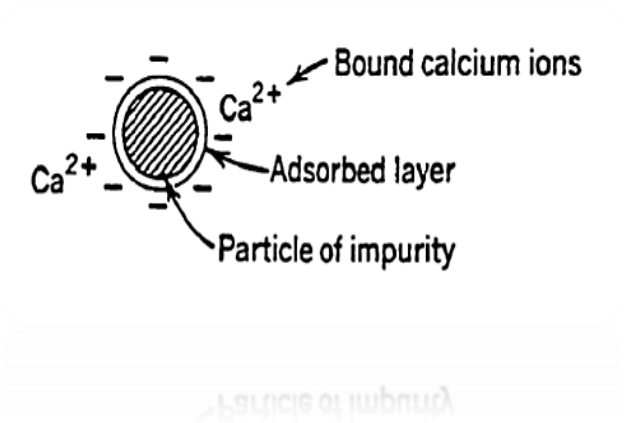

Figure 2. 3: The juice particle (Abayneh, 2011)

By the combination of OH^- ions with cations, slightly soluble hydroxides are formed

$$Fe^{+3} + OH^- \rightarrow Fe(OH)_3$$

$$Al^{+3} + OH^- \rightarrow Al(OH)_3$$

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 reactions also. Because the secondary ionization of calcium hydroxide is small, the concentration of calcium (Ca^+) is low. The secondary ionization is low, with less than 10 % of calcium as Ca^{++}. The reaction of lime and phosphate in juice or filtrate 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 in sucrose solution containing 0.3-0.5 % calcium oxide $Ca_3(PO_4)_2$, 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 the juice is heated (Honig, 1963).

2.5.6 Role of phosphate in juice clarification

The phosphate content of the juice is the most important factor in efficient clarification. In sugar cane, the phosphates are found as inorganic as well as organic forms. The inorganic phosphate exists as free phosphate ions, whereas the organics exist in the form of phospholipids, phosphoproteins, hexophosphates, etc. It is obvious that only the free phosphate ions (inorganic form) take part in juice clarification. Therefore, juices with adequate quantity of inorganic phosphate are most desirable. Phosphatation removes the colloidal and other impurities, absorbs much of the coloring matter and diminishes the calcium content of the clear juice (Mathur, 1981).

During the cane growing, if fertilizers are not properly applied, there may be more organic phosphate in the juice than inorganic phosphate. Then the juice may not respond well to clarification. It is demonstrated that if the inorganic phosphate level in raw juice is less than 300 ppm (W/V), the juice cannot be properly clarified and addition of phosphate is required. Phosphate reacts with the calcium ions present in the juice or syrup to form a primary

flocculation of insoluble calcium phosphate. The precipitate formed entrains suspended impurities and also adsorbs high molecular weight dissolved impurities from the juice and finally separates to give a clear juice. It is generally added at a level corresponding to 200 to 400 ppm by weight of P_2O_5 on the basis of the sugar in juice or syrup (Pottage, 1975).

Sugar cane juice naturally contains phosphorus. In primary juice clarification techniques, inorganic phosphate ions have played a substantial part in reactions with calcium (Baton, 2000). 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 (E.Hugot, 1986).
(Edey, 1999) Quote a rule that satisfactory clarification can be achieved when the cane juice contains 300 mg/kg phosphate (as P_2O_5). (Julienne, 1996) Quote 200 mg P_2O_5 /kg juice as the minimum level of phosphate.

In the phosphate deficient cane juice, the addition of soluble phosphate to mixed juice to bring percentage to the minimum (0.03%) triple superphosphate (48%) P_2O_5 is generally used. Di calcium phosphate, ammonium phosphate or syrupy phosphoric acid may be used for this purpose. A controlling factor is the relative cost per unit of P_2O_5.

2. 5. 6. 1The advantages and disadvantages of phosphate addition

The advantage of phosphate addition in juice improves: Greater colloid elimination, fewer lime salts in clarifying juice, more rapid settling, faster mud filtration and better working conditions of low grade massecuite while the disadvantages are of the precipitate tri calcium phosphate is difficult to filter, increased mud volume and higher lime consumption.

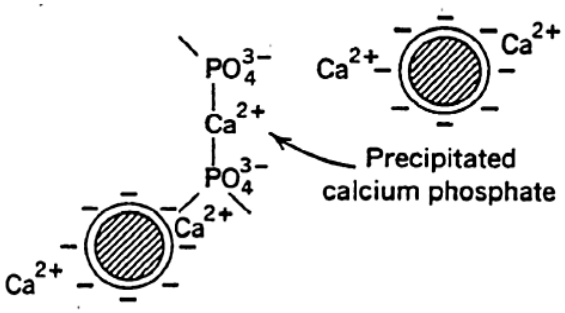

Figure 2. 4: Calcium phosphate bridging in clarification (Abayneh, 2011)

2.5.7 Summary of lime action on juice clarification

The following are the activities of lime on clarification system

1. Lime 1st neutralizes the free organic acids present in the cane juice forming organic salts of calcium.
2. On continuous addition, the lime reacts with phosphoric acid present in the juice and forms calcium phosphate.

$$3Ca(OH)_2 + P_2O_5 \longrightarrow Ca_3(P_2O_5)_2 + 3H_2O$$

3. Lime combines with nitrogen impurities like albuminoids and gummy substances, which are partly precipitated.
4. Lime combines with pectin to form soluble and insoluble compounds.
5. Lime precipitates coloring matters like chlorophyll and anthocyanin.
6. Lime acts on sucrose to form saccharates and acts on reducing sugars to form glucosates. The action of lime on reducing sugars is undesired which increases the undesirable soluble calcium salts.

2.6 Milk of lime preparation

Milk of lime was prepared at a concentration between 6 to 10^0B'e. The factory was using both cold and hot water simultaneously. The lime slaking process is also an exothermic and around 15.2 Kcal of energy as a form of heat evolved.

Figure 2. 5: The WSSF milk of lime preparation plant

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

The solubility calcium oxide in water is very small and 1 part of calcium oxide in 790 parts of water. In order to increase the suspension of calcium oxide with water by the lime slaking process 3 parts of hot water are used to one part of lime. Thus a finely divided milk of lime as a particle form is produced. More than 95% of these particles vary in size from one to five microns for soft burnt lime and 3 to 25 microns for the lime of lower reactivity with the sub microns particles present.

The milk of lime is prepared in a revolving drum with interior baffle plates, serves to mix quicklime and water. The drum is revolved by means of an encircling sprocket and chain. Fresh lime is charged in the rotary slacker through the inlet and sufficient quantity of water is added. The milk of lime is discharged at the other end. The unslaked pieces of limestone or grits are lifted by mechanical and discharged outside. The milk of lime emerging from the slacker contains fine impurities. It is strained through a screen of 8 meshes. This lime still contains fine grits, which is separated through a hydro cyclone (Dorr clone) mechanism. The milk of lime is collected in two tanks. The tanks have stirrers running at 8 to 10 rpm. The outlet of the tank is supplied with a sieve of 1.3 mm opening to separate foreign matter. The milk of lime is a suspension not a solution and because of that, it has to be kept in a constant motion to prevent deposition of lime. Finally, milk of lime is pumped to juice and syrup clarification station points with the excess recycling back to the tank.

The velocity of milk of lime in the piping must be greater than 1 m/sec. Centrifugal pumps are used, for milk of lime. Copper or brass parts are not resistant to alkalinity and they should be avoided from the lime station (Abayneh, 2011).

Density of lime

The relation between degrees Baume and lime content of milk of lime is given by (E.Hugot, 1986). The concentration of milk of lime is measured in degree Baume.

Table 2. 1: The relation between degree Baume and density

Degree Baume	Density g/cm^3	CaO content g/cm^3
5	1.037	46
6	1.045	56
8	1.060	75
10	1.075	94
15	1.116	148
20	1.162	206
22	1.180	229

Lime and its quality

Whatever process of juice clarification is followed, lime is the main clarification agent. Lime is transported in trucks to the sugar factories from lime kiln. Lime can be also purchased in paper bags. The amount of lime received by the sugar factory should be kept low and fresh enough since lime attracts moisture and carbon dioxide from the atmosphere. Compact stacking is essential in order to reduce absorption of moisture and carbon dioxide. The quality of lime is important, as impurities will pass into the juice, which brings negative effect on clarification and subsequent processes.

Lime is received from lime kiln where limestone (calcium carbonate) burns at 1200 to 1300 ^{0}C and dissociated into quicklime and carbon dioxide. Now a day the WSSF is using from Zeway lime Factory. A good quality of lime should contain above 95% $CaCO_3$ and less than 2% insoluble matter. The maximum impurities allowable are listed in the table 2.2 recommended by (E.Hugot, 1986).

Table 2. 2: The lime allowable impurities

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

The effect of high impurities in lime

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

a) 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.
b) High percentage of magnesia retards filtration and rate of settling.
c) High percentage of iron affects the color of the juice. Both iron and aluminum salts cause scaling on tubes.

2.7 The pH control in the sugar industry

The sugar industries, before the introduction of the pH method for measuring the reactions of sugar juices and intermediate liquors, the only means available were test paper or titration with standard acid or alkali to an end point depending on some indicator. These methods measured only the quantity of acidity or alkalinity, whereas the hydrogen ion concentration or pH is the measure of the intensity of the acidity or alkalinity. Because the sucrose inverting power of an acid is a direct function of dissociation (i.e. of the amount of hydrogen ion concentration in the solution). Thus, the importance of pH control in sugar industries is highly emphasized. It should be remembered that pH scale is a logarithmic scale representing the power of 10.

2.7.1 Conditions for efficient pH control

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

2.8 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. 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. 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.

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 flocculant is essential.

Thus, the introduction of synthetic polyacrylamide as settling aids has been a significant advance in the process of clarification.

- 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.

Turbidity due to un-scavenged colloids and other particles is highly undesirable, as it ultimately causes low filterability in sugars. Poor flocculant addition, bad design, and bad liming control can contribute to high turbidity (Peter, 2007).

The recommended dosage of flocculant added to a clarifier is 2 to 4 ppm. Previous studies (Crees, 1974) have shown that even when as low as 2 ppm of flocculant is used, carryover of flocculant still occurs. Clarifying the filtrate may reduce the amount of flocculant reintroduced back to mixed juice. As flocculants are of very high molecular weight, their presence even in minute quantities can promote the precipitation of scale forming compounds.

Rapid flocculation and sedimentation of suspended particles in primary cane sugar juice is achieved using a high molecular weight anionic polymer flocculant. Flocculation removes insoluble suspended particles and colloidal impurities by aggregating them into flocs. The performance of flocculants to enhance the flocculation and sedimentation of cane sugar juice particles is evaluated by turbidity and settling rate measurements (Edey, 1999).

The addition of one up to three ppm of anionic polyacrylamide flocculant (APF) into sulfited juice improves the clarity of the juice and mud separation (E.Hugot, 1986).
To improve decolorization of the juice, phosphatation can be supported by a flocculating agent (Cartier, 1997).
When flocculants (anionic) are applied in mud filtration the following advantages are achieved:

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

2.8.1 The action of flocculants in juice clarification

The activities of flocculant in juices clarification were producing secondary flocculation increases the settling rate, reduce mud volume, decreases pol in cake and increases the clarity if

clarified juice. There is no universal polymer that will suit all diversified conditions, but for a particular factory, there may be one polymer that will give consistent performance.

2.8.2 Separan AP 30

This is a coagulant added to the juice before clarifying or to the mud before filtration. It is expensive, but appears to improve the clarity of the juice and the subside process. It forms an integral part of the Rapi-Flock process.

The variability in the composition of the juice as a result of differing geographical location, and seasonal influence as well as the conditions to which the cane is subjected during harvesting and transportation makes the nature of the research to be area specific and hence there is no universal dose of phosphate and flocculant so as to produce the best possible quality clear juice. Furthermore, the chemical reactions that occur during the clarification process have been made more complex by the use of a variety of clarification aids. Each factory is therefore essentially unique and adopts a system of clarification based on the simple defecation process with modifications usually determined by a combination of research and experience (Simpson, 1996). Report laboratory work to quantify the effects of techniques on clarifying juice quality and floe settling behavior (Edey, 1999).A dose rate of flocculant (saparan A 30) at 5mg/kg juice was applied before settling.

2.8.3 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. The suspension is then added sparingly to warm water (50 to 60^0C) 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 flocculant 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 1^{st} point that flocs once formed are not broken. This is expected to be at the inlet of the clarifier (E.Hugot, 1986).

2.9 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. 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. 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.

2.9.1 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. 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.

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 75^0C, 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.

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.

Settling (sedimentation) rate is the height of the settled particles per unit time. A low settling rate 2 cm/min in the clarifiers is generally an indication of an improper purification process.

The best indicators of proper clarification are the following:

- Reasonable settling rate (4 to 6 cm/min)
- Reasonable concentration of the mud (about 20^0 Baume)
- Reasonable thickening ratio (0.2 to 0.4)
- Reasonable clarity in juice overflow

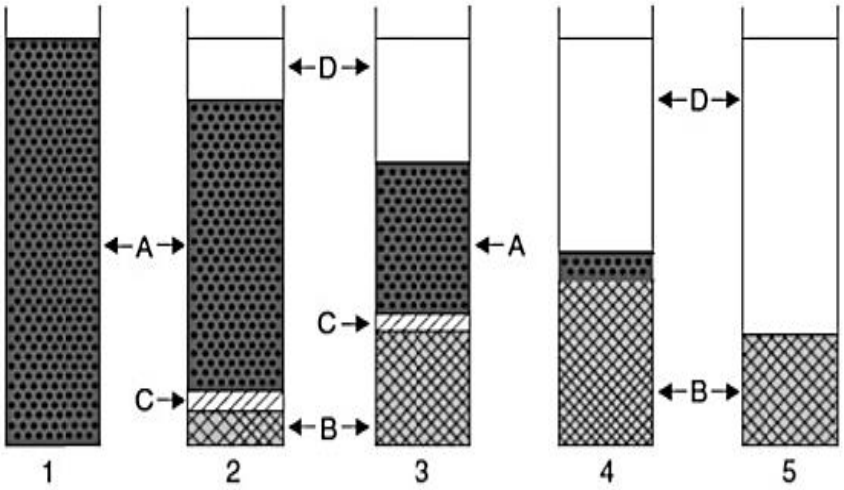

Figure 2. 2: Settling zones in the clarifier

2.9.1.1 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 103^0C). 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.

2.9.1.2 The Dorr clarifier

The flashed juice is directly fed into the Rapidorr 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. 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.

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.

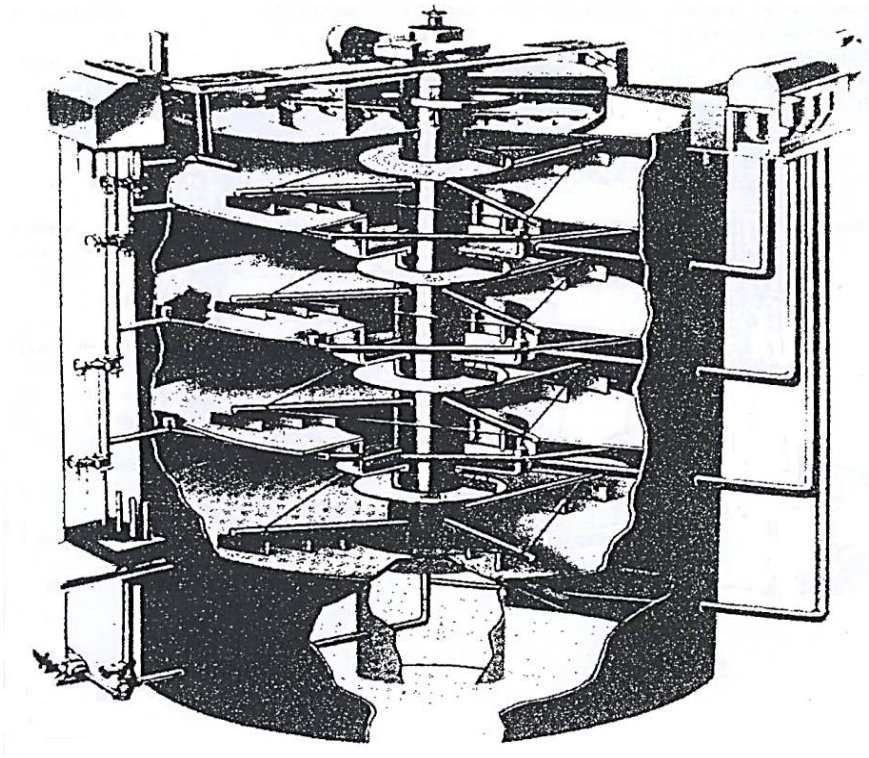

Figure 2. 3: Rapidorr 444 clarifier

2.10 Filtration

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

2.10.1 Types of filters

After the clarification process performed the muddy juice, which is not sent into the evaporation plant has to be filtered because of the presence of sucrose in it. Therefore the sugar factory has been using the following types of filters: Filter presses, mechanical or leaf filters and continuous rotary vacuum filters.

The first two types have disappeared from the defecation and sulphitation factories, to their benefit. However, filter presses still remain the standard system for carbonizations factories, for the filtration of juices after first and second carbonations.

2.12.1.1 Continuous rotary vacuum filters

The filter is composed of a hollow drum rotating about a horizontal axis and partly submerged in the liquid to be filtered. The periphery of the drum serves as the filtering surface. It is divided into 24 independent sections connected individually to a vacuum system by a small pipe terminating in a distributing value situated at one end of the drum and carrying three different sectors as follows:

- One without connecting to the vacuum, but connected to the atmosphere.
- The second connects with a low vacuum chamber, of the order of 15 to 30 cm of mercury.
- The third connects with a high vacuum chamber, of the order of 40 to 50 cm of mercury.

The exterior surface of the drum consists of a sheet of copper or stainless steel with very fine perforation, covering the 24 sections.

The perforated screen of the rotary

The exterior surface of the drum consists of a sheet of copper, or brass or stainless steel perforated with 625 holes of 0.5 mm in diameter. Screen of copper are preferred to those in stainless steel. They are more flexible and less fragile.

The agitator of the mud mixer

In order to avoid allowing the mud in the filter tank to settle out, they are kept in movement by an agitator oscillating to and from, pivoted on the axis of the filter, and driven by a small separate motor. To avoid rupture of the flocculent mud particles, the bagacillo mixture should be fed by gravity.

Figure 2. 4: WSSF Rotary Vacuum Filtration Plant

Speed of the drum

The speed of the drive motor is constant generally 1450 r.p.m. It drives the filter drum by means of a belt and double worm reduction gear. A speed regulation device controlled by a hand wheel permits the speed reduction ratio of the v belt to be varied. The maximum rotation of the drum may be regulated to one rotation in three minutes i.e. twenty rotations per hour. The minimum rotation may be regulated to one rotation in ten minutes. i.e. six rotations per hour.

On average, a speed of one revolution per five minutes, i.e. twelve revolutions per hour is used. It is estimated that by doubling the speed of the drum, the filtration rate is increased only by a factory of 1.4.

Filter cake

The filter cake appears dry and porous. But, nevertheless, contains about 70% of water. Bagacillo is added to the mud to facilitate filtration. It is necessary to reckon on about 3 to 4 kg of filter cake per 100 kg of cane having a specific gravity of 1.3 to 1.4. The thickness of the cake is of the order of 7 to 13 mm. The filter turns about 60 to 70 kg of filter cake per m^2 per hour.

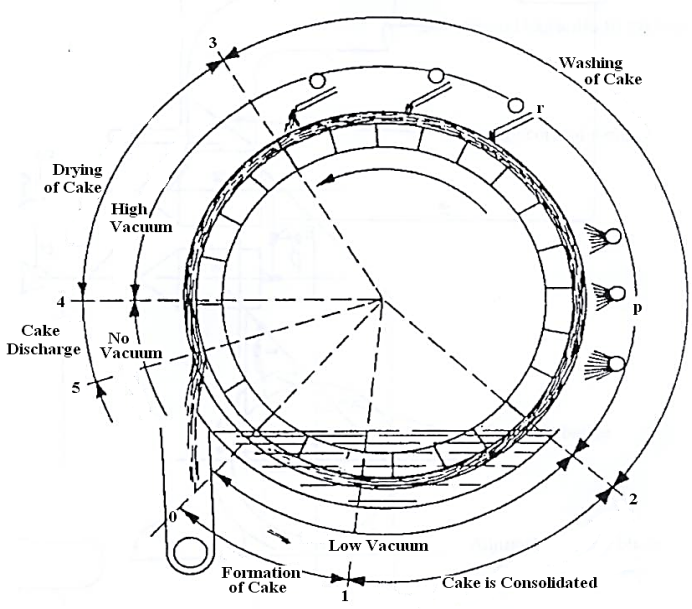

Figure 2. 5: Diagrammatic operation of a continuous rotary vacuum filter

Dry substance in mud: - The proportion of solids in the mud going to the filtration should be not less than 4.5%.

Washing (sweetening of water):- The quantity of wash water, which passes into the clear filtrate, represents only a small fraction of the water used in the filter (20 to 25%) so the greater part remains in the filter cake. Therefore, the quantity of water to be used for washing should be according to the dilution of the filtrate and not according to the quantity of water sent to the filter. Therefore, the washing efficiency is more important than the quantity of water.

Generally, wash water amounting to 100 to 150% on cake is used. Hot condensate water is recommended to minimize bacterial action. The pressure of spray water should be 3 to 4 kg/cm^2.

Sugar loss in cake: - The filter cake contains 0.5 to 3% of sugar, averaging 1 to 2%. This corresponds to a pol loss of about 0.2 to 0.8%, average 0.5 % (E.Hugot, 1986).

Filtration rate: - A filtration rate of 250 liter per square meter of filtering surface per hour may be expected. However, the capacity of filtrate pumps is reckoned as 480 to 500 liter per square meter per hour (E.Hugot, 1986).

Proportion of cloudy filtrate: - For every 100 parts of filtrate supplied by the filter, it may be estimated that there will be 30 to 60% of cloudy filtrate and 70% of clear filtrate.
These two filtrates together may contain between 2 and 12% of suspended matter expressed as dry material.

Disposal of filtrates: - Disposal of filtrates is often difficult to decide depending on what is the best way to deal with these two filtrates.
For the cloudy filtrate, the first idea which comes to mind is to send it to the filter tank or to the mud tank preceding it. However, this dilutes the filter feed, and is not to be recommended. It is necessary to return the cloudy juice ahead of the clarifier. In compound clarification, there is a choice between returning it before the primary clarifier or before the secondary. The former solution is preferable (E.Hugot, 1986).

As regards the clear filtrate, unfortunately it has neither the brilliance nor the clarity of the clarified juice. It is grayish and still slightly cloudy. It would not be desirable to send it to manufacture with the primary clear juice. Accordingly, it is generally returned to process ahead of the clarifier.

In simple clarification and often in compound clarification, it is therefore common practice to mix the two filtrates and return the mixed filtrate to process ahead of the clarifier. This is the disadvantage of this type of filter: it overloads the clarification station. When the latter is working near the limit of its capacity, it is undesirable to increase the volume of the juices, and consequently their speed of circulation in the clarifier, by a fraction which represents approximately (E.Hugot, 1986).

Simple clarification system volume of juice increased in the clarifier is 12-20%. But for compound clarifier, which having primary clarifier and secondary clarifier increased volume in the clarifier is 10-20% and 10-15% respectively.

One solution consists of re-clarifying the two filtrates in a special small clarifier, after reheating and re-liming. A very clear juice is thus obtained, together with thick mud, which is returned to the rotary filter. The filtrates may also be treated in a centrifugal separator of the Westfalia type.

Importance of mud filtration

Mud from the clarifier may appear thick. However, it contains less than 10% mud solidified on dry basis. Over 90% of mud is clear juice that has to be recovered. The purpose of filter station is to separate as efficiently as possible, the mud solids from the clarifier mud. The clear juice fraction is recycled to weigh mixed juice tank. The mud slide together with any added filter aids is called filter cake and discharged as by-product. The filter cake is composed of about: 70% moisture, 29% solids and 1% Brix (E.Hugot, 1986).

2.10.2 Condition for good filtration

Filtration is sometimes a tricky operation, and difficult to control. To combine the best chances of carrying it out satisfactorily, it is necessary to observe several points:

Temperature: - The viscosity of the juices and especially that of the gums and waxes, which have to pass through the filtering surface, decrease as the temperature increases. It is therefore of advantage to filter the juice at a high temperature. Preferably, temperatures between 80 and 85^0c will be used.

Reaction: - Alkaline juices, filter better than acid or neutral juices; hence it was the general custom to add lime to muds before sending them in the filtration process, raising the pH to 8-8.5. This was necessary with filter presses, but is not done for rotary vacuum filters.

The bagacillo: - The bagacillo is added and mixed with mud. The added bagacillo mops up the stickiness of mud, give it body and forms a fibrous mat through which the filtrate will be drawn. The quantity of bagacillo used is equal to the quantity of dry mud solids present (E.Hugot, 1986).

CHAPTER THREE

3. MATERIALS AND METHODS

3.1 Site of the experiment

The samples were taken from WSSF to Research and Development main center. The experiments were applied at the Wonji Sugar Research and development laboratory. The laboratory is found in the Oromiya region, state near to Nazareth city at 110 kilometers from Addis Ababa and 25 kilometers from the factory. Any research associated with the sugar cane production and sugar processing technologies is held in this main laboratory. The experiment was conducted based on filtrate produced from the rotary vacuum and taken from the factory as a sample. The filtrate clarification experimental work and the determination of the response variables were done in the research laboratory service of the station.

Figure 3. 1: Wonji Shoa Sugar Factory

3.2 Materials

Sample for the experiment were filtrate juice from rotary vacuum filters, the prepared milk of lime from the WSSF lime preparation plant, condensate water from the flocculant preparation plant of the condensate water inlet valve, refractometer, saccharometer and spectrophotometer.

3.3 Sample preparation

Filtrate juice of six liters, a liter of condensate water and a half liter of milk of lime from the filtrate juice tank, condensate feeding valve into the tank of flocculant preparation and milk of the lime tank were collected respectively for one run to Sugar Research and Development center laboratory station. Immediately the samples get into the laboratory station parameters undertaken from the filtrate juice like pH, Brix, pol and inorganic phosphate content and flocculant preparation were carried out.

Flocculant is materials of high molecular weight purchased in powder form and it acts as a bridge between two or more particles present in the juice to unite the solid particles into a loose porous state.

During flocculant preparation the first step was sampling the condensate water from the tank of flocculant preparation by opening the condensate feeding pipeline valve and then immediately measuring the temperature of the condensate by digital thermometer. When the measured temperature of the condensate water was between 50 and 60^0c, after that the preparation was taken place. If the condensate water temperature was not between the range of the standard conditions, and then it was prepared to become the optimum soluble conditions by boiling it. The reason for controlling the working condition was due to the nature of the polymer. When the temperature of the condensate water was below the standard condition the solubility of the flocculant (Separan) was very slow and if it was above the standards the polymer large, long chain was being broken down and the result after treating the juice with the help flocculant was not well during the experiment. So maintained the optimum temperature of the condensate water temperature was very crucial in this experimental work. Up next a one liter measuring cylinder was filled by the condensate water having temperature of 50-60^0C and then weighed of different grams of 0.25, 0.5, and 0.75 flocculant powder (Separan) were prepared and added into the

1000ml cylinders at different days and then the solutions were stirring the contents by slowly and they were waiting for two hours.

The filtrate juice, which was obtained from the factory transferred into the stainless steel kettle type and then heated on the stove to 70-75^{0}C and then the heated filtrate juices were treated with the milk of lime at different pH until the required pH was obtained. Next the heating continued until the temperature to become 95^{0}C. Then the heated filtrate juice was poured into the one liter measuring cylinder and they were treated by pipetting different concentration of saparan A-30 solution (3.17-8.83ppm). Finally, then the clarified juice samples were examined for the determination of physicochemical properties and for comparison between the standard operation of clear juice when they were not recycled with the filtrate juices. During the clarification process parameters which were determined like pol%, Brix%, purity%, pH, turbidity, color, mud volume, settling rate, calcium and magnesium contents.

3.4 Analytical methods

The majority of the analytical results were found by the method of Handbook of Laboratory Methods and Chemical Control for Ethiopian Sugar Factories (H, 2009) while the remains methods were adequately referred.

3.4.1 Brix measurement of the filtrate juice and the clear juice

The brix of the filtrate juice and clear juices were determined according to the method 6.1.8 (SASTA, 2005) using refractometer method. The refractometer had circulatory water bath to maintain the juice temperature at 20^{0}C and zeroing was taking place. A few drops of the sample were placed to the refractometer prism. Then, the measured Brix value was directly taken from the refractometer reading.

3.4.2 The pH measurement of filtrate juice and clear juice

The pH meter was standardized with buffer solutions of pH 4, 7 & 9.2. Prior measurement when the sample was hot passes through refrigerator until the sample temperature equal to the room temperature. 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.

3.4.3 Determination of phosphate in the filtrate juice

The soluble phosphate of the filtrate juice was determined according to the method 6.3.8 of (SASTA, 2005). The filtrate juice was filtered through a fluted filter paper in a funnel. The first ten ml of the filtrate was rejected as first runner and about 20 ml of clear filtrate was collected. The Brix of the sample was measured in the refractometer. Filtrate (10 ml) was pipetted into a 200 ml volumetric flask and made to the mark with distilled water and then mixed thoroughly. Twenty ml of this solution was pipetted into two 100 ml volumetric flask, one of which was a blank. To the first flask, 50 ml of water, 10 ml of ammonium molybdate solution and 10 ml of reducing solution (ascorbic acid) was added followed by mixing by swirling and diluted to volume. The absorbance of the solution was measured in a spectrophotometer (Model CECIL 2021) exactly ten minutes after the addition of the ascorbic acid at 700 nm in a 10 mm cell using distilled water as the reference. The second flask (blank) was made to volume with distilled water. The absorbance of the blank was determined at 700 mm in a 10 mm cell using water as a reference.

$$P_2O_5 \frac{mg}{L} in\ the\ sample\ juice = \frac{\frac{Mgp_{2O_5} \times 2 \times 1000}{50ml\ test\ solution}}{Aliquot\ of\ diluted\ juice\ used} \quad (3.1)$$

3.4.4 Determination of pol in factory products

The polarization of the mixed and clear juices was determined according to the method 6.1.7 of (SASTA, 2005). 200ml of the juice sample was poured into the beaker provided with a stopper. Two grams basic lead acetate was added and shaken vigorously, 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 25ml of filtrate were discarded and the saccharometer tube rinsed twice with a portion of the filtrate. The tube was filled with the remaining filtrate and then the saccharometer reading was taken. Finally, the pol% of the juices

samples were calculated based on the refractometer Brix reading of the juice sample and saccharometer reading from the spectrometer and pol factor from appendices H, table 1.

3.4.5 Determination of the juice purity

The juice purity was determined when the Brix% and the pol% of the juice were initially measured and then it was calculated by using the following formula:

$$Purity = \frac{\%\ Pol \times 100}{\%\ Brix} \quad (3.3)$$

3.4.6 Determination of the mud volume in clarification system

The volume of the mud was calculated when the height of mud formed in the one liter measuring cylinder was known before and in the same way the internal diameter of the cylinder used was measured. Finally the volume of the mud formed in the cylinder was calculated by the following formula:

$$V = \pi \times R^2 \times H \quad (3.4)$$

Where R is the radius of the internal cylinder

H is the mud height formed in the cylinder

3.4.7 Determination of calcium oxide in clarifying juice

3.4.7.1 Complex metric method (ICUMSA GS7-19 1994, EDTA titration)

A complex is a molecule or ion formed from the reaction of two or more individually stable ions or molecules, but the reaction is driven by the electron deficiency of metal ions. The most important complication reactions from an analytical point of view are those between a metal ion in solution and a complexing agent known as a ligand or chelate. The metal ions are Lewis acids and readily share electron pairs with the electron donating ligands (Lewis bases). As an example, cyanide (CN^-) binds to a metal ion through a lone pair on one atom and is thus a monodentate (one tooth) ligand. Many transition metal ions will share up to six electron pairs with ligands. A ligand that bonds to a metal ion using more than one ligand atom is called a multidentate (many toothed) ligand or a chelate (or sometimes a chelating agent).

In a complex metric titration, a solution containing the free metal ion of interest was titrated with a solution of a chelating agent until all of the metal ions were bound. The endpoint was usually signaled by an indicator ligand that forms a colored complex with the free metal ion.

The concentration of calcium and magnesium in clear juice was determined approximately by complex metric titration against EDTA solution, just like hardness in boiler water. For more exact determination, precipitation with oxalate and titration with potassium permanganate was recommended. However, the titration with EDTA was given accurate enough results for routine factory control (ICUMSA GS7-19, 1994).

Pipettes 50ml of the filtered juice into 100ml volumetric flask and made up to mark with distilled water (50% solution v/v) .Then pipettes 10ml the diluted solution into a 250ml Erlenmeyer flask, added about 50ml of distilled water, added 2ml ammonium buffer solution and about 0.1gram of indicator Erichrome black T. Stirred the solution with a magnetic stirrer and titrated against standard EDTA solution from the burette until pink color of the solution was first changed to light green.

Expression of the result was as follows:

$CaO + MgO\ expressed\ as\ CaCO_3$

$$= \frac{0.574 \times a \times 1000 \times dilution\ factor(2)}{c} \qquad (3.5)$$

Where: a. EDTA solution found by titration

c. Diluted solution for titration

3.4.8 Determination of magnesium oxide in clear juice

The 5% ammonium oxalate solution was made by weighing 50 grams of ammonium oxalate in solid form and transferred to a 1000 ml reagent bottle. Then 900ml of distill water was added and mixed well in it. After the solution was prepared a 100ml of the filtered juice was pipetted into 250ml of volumetric flask and then a 25ml prepared ammonium oxalate solution was added and filled up to mark by distill water. Mixed thoroughly and allowed the solution to stand for one hour to ensure complete precipitation of the calcium oxalate. Then it was filtered through Whatman No 40 filter paper. Pipette 10ml of this solution into 250ml Erlenmeyer flask and diluted to about 50ml with distilled water. Next added 2ml of ammonium buffer solution and

0.1gram of Erichrome black T indicator in solid form. Stirred the solution with magnetic stirrer and titrated against standard EDTA solution until the pink color of solution first change to light green.

Expression of the result was given below:

$$CaO\ in\ 1\ liter\ juice = \frac{0.574(a-b)\times 1000\times dilution\times dilution\ factor(2)}{C} \quad (3.6)$$

$$MgO\ in\ 1\ liter\ juice = \frac{0.413\times b\times 1000\times dilution\ factor\ (2)}{C} \quad (3.7)$$

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

b. EDTA is taken for titration of magnesium oxide

c. Diluted juice for titration

3.4.9 Determination of turbidity in clarifying juice

The turbidity of clarified juice is a measure of the effectiveness of the clarification process. The turbidity of the clear juice was determined according to the method 3.5 of (SASTA, 2005).

The ICUMSA colors of unfiltered and filtered samples are measured at 420nm and the difference between the readings is the turbidity of unfiltered juice (MSIRI, 1991).

The Brix of clarified juice was initially measured and then the conversion of the original Brix into a 2^0Brix was carried out. After conversion the required gram of clear juice was weighed and filled with distill water until the weight became hundred grams. This sample divided into two equal portions and the first portion was subjected to filtering through a Buchner funnel with Whatman No 5 filter paper using kieselguhr filter aid. Next the two sample portions were adjusted by using a 1M NaOH and 1M HCl until the pH of the clear juice became 7±0.2. Then the absorbance of filter (A_1) and unfiltered (A_2) samples was measured in a 1cm cell using spectrophotometer at 420nm light wave. Finally the results were expressed in the following formula.

$$ICUMSA_{420}\ Colr\ C_1 = \frac{A_1\times 1000}{1cm\times concentration\ of\ total\ solids(\frac{g}{ml})} \quad (3.8)$$

$$= \frac{A_1\times 1000}{1\times 2\times 1.00602/100} = \frac{A_1\times 1000}{0.0201}$$

$$ICUMSA_{420}\ Color\ C_2 = \frac{A_2 \times 1000}{1cm \times total\ solids\ concentration(\frac{g}{ml})} \quad (3.9)$$

$$= \frac{A_2 \times 1000}{0.0201}$$

$$Turbidity\ of\ clarified\ juice\ C_2 - C_1 = \frac{(A_2 - A_1)}{0.0201} \times 1000 \quad (3.10)$$

3.4.10 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 effectiveness of clarification. The color index of the clear juice was determined according to the method 3.4 of South African Sugar Technologists' Association (SASTA, 2005).

Initially the brix of the clarified juice was measured and then the conversion of the original brix into a 5^0brix was made and then the desired amount was known. After conversion the required gram of clear juice was weighed and filled with distill water until the weight became hundred grams. This solution was adjusted by using 0.05MHCl and 0.05MNaOH until the pH became 7.0 $\pm$ 0.2. Then the solution was filtered through a membrane had a pore size of 0.45 μm. The solution was placed in an optical cell and measured absorbance, at 420nm in a spectrophotometer using distilled water as reference standard of zero color. The cell length was chosen so that the instrument reading was taken between 20 to 80% transmittance and 0.1 - 0.7 absorbance.
Expression of the result was as shown below

$$ICUMSA_{420}\ color\ unit = \frac{Absorbance \times 1000}{b \times c} \quad (3.11)$$

Where: b - cell length (cm)

c - Concentration of total solids (g/ml)

3.4.11 Determination of the settling rate

The 1000ml graduated cylinder marked in ml increments was filled with the treated juice. Then, timing the settling rate was started as soon as the sample showed the first sign of separation (a very thin layer of clear juice above the solids starts to occur). After exactly five seconds and the

level of the settled mud (the boundary between clear juice and solids) was measured. The settling rate is the rate at which the height of the settled mud formed per unit time.

3.4.12 Diameter of the cylinder

The experiment was carried out at different cylinders having an internal radius of 3, 3.2 and 3.4 cm and the measurement was taken simply by using ruler.

3.5 Analysis of Lime

The complete analysis of limestone comprises the determination of moisture, organic matter, and solids insoluble in HCl, silica, iron and aluminum oxide, magnesium oxide, sulphates, calcium oxide and carbon dioxide.

3.5.1 Solid insoluble in HCl (in lime)

Ten grams of limestone was weighed and transferred into a 300ml Erlenmeyer flask with 50ml of water and added 25ml of concentrated HCl with the stem of stem less funnel. As soon as the effervescence was stopped, the limestone was totally dissolved; added 0.5 to 1ml concentrated HNO_3 and boiled for five minutes. Filtered through an ash free filter paper and collected the filtrate quantitatively in an Erlenmeyer flask. The precipitate was washed with hot distilled water until no chloride reaction was observed with silver nitrate. The filtrate was kept for determination of silica and ignited the filter with the precipitate in a tarred porcelain crucible to a constant weight.

Expression of the result

$$\%\, Insoluble\ in\ HCl = \frac{Weight\ of\ the\ precipitate \times 100}{Weight\ of\ sample} \qquad (3.12)$$

3.5.2 Silicic acid (SiO_2) in lime

The filtrate obtained for the determination of HCl from above was took and heat until the boiling temperature becomes and at the same time sufficient amount of 10% ammonia was added up to the color litmus paper turned to blue. The solution was boiled the for some minutes more and it settled by placing the flask in leaning position and then filtered the clear liquid before dumping

the precipitate. The precipitate washed quickly and completely until the filtrate shown no reaction with silver nitrate. The filtrate was collected and washings in a one liter volumetric flask (Filtrate A) for the determination of Ca, Mg and sulphates. The precipitate contains silica, iron and aluminum hydroxide. Placed the funnel with filter and precipitate above a porcelain evaporating dish and washed out with small quantities of warm HCl (1:1) through the filter and repeated this operation. The precipitate was dissolved quantitatively. The filter was washed the filter with hot water until chlorides could no longer be detected in the washings. The HCl solution of silica, iron, and alumina on hot water bath was evaporated to dryness. The solutions again moisten the residue with concentrated HCl and evaporated to dryness. The evaporating dish was dried with content in an oven at 1200 for two hours. Cooled and moisten with concentrated HCl and let the acid work for ten minutes. Added 50 ml hot distilled water and while shaking, brought to boiling. The precipitate was filtered through ash free filter paper. The filter was washed the filter until the washing contains no more chloride. The washing was collected in an Erlenmeyer flask. (Filtrate B) contains iron and aluminum. The filter with precipitate ignited in previously tarred porcelain and crucible to a constant weight.

Expression of the result

$$\%\, SiO_2\ in\ limestone = \frac{Weight\ of\ SiO_2 \times 100}{Weight\ of sample} \qquad (3.13)$$

3.5.3 Fe_2O_3 and Al_2O_3 in lime

Took filtrate "B" from silica determination and added hot water to a volume of 200ml in a beaker. Heated to boiling and added so much ammonia solution (10%) until the litmus paper was colored slightly blue. Continued boiling for five minutes and let the precipitate settled out in an inclined position of the flask. The precipitate was filtered through an ash free filter paper. It was washed out with hot 2% ammonium nitrate solution until the washings were free chlorides. The filtrate was added and washings back to the one liter volumetric flask (filtrate “A"). The filter and precipitate were ignited in porcelain crucible, previously tarred until constant weight.

Expression of the result

$$\%\, Fe_2O_3 + \%\, Al_2O_3\ in\ limestone = \frac{Weight\ of\ (Fe_2O_3 + Al_2\ O_3) \times 100}{Weight\ of\ limestone} \qquad (3.14)$$

3.5.4 Magnesium oxide in lime

For the contents of silica determination took one liter graduated flask (filtrate A). It was cool, filled to the mark and mixed the content become well. Took 400 ml of this solution in a 700 ml conical flask; added excess of 12% ammonium oxalate solution and heated, to the boiling point. The calcium present was removed. Allowed to boil for a few minutes and allow standing in a hot place for two hours. The content was cool and rinsed into 500ml graduated flask. Made to mark, mixed and filtered discarding the first 10ml. Collected 250 ml of the filtrate and evaporated in a porcelain dish till the volume was 50ml. Rinsed the concentrate quantitatively in to a 250ml beaker and diluted with water approximately 100 ml. The liquid was heated to boiling point and added 10ml of a 10% ammonium phosphate solution and three drops of phenolphthalein. While stirring, added drop wise (a long glass rod along the wall of the beaker) so much concentrated ammonia to color the liquid vividly red. Finally 25 ml of 10% ammonia was added. The liquid allowed to stand for twelve hours and after that, filter off the magnesium ammonium phosphate precipitated. It washed with 2.5% ammonia until the wash shows no chloride. Dried the filter paper with the precipitate; carefully transferred the precipitate from the filter paper to previously tarred porcelain crucible. Incinerated the filter paper on a platinum wire over the crucible to add the ash to the precipitated. The crucible at the start slowly glow and strongly later, so that the precipitate was completely changed to magnesium pyrophosphate ($Mg_2P_2O_7$). The precipitate was weighed as $Mg_2P_2O_7$ and multiplied by 0.3624. The magnesium obtained for the analysis of one fifth of the filtrate of one liter was used. Therefore, the obtained magnesium oxide came also from the one fifth of this weighed of limestone.

Expression of the result

$$\%\, MgO = \frac{Mg_2P_2 \times 0.3624 \times 5 \times 100}{Weight\ of\ limestone} \qquad (3.16)$$

3.5.5 Sulphate (as SO_3) in limestone

Took 200ml of the contents of the one liter graduated flask (Filtrate A) and concentrated in an evaporating dish to approximately 50ml and quantitatively rinsed into 250ml beaker.

Added HCl (1:1) till litmus paper was colored slightly pink. Added one ml more HCl and heated to boiling, the sulphates were precipitated at boiling temperature with approximately 5ml of a

10% barium chloride solution. After a few minutes of boiling, allowed the solution to stand for four hours in a hot place. The $BaSO_4$ was filtered through ash free filter paper washed completely with cold water until the wash reacted negative for chloride. Then dried the filter and precipitated the precipitate was carefully transferred into previously tarred crucible. The filter paper was burned over the crucible using platinum wire. The ignited precipitate was moist with a few drops of 50% H_2SO_4 (1:1) to convert a small amount of barium sulphide into barium sulphate. The crucible hated till white fumes, no longer escaped and then ignited till constant weight.

Expression of the result

$$Sulphate\ as\ SO_3 = \frac{Weight\ of\ BaSO_4 \times 0.3429 \times 5 \times 100}{Weight\ of\ limestone} \quad (3.17)$$

3.5.6 Calcium carbonate in lime (purity of lime)

Determination of calcium carbonate content in limestone in the absence of a substantial quantity of magnesium carbonate gives sufficient information about the quality of limestone. The tube was filled on the right with 1:1 HCl acid and that on the left with concentrated H_2SO_4. Transferred 0.2 gram of sample to the bottle and weighed the apparatus. Allowed the HCl acid in the tube on the right side to enter the bottle drop by drop until the solution was complete. The CO_2 escaped through the tube containing H_2SO_4 which would absorb the water vapor, which may be carried away by the CO_2 gas. The apparatus on the sand bath heated until all CO_2 is expelled. The apparatus was cool and weighed record the difference as the weight of CO_2.

Expression of the result

$$\%\ CaCO_3 = \frac{100}{44} \times \frac{Weight\ CO_2 \times 100}{0.2} \quad (3.18)$$

3.5.7 Calcium oxide in lime

Weighed 0.5 gram of limestone and transferred quantitatively into 300ml conical flask with 100ml of water. The flask was covered with small funnel and added 3ml concentrated HCl acid. On completion of the reaction, added a few drops of nitric acid. Then boiled the mixture for a few minutes and added 10% ammonia solution till litmus paper was colored slightly blue (Impurities were precipitated). The content was filtered and washed the precipitate with a hot 2%

ammonium nitrate solution. Collected the filtrate and washings in a 400ml beaker diluted the content to 200ml and added 20ml of hot ammonium oxalate solution (4%) at the boiling point. In this case calcium was precipitated. Boiled the liquid for a few minutes and allowed to stand for four hours in a hot place. Decanted the supernatant liquid through a filter and washed the precipitate repeatedly by adding hot water, till the washing no longer contains chlorides. Placed the filter above the beaker and perforate with platinum wire. Calcium oxalate, which was present, then rinsed into the beaker. Rinsed the filter with 10ml of hot diluted (1:5) sulphuric acid twice and next with hot water until the total volume was approximately 250ml. Added 20ml sulphuric acid (1:1) and titrated the oxalic acid with potassium permanganate.

Expression of the result

$$\%\, CaO = \frac{Burette\ reading\ (ml) \times titre\ of\ permanganate\ \times 0.028 \times 100}{Weight\ of\ limestone\ sample} \quad (3.19)$$

$$\%\, CaCO_3 = \frac{\%\, CaO \times molecular\ weight\ of\ CaCO_3}{Molecular\ weight\ of\ CaO} \quad (3.20)$$

Carbon dioxide

The percentage of CO_2 is usually determined by the following formula.

$$\%\, CO_2 = (\%\, CaO \times 0.7851) + (\%\, magnesium\ oxide \times 1.091) \quad (3.21)$$

3.5.8 Determination of available CaO in lime

In advanced sugar factories the introduction of powdered hydrated lime $Ca(OH)_2$ and pulverized quicklime of extreme fineness has made the use of rock lime or lump lime obsolete in cane sugar factories developed nations. In Ethiopian sugar factories, lime is delivered in large lumps of quicklime (CaO). The lump lime slacked in a special rotary lime slacker to separate dead burned particles, unburned limestone, and other foreign materials. The quality of quick lime should be analyzed periodically. The sample is dissolved in appropriate solvent prior to analysis. According to Douwes-Dekker, 5% phenol solution is recommended instead of sucrose solution to save time. The phenol lime solution requires only an hour with shaking, whereas two to three hours of frequent shaking was required with sucrose solution. In both cases, the available calcium oxide should combine completely with the phenol or sucrose solution (Honig, 1963).

3.5.8.1 Sucrose method

Weighed out ten grams of finely ground lime sample into a beaker and added about 30ml distilled water to form thick milk. Transferred quantitatively with about 200ml of 40 Brix sucrose solutions into a 250ml flask. Then stirred for three hours using magnetic stirrer, removed the stirring bar by means of a retriever and made up to mark with the 40 Brix sucrose solution and filtered through fluted Whatman No. 1 filter paper and discarded the first 25ml filtrate. Finally pipettes 100ml of the filtrate and titrated with 1N HCl using phenolphthalein as indicator until there was the first change from pink to colorless.

Expression of the result

$$\%\, Available\ CaO\ in\ lime = \frac{Titre\ HCl \times burette\ reading \times 250 \times 0.02804}{Weight\ of\ sample \times 100} \quad (3.22)$$

$$= Burette\ reading\ \times 0.250\ \times 0.02804\ \times 100 \quad (3.23)$$

3.6 Experimental design

All data were analyzed by Design Expert software 6.8 to determine the significance among treatments of different response variables at the 5% level of significance. Furthermore, optimization of the factors and response variables were also carried out using the same software. The consequence of two factors (flocculation and liming of filtrate) which were independent on the optimization of filtrate clarification was measured and Central Composite Design (CCD) for two factors was used to conduct the experiment. The reason why the researcher chosen was the CCD response surface reduces the number of replications. So it gives the center point of treatment for carrying out of experiments.

The main variables studied here are: Turbidity, color, purity rise, pH, mud volume, calcium oxide and magnesium content. Then the optimum pH level combinations of the factors were found out. The experiments were conducted using a lower and upper real value of 4ppm and 8ppm for Separan flocculent respectively. In addition to the lower and the upper real values of lime filtrate juice were 6.8 pH and 7.4 pH respectively.

The lower and upper real values were coded as -1 and +1 respectively, and that of the center points was coded as 0. The lower and upper axial points were coded by –α and +α respectively as shown in Table below.

Table 3. 1: Coded the actual value of the factors used in the central composite design

Notation	Factor	Fixed temperature	Coded levels				
			-α	-1	0	+1	+α
A	Flocculant	50 - 60^0c	≤ 4	4	6	8	8 ≥
B	Limed filtrate juice	70 - 75^0c	≤ 6.8	6.8	7.10	7.4	7.4 ≥

Flocculant prepared temperature, filtrated juice before liming and gas heating after liming parameters required no further optimization and remained constant at a temperature of 50 to 60^0c , 70 to 75^0c) and 95°C respectively.

3.6.1 Statistical analysis

The data were analyzed and modeled using the Design Expert software. Second degree polynomial models were generated. The significant terms in the models were identified by analysis of variance (ANOVA) for each response. Significance was judged if the probability level of the F-statistic calculated from the data was less than 0.05. The model adequacy was checked by R^2, R^2adj and, Lack of Fit test. The Tables of summary of fit, ANOVA, lack of fit and parameter estimates which were generated by the software were presented in the Appendix A, B, C and D part. Contour plots were developed to search the optimum conditions for the three main responses namely, the clear juice pH, the turbidities and the colors.

CHAPTER FOUR

4. RESULTS AND DISCUSSIONS

4.1 Results of the filtrate juice analysis before clarification

Samples of sugar cane filtrate juice were tested about one every week along the course of crushing season (February to May), that was, sampling from eight days.

The filtrate juice and milk of lime taken from the factory were analyzed without any treatments have the following values for Brix, pol, purity, pH, calcium and phosphate contents.

Table 4. 1: Analytical results of filtrate juice before clarification

Trial no.	Brix of MOL	Brix of FJ %	Pol %	Purity %	CaO ppm	P_2O_5 ppm	pH
1	6.80	8.77	5.37	61.23	378	360	5.25
2	6.70	8.25	5.21	63.13	356	340	4.47
3	7.00	6.39	3.12	48.87	395	310	4.46
4	7.40	8.50	6.36	74.86	348	353	5.03
5	6.50	8.70	5.19	59.70	323	357	4.97
6	6.90	8.50	6.36	74.86	376	353	5.03
7	6.90	8.50	6.36	74.86	376	353	5.03
8	6.90	8.50	6.36	74.86	376	353	5.03
9	6.90	8.50	6.36	74.86	376	353	5.03
10	6.72	8.70	5.19	59.70	385	357	4.97
11	6.72	8.70	5.19	59.70	385	357	4.97
12	6.72	8.70	5.19	59.70	385	357	4.97
13	6.72	8.70	5.19	59.70	385	357	4.97
14	6.60	15.70	10.12	64.47	381	349	5.46
Average	7.10	8.94	5.83	65.04	373	350	4.97

4.1.1 Brix of milk of lime

The standard operation of milk of lime in WSSF case was 8^o to 10^oBaume. But the existing operation in average is 7.10^0Baume. This is not good for clarification, due to an increase of mixed juice volume by extra water imbibition.

4.1.2 Brix of filtrate juice

The majority of filtrate juice samples Brix which were taken from the factory where between the standard operation, except two samples which having 6.39 below and 15.7 $Brix^0$ above. But its average fit the standards of filtrate juice, which are of 8.94.

4.1.3 Purity of filtrate juice

Purity of filtrate juice in average was 65.04. But there was also a minimum purity of filtrate juice having 48.87% was registered during the experimental work. This was due to the process flow fluctuations and improper operational controls of the filtration plant. Recycling low figure purity juice in the mixed juice tank disturbs the fresh juice at all.

4.1.4 The pH of filtrate juice

In sugar processing, it is essential to keep the pH in the range of acid base neutrality for the production to be a yield of maximum. Lower values of pH less than seven are avoided because of sucrose inversion and higher values are undesirable, since chemical reactions leading to alkaline degradation at the processing temperature.

Juice keeping a long time within the tank provides the formation of monosaccharaides. The minimum and maximum pH of filtrate juice was 4.46 and 5.46 respectively. The average pH of filtrate juice as it was shown from the table 4.1 is below the mixed juice pH. So recycling this into mixed juice is affecting the mixed juice pH and it contributed to rise up the rate of formation of non-reducing sugars become high and reduces juice quality.

4.1.5 Calcium oxide content of filtrate juice

Checking the existence of lime in filtrate juice is an essential issue to safeguard the forward machinery life, especially the heaters and the evaporators. So the analyzed calcium contents in the form of calcium oxides are very low when they are compared with clear juices. Therefore, clarifying the filtrate juice by adding milk of lime is possible without any fairly large effect on heat exchangers. Finally, it played a role as a basic processing parameter during the lab scale test.

4.1.6 Phosphate content of filtrate juice

The amounts of phosphate contents shown in filtrate juice were 350 ppm and above. They fit the required standard operation so the addition of phosphates during filtrate juice separately are not advisable because of the formation of precipitates of Tri calcium phosphates which are difficult to filter, increased mud volumes and higher limes consumption.

4.2 Mixed juice

The mixed juice analytical parameters obtained during the crushing season of the research working time were such as pH, purity and sucrose were good and data analyzed were in the range of standard operations presented in table 4.2. But the mixed juices Brix are over the range of standard operations. These may bring a negative consequence of the difficult to control in the evaporation plant during the boiling house processing of syrup because of the fast formation of crystals. When crystals are formed in the evaporation plant there are losses through vapor lines. Reducing sugars are also not in the range of standard operations. These show there are inversions during juice extractions system.

Table 4. 2: Analytical results of mixed juice

Trial no.	Brix %	Pol %	Purity	Glucose	Sucrose	pH
1	17.58	15.28	86.9	0.54	12.83	5.5
2	16.32	14.11	86.5	0.57	13.35	5.3
3	15.49	13.31	85.9	0.60	12.65	5.4
4	17.61	15.47	87.8	0.52	13.08	5.3
5	18.01	16.01	88.2	0.56	13.51	5.4
6	17.43	15.47	89.1	0.55	13.76	5.4
7	18.20	16.43	90.3	0.54	14.15	5.3
8	16.79	15.05	89.6	0.57	14.18	5.3
9	17.21	15.19	88.3	0.56	13.51	5.3
10	14.65	14.71	89.1	0.55	13.78	5.3
11	21.55	19.33	89.7	0.53	13.65	5.4
12	22.54	20.00	88.7	0.54	13.28	5.3
13	20.10	17.65	87.8	0.59	13.59	5.4
14	20.63	18.41	89.2	0.58	13.88	5.4
average	18.15	14.92	88.36	0.56	13.51	5.4

4.3 Clarified juice with recycling of the filtrate into the mixed juice

4.3.1 Brix of the clarified juice in the factory

Determination of brix in factory is one of the quality parameters in operation. So analyzing it in WSSF often carries out within two hour intervals and shown in table 4.3. Hence the obtained Brix were not in range of the standard operations. They were almost equal to the output of the first effect of the evaporator Brix. If the evaporation plants, feeding continued by this clear juice Brix, then they will come to face higher fouling rate and maintenance cost become high.

Table 4. 3: Analytical results of clear with recycled filtrate juice

Trial no.	Brix %	Pol %	Purity	pH	Lime content ppm
1	15.31	13.23	86.4	6.3	620
2	17.22	14.96	86.9	6.8	430
3	17.43	15.26	87.6	6.9	460
4	16.36	14.09	86.1	7.9	565
5	17.43	15.17	87.0	7.1	551
6	19.51	17.17	88.4	7.2	649
7	18.96	16.91	89.2	7.2	631
8	18.85	16.68	88.5	7.5	635
9	20.39	18.00	88.1	7.4	1012
10	18.36	15.99	87.2	7.0	567
11	20.15	17.96	88.8	7.2	609
12	19.21	16.85	87.7	7.4	689
13	17.09	15.14	88.6	7.3	654
14	18.14	15.87	87.5	7.1	610
average	18.17	15.95	87.7	7.2	620

4.4 Lime quality

The lime quality is one of the performance indicators for white sugar production process. A good quality of lime should contain over 95% $CaCO_3$ and less than 2% insoluble matter (E.Hugot, 1986). But the factory has been still using about 81.32% of available lime which is not recommended for clarification. The allowable impurity of SiO_2, sulfate as SO_3, iron oxide, and aluminum oxide in this lime is not significant because its composition is below the standards limit. But magnesium oxide has a great significance effect and containing 12.89%. For this reason a high percentage of magnesia retards filtrations and reduce the rate of settling in clarification.

Table 4. 4: Analytical results of lime purity

Impurities	Percentage (%)
SiO_2	0.69
$Fe_2O_3 + Al_2O_3$	0.13
MgO	12.89
Sulfate as SO_3	0.23
Available CaO	81.32

4.5 Results of the clear juice after the clarification process

The clear juice results found during the experimental work at different concentrations of flocculants and limed filtrate juices in the laboratory and analytical response variables are shown in the table 4.5 below. Therefore, the effects of the response variables like pH, calcium oxide contents, turbidities and colors of the clarified juice were put into as a sequence of CCD order and analyzed.

Table 4. 5: Analytical average results obtained after clarification

Separan A30 ppm	pH of LFJ	Pol %	Brix %	pH	Purity %	Purity rise	CaO ppm	MgO ppm	Turbidity ICUMSA	Color ICUMSA	Mud height in cm	Cylinder radius in cm	Initial settling rate cm/min	Final settling rate cm/min	Mud volume in cm^3
4.00	6.80	11.59	14.75	6.77	78.58	14.11	348.23	39.92	995.03	1075.43	20.00	3.25	0.17	0.25	663.33
8.00	6.80	5.11	6.34	6.83	80.59	17.46	324.31	30.97	7960.00	5482.46	10.50	3.00	72.00	2.10	296.73
4.00	7.40	5.01	6.59	6.96	76.02	12.89	358.80	37.17	10199.00	3204.13	13.20	3.40	5.20	0.36	479.14
8.00	7.40	6.35	8.05	7.52	78.89	4.03	338.66	45.43	16998.12	1330.54	8.70	3.45	5.01	0.32	325.15
3.17	7.10	11.96	15.29	6.48	78.22	13.75	357.79	52.31	1492.53	1020.50	21.20	3.25	0.15	0.24	703.12
8.83	7.10	6.69	8.51	7.09	78.61	3.75	424.76	45.43	6517.41	1646.28	8.30	3.25	5.40	0.42	275.28
6.00	6.68	6.73	8.41	6.83	80.02	5.16	401.80	44.05	3582.09	1974.28	8.50	3.00	5.11	0.41	240.21
6.00	7.52	6.82	8.45	7.80	80.71	5.85	406.58	34.48	34129.35	3439.21	8.60	3.20	21.60	0.34	276.52
6.00	7.10	6.58	8.25	6.84	79.95	5.09	429.93	48.18	10497.51	1932.48	11.70	3.00	2.62	0.39	330.64
6.00	7.10	5.39	8.72	7.17	61.81	2.11	298.48	35.79	6815.92	2107.24	15.60	3.25	21.00	0.78	517.39
6.00	7.10	5.35	8.90	7.18	60.11	0.41	332.92	33.04	4676.62	2618.58	19.30	3.00	39.00	0.96	545.42
6.00	7.10	5.33	8.81	7.09	60.50	0.80	323.35	33.04	4179.10	2038.49	11.00	3.00	126.00	2.20	310.86
6.00	7.10	5.42	8.88	7.12	61.04	1.34	267.87	37.17	4029.85	2129.83	11.50	3.20	128.00	2.30	369.77

4.6 The purity rise in the clarifying juice

Purity rise is one of the most important indicators of performance evaluation in the clarification process to identify whether the operational system is good or not. It is the difference between after and before the clarification process. For a factory of processing about 230 tones cane per hour one unit of purity difference improvement can mean a gain in revenue of approximately R1 million (Rajoon, 2006). In case of WSSF the factory is processing about 272.9 canes per hour, can mean a gain in revenue of 1.19 R1.The purity rise obtained by the minimum of 0.41 can mean a gain in revenue of 476000 R1 while the purity rise for the maximum of 17.46 can mean a gain of 20.78R1 million. The maximum and minimum purity rise obtained when the 8.00 ppm of flocculant treated with 6.80 pH of filtrate juice and 6.00ppm treated with 7.10 pH of filtrate juice were 17.46 and 0.41 respectively. For the highest level of treatment of flocculant especially 8.83ppm, the purity rise was not sufficient as compared with majority of treatments, because it may make the sucrose to settle together with the mud.

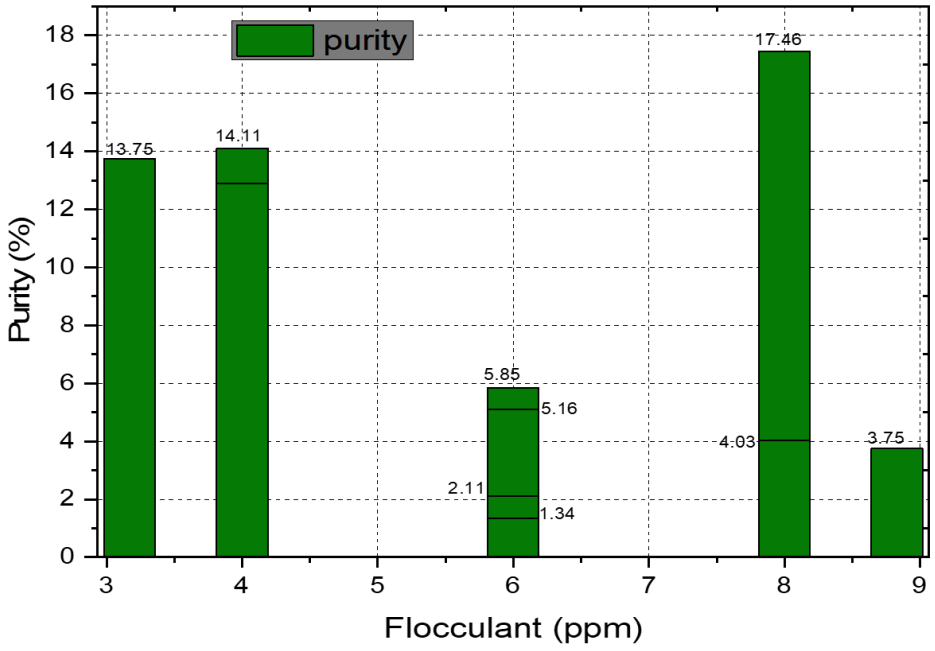

Figure 4. 1: Relation between purity rise and flocculant dosage

4.7 Mud volume

Mud volume is obtained when the mud is settled in the cylinder by measuring the height of formed mud and measuring the internal diameter of the cylinder. Finally the mud volumes were calculated by using formula of volume of cylinder. The maximum mud volume created when it was subjected with 3.17 ppm of flocculant and pH of 7.10 of limed filtrate juice treated together. This maximum mud volume formed has a slow initial settling rate of 0.15cm/min. High mud volume is not needed as it occupies the space of the clear juice, creates load on mud filters and increases the amount of sugar loss in filter cake. On the other hand, highly compacted mud (too low volume) that is caused by over dosing of APF is not needed as it suppresses the flow of mud to vacuum filters. Therefore, it is advisable to set the levels of phosphate and APF in such a way that it gives reasonable mud volume studied by (Hussein, 2013).

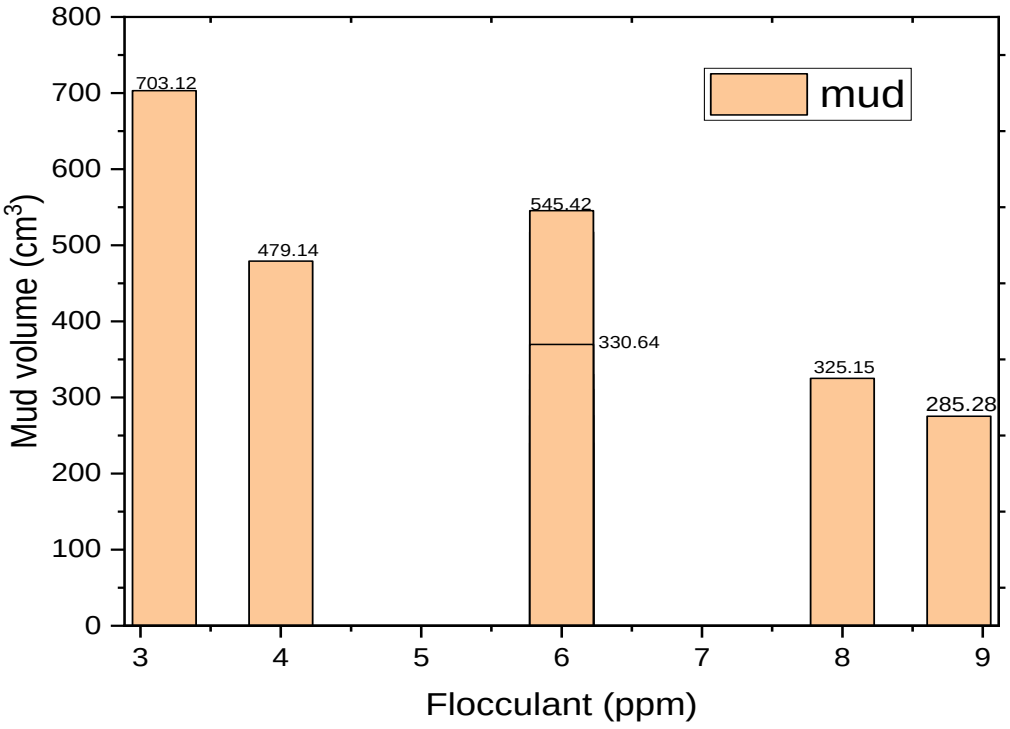

Figure 4. 2: Relationship between mud volume and flocculant dosage

4.8 The colors of the clear juices

Color increases after the liming process due to the reactions of alkaline degradation of reducing sugars agree that color decreases during clarification due to the removal of colorants by the calcium phosphate flocs (Eggleston, 1998). As the Brix content increases during the evaporation stage, the juice color increases. Several factors such as reaction time, juice pH and sugar concentration contribute to the increase in color. Color formation from the Maillard reaction is dominant at the earlier stages of evaporation followed by alkaline degradation reactions (Eggleston, 1998).

The colors of clear juice after clarification are presented in table 4.5. They were observed that low and high colors values were 1020.50 and 5482.46 IU, respectively, and these were gained after treatments with different dosages of the flocculant and limed pH of filtrate juice. These wide ranges of responses indicated that the colors values were significantly affected by the treatments of limed filtrate at different pH values and at different dosages flocculant. For example, the combination of 3.17 ppm flocculant with the 7.10 pH of limed filtrate juice generated a color value of 1020.50 whereas that of 8ppm flocculant and 6.80 pH of limed filtrate juice generated a color of 5482.46 IU. The result shows high color to mean having a higher proportion of juice pigment which was not removed during the clarification and containing higher amount of impurities vice versa.

The model F value of 8.48 implies the model is significant. There is only a 0.56% chance that a Model F Value this large could occur due to noise. Values of probability F less than 5% indicate model terms are significant. In this case B, A^2, AB are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The lack of fit F value of 3.30 implies the lack of fit is not significant relative to the pure error. There is a 13.68% chance that a lack of fit F value this large could occur due to noise.

The predicted R Squared of 0.3090 is not as close to the adjecent RSquared of 0.7137 as one might normally expect. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case ratio of 9.365 indicates an adequate signal. This model can be

used to navigate the design space. So a model equation in terms actual factor was developed as shown below:

$$C = -60530.54135 + 9408.16734 \times A + 8250.69745 \times B - 120.61573 \times A^2 - 1122.71250 \times A \times B$$

Where C=Predicted color, A=Flocculant, B= Limed filtrate juice pH values before clarification.

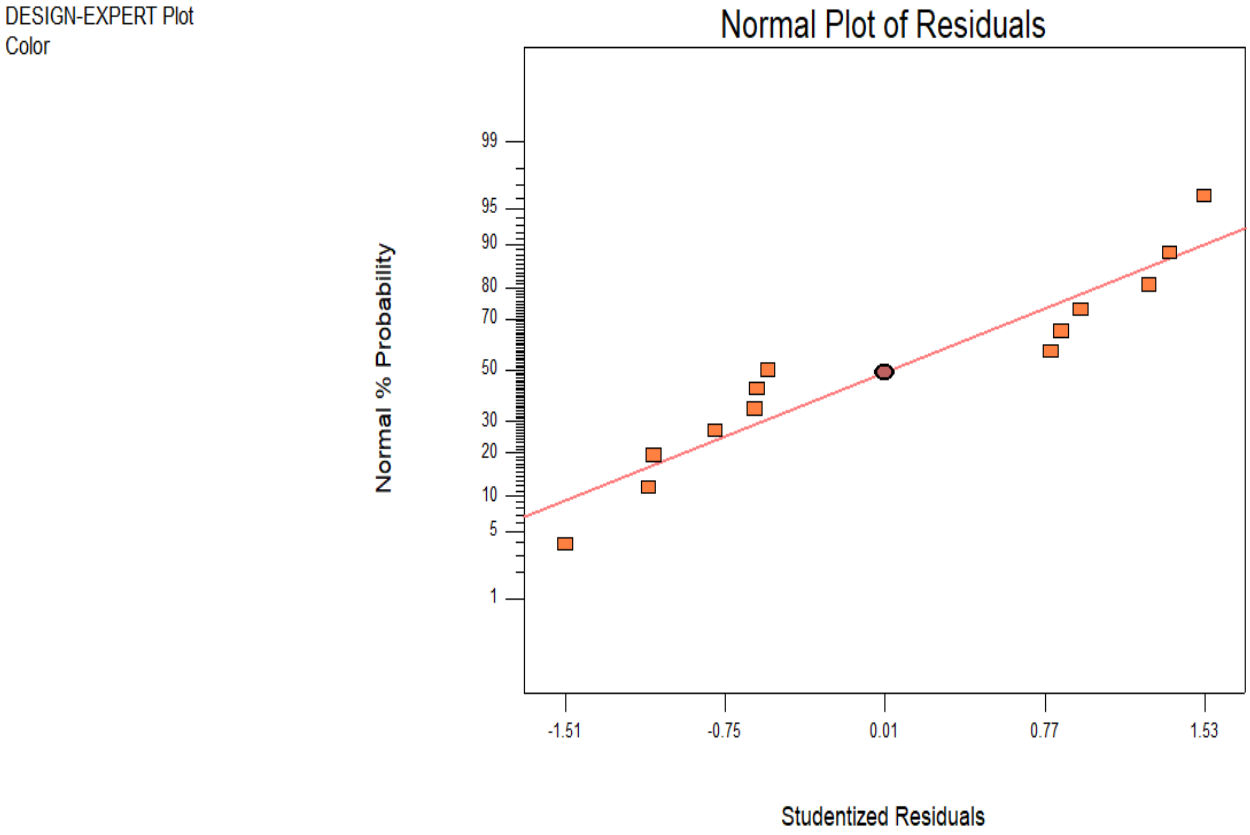

Figure 4. 3: The actual by predicting plot for color points

The actual by predicting plot shows how well it was fitted. The actual values form a scatter of points around each mean curve. It shows the actual color values on the y-axis and the predicted values on the x-axis. The points were randomly distributed along the diagonal line within 95% confidence limit region.

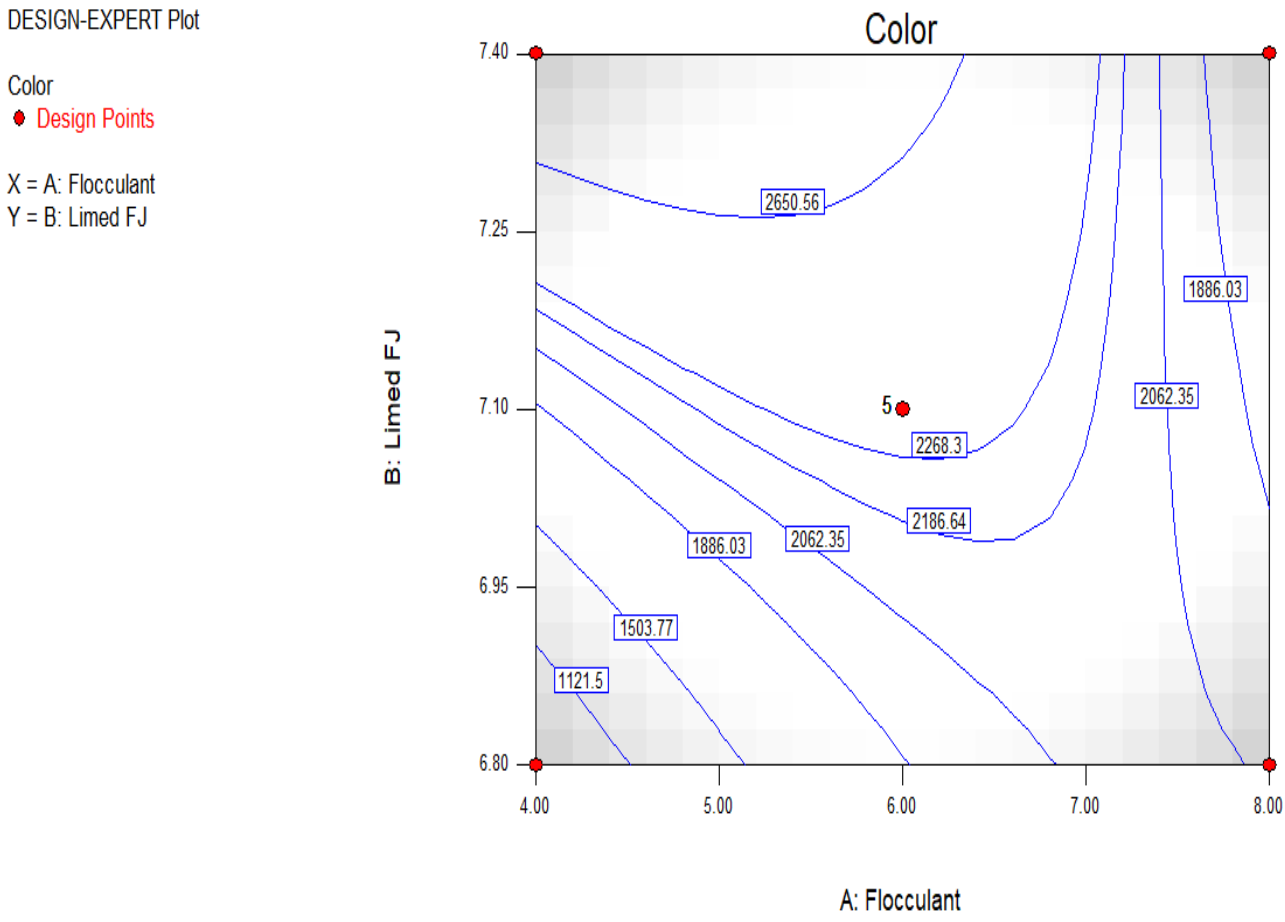

Figure 4. 4: Contour plot of color verses factors

Initially color increase as the liming increases from 6.80 to 7.10 and flocculant dosing from 4 to 7ppm. The color also increases as the liming increases from 7.10 to 7.40 at the range of flocculant 4 to 7.10. But it as well became decrease when the dosing of flocculant increases from 7.01 to 8 ppm with the increase of liming up to 7.40 pH. As a conclusion from the above contour and three dimensional figures even if liming increases to 7.40, and the tendency of flocculant to retard formation of color begins from 7.01 to 8ppm.

4.8.1 The interaction effect of the colors, flocculant dosages and LFJ pH

The interaction effect between the factors and the response of the colors in clear juice were significant as they shown in the graph below. But the general fact from the graph shows us at high levels of limed filtrate juice pH and high level of flocculant dosages the colors obtained were low, and in the same time at low pH and at high concentration of flocculant the colors of clear juice also low. When colors are removed from the clarification system, they show that colorant matters reduced as much as possible when subjected with higher values of the factors. The colors removal as seen from the interaction graph below show, they mostly affected by the concentrations of flocculant dosages.

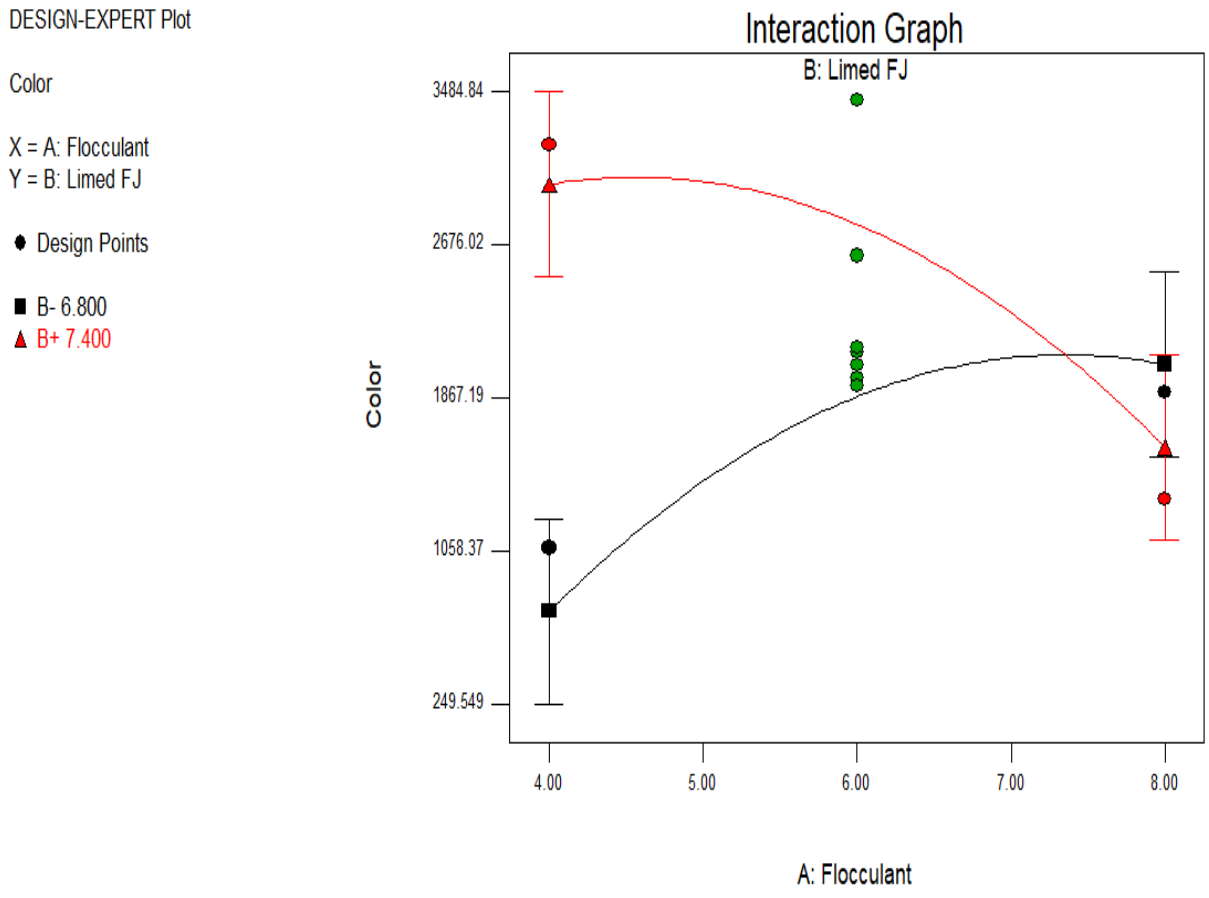

Figure 4. 5: Interaction curve of the colors verses the factors

4.9 The turbidity of the clear juice

The turbidities were observed that the minimum and the maximum turbidity values were 995.025 IU and 34129.3 IU respectively. These wide range of responses indicated that the turbidities values were significantly affected by the dosages flocculant and pH of limed filtrate juice. For example, the combination of 4 ppm flocculant with the 6.8 pH of limed filtrate juice generated a turbidity value of 995.025 whereas that of 6 ppm flocculant and 7.52 pH of limed filtrate juice generated a turbidity of 34129.3 IU.

The model F value of 16.44 implies the model is significant as it was shown in appendix C (3.1 and 3.2). There is only a 0.05% chance that a model F value this large could occur due to noise. Values of probability F less than 5% indicate model terms are significant. In this case B, B^2 are significant model terms.

The lack of fit F value of 3.03 implies the it is not significant relative to the pure error. The predicted R squared of 0.4852 is not as close to the adjusted R squared of 0.7943 as one might normally expect. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case 12.295 indicates an adequate signal. This model can be used to navigate the design space. So a quadratic model of turbidity was developed in terms of the actual factors as shown below:

$$T = 6 + 3.18753\varepsilon + 1304.39584 \times A - 9.24316\varepsilon + 005 \times B + 66895.54348 \times B^2$$

Where T is the turbidity of the clear juice

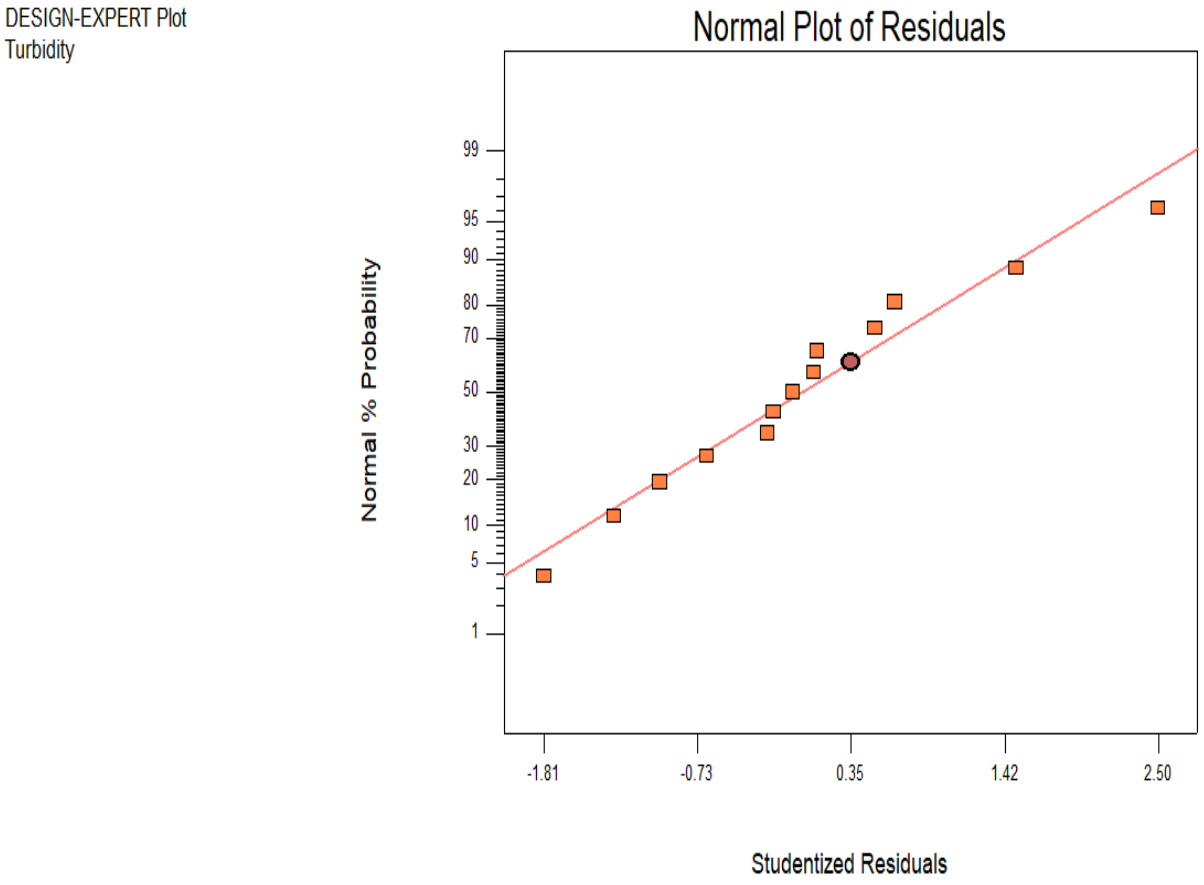

Figure 4. 6: The actual by predicting plot for turbidity points

The actual by predicting plot shows how well it was fitted. The actual values form a scatter of points around each mean curve. It shows the actual turbidity values on the y-axis and the predicted values on the x-axis. The points were randomly distributed along the diagonal line within 95% confidence limit region.

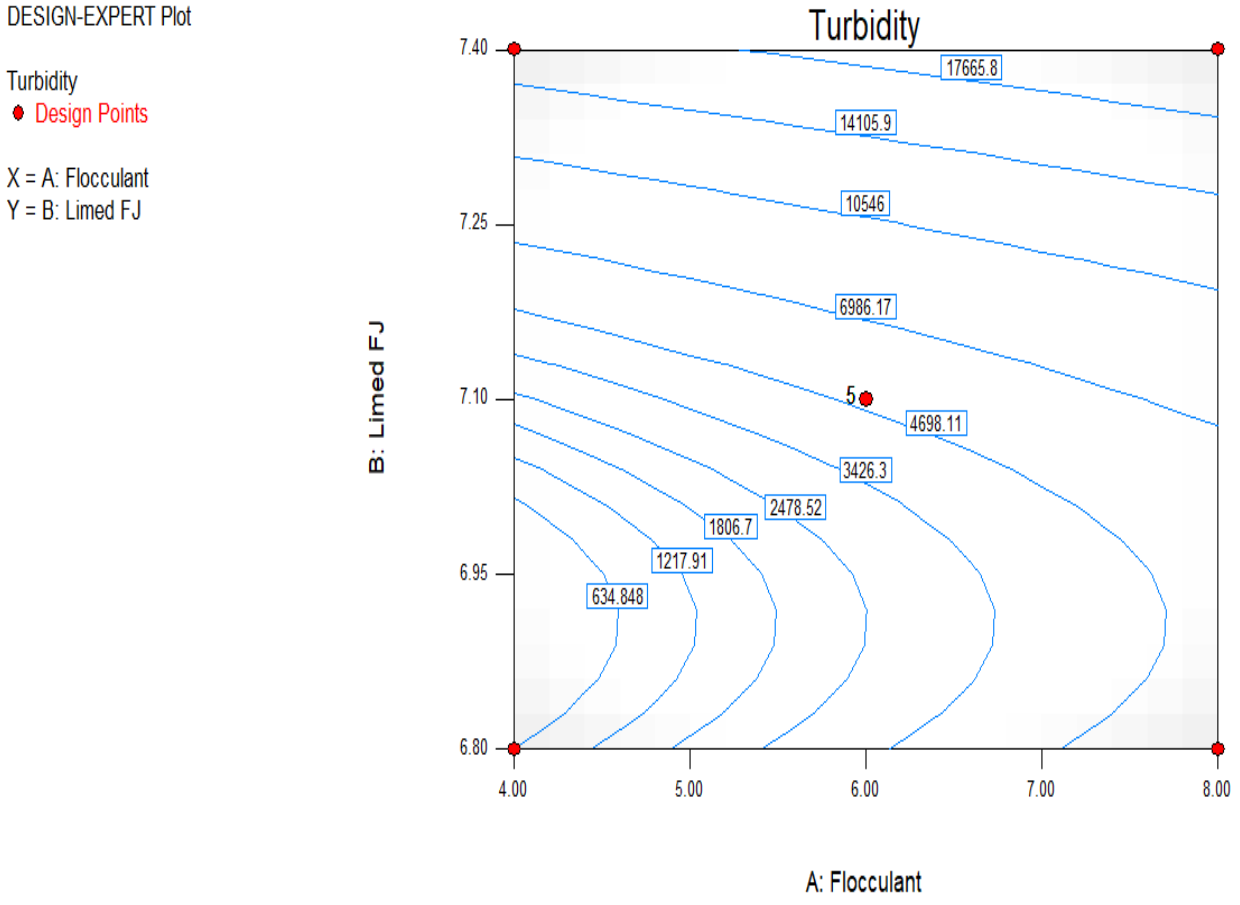

Figure 4. 7: Contour plot of the turbidity verses the factors

Minimum turbidity was observed for filtrate juice pH of acidic medium, but in the range of flocculant concentration. At a constant concentration of flocculant especially 8ppm and pH of lime filtrate starting from 7.25 to 7.40 the turbidity is significantly increased as it seen from the contour graph. Therefore, it is difficult to remove turbidity of clear juice for filtrate juice which has been treated within the range of basic even though the concentration of flocculant has become higher.

The plots assure turbidity is greatly affected or disturbed for the increase of liming even if flocculant dosing is a significant factor. This means for the increasing the level of limed filtrate juice especially at pH of 7.52 and above the clarified juice is more turbid. So during the liming process in filtrate clarification care should have to take place.

In the same way by studying the interaction figure below, there was an insignificant interaction effect among the factors and the turbidity of clear juice. Furthermore turbidity increases as liming increases.

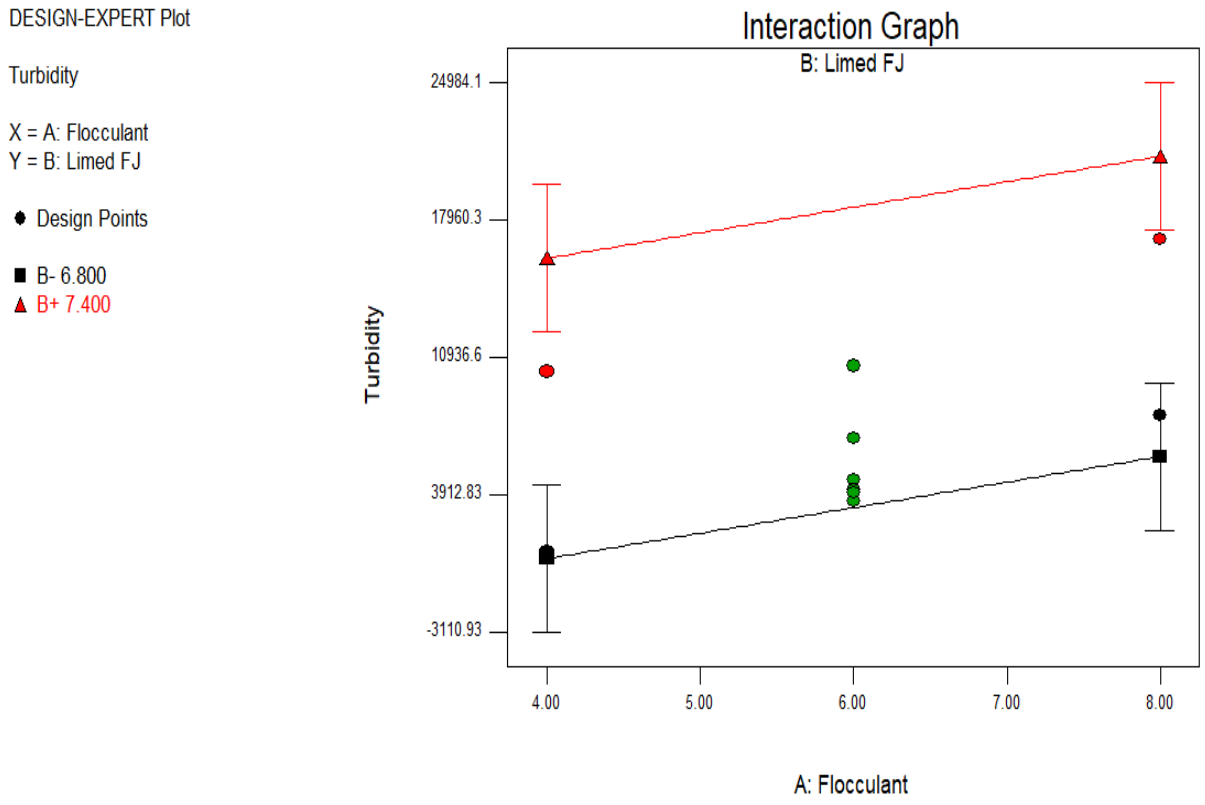

Figure 4. 8: Interaction curve of turbidity verse factors

4.10 The clear juice pH

It was observed that the minimum and the maximum the pH of clear juice values were 6.48 and 7.8 respectively. This wide range of responses indicated that the clear juice pH value was significantly affected by the dosage flocculant and pH of limed filtrate juice. For example, the

combination of 3.17 ppm flocculant with the 7.10 pH of limed filtrate juice generated a clear juice pH value of 6.48 whereas that of 6 ppm flocculant and 7.52 pH of limed filtrate juice generated a clear juice of 7.8.

The Model F-value of 12.66 implies the model is significant. There is only a 0.14% chance that a Model F Value this large could occur due to noise. Values of Probability F less than 5% indicate model terms are significant. In this case A, B, A^2 is significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The predicted R Squared of 0.6010 is in reasonable agreement with the adjacent R Squared of 0.7445. Adequacy precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case the ratio is 10.897 it indicates an adequate signal. This model can be used to navigate the design space. Therefore, a quadratic model of clear juice pH was developed in terms of the actual factors as shown below:

$$pH = -1.58515 + 0.60071 \times A + 0.93824 \times B - 0.042337 \times A^2$$

Where pH= clear juice pH, A=Flocculant, B= Limed filtrate juice pH before clarification

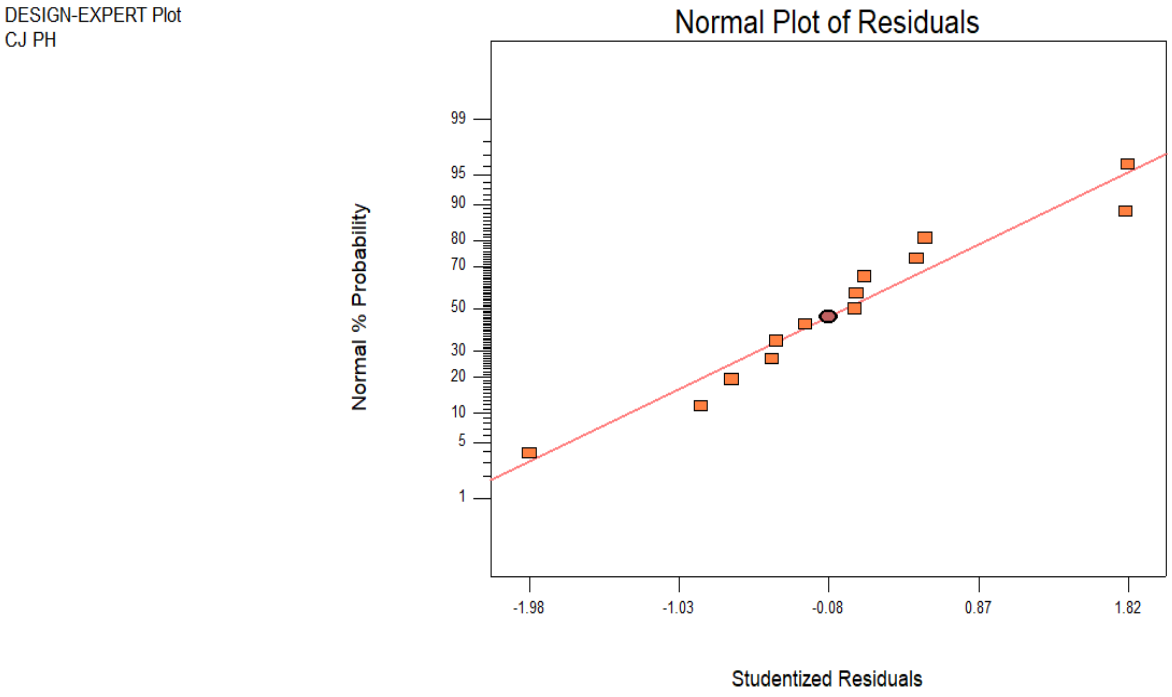

Figure 4. 9: The actual by predicting plot for clear juice pH points

The actual by predicting plot shows how well it was fitted. The actual values form a scatter of points around each mean curve. It shows the actual clear juice pH values on the y-axis and the predicted values on the x-axis. The points were randomly distributed along the diagonal line within 95% confidence limit region.

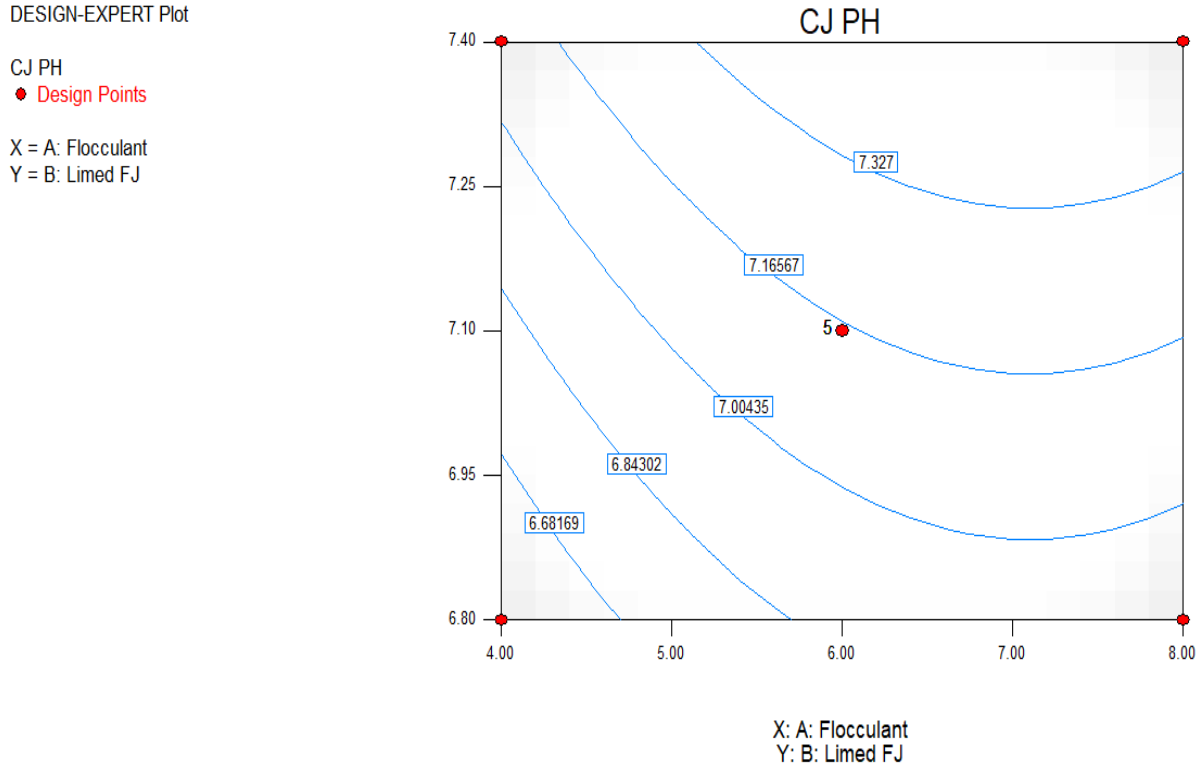

Figure 4. 10: Contour plot of CJ pH verse factors

The above plot shows that as the liming and flocculant dosing decreases the formed clear juice pH at the same time decrease. For the increase of liming of filtrate and dosing of flocculant within the range the clear juice increases proportionally. But for the pick of the range of flocculant and low level of liming the obtained pH was between 6.7 and 7.0. In general the potential of liming is greater than flocculation on clear juice pH.

4.10.1 Interaction effect between clear juice pH, flocculant and the LFJ pH

In general, there was no interaction between the factors and clear juice pH response. But as it is seen from the above graph for the increment of flocculant and limed filtrate juice having 7.4 pH, and there was a visible increasing of the clear juice pH. On the other side there was a decrease of clear juice pH when the flocculant is 6 to 8 ppm and the limed filtrate juice 6.80 and below.

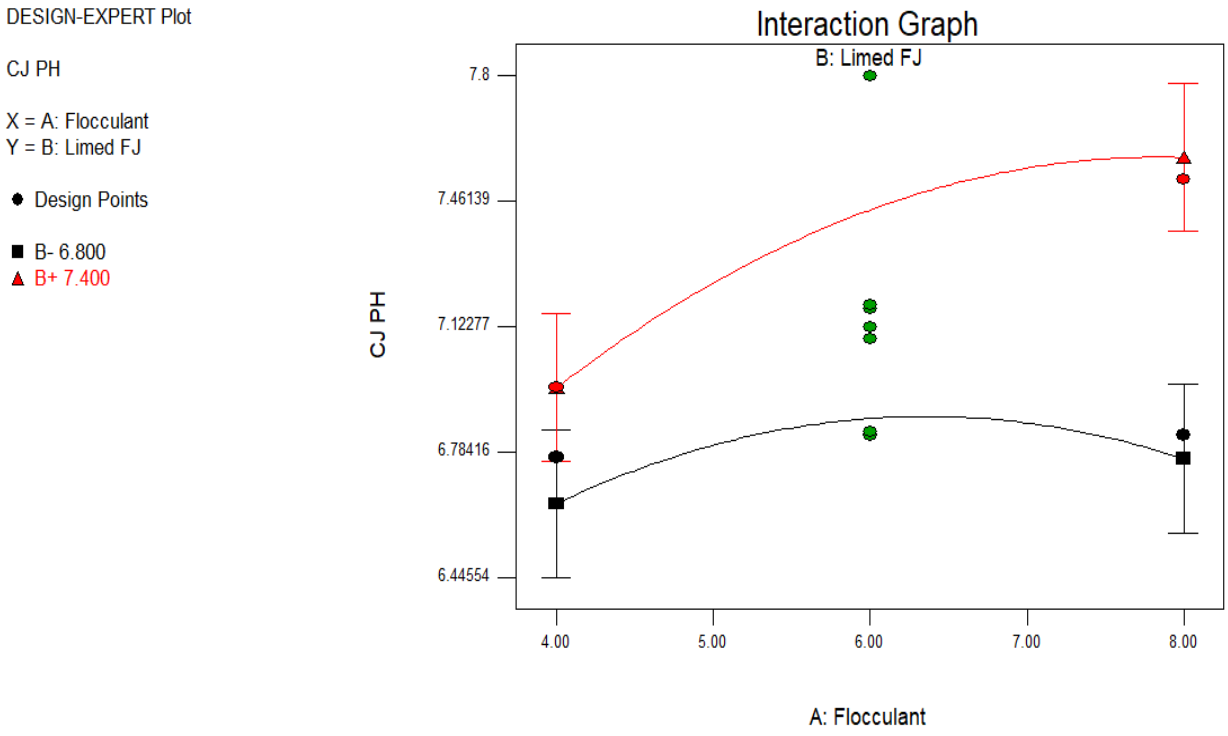

Figure 4. 11: Interaction curve of CJ pH verses factors

There was no interaction effect among the factors and clear juice pH at all. But for the treatment of liming below 6.8 and dosing of high level flocculant a slight increase of pH was observed before.

4.11 ANOVA for results of calcium oxide content for clarified filtrate juice

The appendix B of ANOVA result show model F value of 0.08 implies the model is not significant relative to the noise. There is a 92.51 % chance that a model F value this large could occur due to noise. Values of probability F less than 5% indicate model terms are significant. In this case there are no significant model terms. Values greater than 0.1000 indicate the model

terms are not significant. A negative predicted R squared implies that the overall mean is a better predictor of the response than the current model. Adequate precision measures the signal to noise ratio. A ratio of 0.80 indicates an inadequate signal and this was not used to navigate the design space. So that was the reason for calcium content in clear juice become insignificant.

4.12 Settling rates

The settling rate of the mud is very important parameter for juice clarification. The volume of clear juice produced in a Dorr clarifier is directly related to the mud settling rate. Mud-related trouble is the result of a lower settling rate of muddy juice after liming and sulfitation. (Edey, 1999) Suggested that in order to increase the settling rate of the calcium phosphate microfloc particles and improve the efficiency of separation of the liquid–solid system, an anionic flocculant (a high molecular weight copolymer of acrylamide and sodium acrylate) is added to the juice.

The fastest final settling rate was observed with the combination of flocculant at 6.00 ppm and limed filtrate pH of 7.10 having 2.3cm/min, whereas the slowest rate was at 4.00ppm and 6.80 pH of 0.25 cm/min. In addition, the fastest initial settling rate was found at 6.00ppm of flocculant and 7.10 pH of limed filtrate juice whereas the slowest rate was at 4.00ppm and 6.80 of pH. The different combinations of milk of lime addition and flocculant gave various settling rate values ranging from 0.25 to 2.3 cm/min.

4.13 Optimum points for combined pH, the turbidity and the color

By using the Design Expert software of 6.0.8 ten solutions were observed in this study having an optimum range of color, clear juice pH, minimum range of turbidity and a hundred percent desirability. The results were shown in the table 4.6 below.

Table 4. 6: The optimum solution at which the flocculant dosage and LFJ of pH

Number	Flocculant	Limed FJ	CJ pH	Turbidity	Color	Desirability
1	4.23	6.85	6.62977	358.191	1110.19	1.000
2	4.52	6.87	6.71368	622.686	1359.02	1.000
3	4.40	6.84	6.6557	698.224	1175.03	1.000
4	4.35	6.87	6.67428	395.753	1250.07	1.000
5	4.45	6.93	6.74627	457.971	1483.91	1.000
6	4.35	6.91	6.70514	311.149	1360.78	1.000
7	4.05	6.97	6.69132	164.904	1404.57	1.000
8	4.67	6.95	6.81691	841.234	1676.5	1.000
9	4.29	6.88	6.66562	294.567	1230.75	1.000
10	4.21	7.01	6.77431	869.28	1657.12	1.000

4.13.1 Process optimization and validation

The desirability function is expressed numerically from a scale of 0 to 1 (lowest to highest desirability) and denotes the degree of importance in obtaining the desired response value (Harrington, 1965). A desirability function value can be constructed by using two different goal optimization constraints: minimum and within range. On the basis of the fitted quadratic models, an optimized response value can then be predicted by using the chosen goal optimization criteria that maximizes the desirability function. So observing table 4.6 the desirability of each combination with different concentration of flocculant and limed filtrate juice was one hundred percent.

CHAPTER FIVE

5. CONCLUSIONS AND RECOMMEDATIONS

5.1 Conclusions

All Ethiopian sugar factories have been practiced for the production of white sugar plantation by recycling of filtrate juice into the mixed juice tanks. This method has shown that the impossibility of getting an efficient yield. So this study has shown to address the possibility of clarifying the filtrate juice separately in the specified plant would make the factory to more efficient instead of recycling. Even if the installed plant was supposed to process the filtrate juice by the floatation system and the possibility through this method is very difficult since the filtrate juice measured during this study was having at about 6 to 15ºBrix which is not viscous. In addition, the pH of the filtrate juice in average was 4.97. This shows it is not in the range of mixed juice pH so the filtrate juice become under conditions of high acidic media. Therefore, in filtrate juice, one can conclude that the opportunity of formation of non-reducing sugar might be high. The measured pH of filtrate juices before carrying out of the clarification was 4.46-5.46 and which were acidic. To clarify these filtrate juices in a separate stream the addition of milk of limes were mandatory to bring the juices in neutral media in order to reduce the inversion and also determination of phosphates in filtrate clarification were analyzed, but there concentrations within juices were so sufficient and need not addition during the clarification process.

Finally, the clarifications systems were performed in lab scale by optimizing only the flocculant dosages and liming the filtrate juices pH. The optimum points of the study were discussed in table 4.6. The required ranges of the levels of constraints obtained were 6.84-7.01 pH for limed filtrate juice and 4.05-4.69 ppm for flocculants. So the results obtained from this work indicated that the power to produce sugar can be improved if the optimum levels of the chemical dosages are practiced after the plant will be installed or planted. Experiencing the process under these ranges of conditions of levels improve the clear juice pH, the turbidity, the color, the purity rise, the settling rate and mud volume.

5.2 Recommendations

A clarification process through floatation carried out when the Brix of juice is 60^{o}c and above, so the Brix of filtrate juice is between 8 and 9^{0}Brix, due to this it is impossible to process by floatation system. Standard clarifier design produces mixed, which agitate the liquid more than necessary leading to longer settling and residence times. If they are too much turbulence in the tank, it takes longer to mud settle. More settling time means more inversion of sugar, which means sugar loss and more color. Additionally, improper placed flash tank allows more air to get into the tank bond to the flocculant preventing the mud from settling properly. Crampion LLT clarifier can actually help to increase efficiency and reduces sugar losses use a tip flash tank clarifier. It contains troughs making quicker and easier to distribute juice and reduce the opportunity of air entrainment. So this plant has well diffusing turbulence which can shorten 75% residence times and reduces turbulence and faster settling time leads increase sugar production as well as improve color and purity. So this plant reduces the settling time from 120minutes to 30 minutes. So this plant can be used for filtrate clarification. Its full design will be further studied by me and design and fabrication engineers.

The second recommendation is lime purity, the reason for this is impure lime will produce fouling on heat exchangers and increase maintenance cost so that the lime should be exchanged by another which fulfills the standard quality of lime.

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APPENDICES

Appendices A

Model fitting to results of clear juice for clarified filtrate juice

1. 1 ANOVA for results of clear juice pH after clarification

Source		Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model		1.11	3	0.37	12.66	0.0014	significant
	A	0.27	1	0.27	9.39	0.0135	
	B	0.63	1	0.63	21.65	0.0012	
	A^2	0.20	1	0.20	6.93	0.0272	
Residual		0.26	9	0.029			
	Lack of Fit	0.19	5	0.037	1.92	0.2730	not significant
	Pure Error	0.077	4	0.019			
Cor Total		1.38	12				

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1. 2 Summary fit for results of pH for clear juice

Std. Dev.	0.17	R-Squared	0.8084
Mean	7.05	Adj R-Squared	0.7445
C.V.	2.43	Pred R-Squared	0.6010
Press	0.55	Adeq Precision	10.897

1. 3 Summary for coefficient estimate of pH for clear juice

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High	VIF
Intercept	7.16	1	0.062	7.02	7.30	
A-Flocculant	0.19	1	0.060	0.048	0.32	1.00
B-Limed FJ	0.28	1	0.060	0.14	0.42	1.00
A^2	-0.17	1	0.064	-0.31	-0.024	1.00

Final equation in terms of coded factors:

$$CJ\ pH = 7.16 + 0.19\ \times A + 0.28\ \times B - 0.17 \times\ A^2$$

Final equation in terms of actual factors:

$$CJ\ pH = -1.58515 + 0.60071 \times A + 0.93824\ \times B - 0.042337 \times A^2$$

1. 4 Summary for diagnostic statistics of pH for clear juice

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outlier t	Run Order
1	6.77	6.52	0.25	0.348	1.807	0.435	2.134	12
2	6.83	6.89	-0.061	0.348	-0.442	0.026	-0.421	7
3	6.96	7.08	-0.12	0.348	-0.892	0.106	-0.881	8
4	7.52	7.45	0.066	0.348	0.478	0.030	0.456	9
5	6.48	6.56	-0.076	0.598	-0.698	0.181	-0.677	10
6	7.09	7.08	0.010	0.598	0.093	0.003	0.088	4
7	6.83	6.76	0.072	0.380	0.531	0.043	0.509	1
8	7.80	7.55	0.25	0.380	1.822	0.510	2.163	13
9	6.84	7.16	-0.32	0.130	-1.984	0.148	-2.493	2
10	7.17	7.16	0.013	0.130	0.084	0.000	0.080	3
11	7.18	7.16	0.023	0.130	0.147	0.001	0.139	11
12	7.09	7.16	-0.067	0.130	-0.417	0.007	-0.397	5
13	7.12	7.16	-0.037	0.130	-0.229	0.002	-0.216	6

Appendices B

Model fitting to results of turbidity for clarified filtrate juice

2. 1 ANOVA for results of turbidity of clarified filtrate juice

Source		Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model		7.829E+008	3	2.610E+008	16.44	0.0005	significant
	A	5.445E+007	1	5.445E+007	3.43	0.0970	
	B	4.719E+008	1	4.719E+008	29.73	0.0004	
	B^2	2.565E+008	1	2.565E+008	16.16	0.0030	
Residual		1.428E+008	9	1.587E+007			
	Lack of Fit	1.130E+008	5	2.260E+007	3.03	0.1526	not significant
	Pure Error	2.983E+007	4	7.458E+006			
Cor Total		9.257E+008	12				

2. 2 Summary fit for results of turbidity of clarified filtrate juice

Std. Dev.	3983.96	R-Squared	0.8457
Mean	8620.96	Adj R-Squared	0.7943
C.V.	46.21	Pred R-Squared	0.4852
Press	4.766E+008	Adeq Precision	12.295

2. 3 Summary for coefficient estimate of turbidity of clarified filtrate juice

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High	VIF
Intercept	4915.98	1	1438.84	1661.10	8170.86	
A-Flocculant	2608.79	1	1408.54	-577.55	5795.14	1.00
B-Limed FJ	7680.30	1	1408.54	4493.96	10866.65	1.00
B^2	6020.60	1	1497.59	2632.82	9408.38	1.00

Final equation in terms of coded factors:

$$T = 4915.98 + 2608.79 \times A + 7680.30 \times B + 6020.60 \times B^2$$

Final equation in terms of actual factors:

$$T = 6 + 3.18753\varepsilon + 1304.39584 \times A - 9.24316\varepsilon + 5 \times B + 66895.54348 \times B^2$$

2. 4 Summary for diagnostic statistics of turbidity of clarified filtrate juice

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outlier t	Run Order
1	995.03	647.48	347.55	0.348	0.108	0.002	0.102	12
2	7960.00	5865.07	2094.93	0.348	0.651	0.057	0.629	7
3	10199.00	16008.09	-5809.09	0.348	-1.806	0.435	-2.132	8
4	16998.11	21225.67	-4227.56	0.348	-1.314	0.230	-1.378	9
5	1492.53	1226.59	265.94	0.380	0.085	0.001	0.080	10
6	6517.41	8605.37	-2087.96	0.380	-0.666	0.068	-0.644	4
7	3582.09	6095.59	-2513.50	0.598	-0.995	0.368	-0.994	1
8	34129.35	27818.77	6310.58	0.598	2.498	2.318	4.251 *	13
9	10497.51	4915.98	5581.53	0.130	1.502	0.085	1.636	2
10	6815.92	4915.98	1899.94	0.130	0.511	0.010	0.489	3
11	4676.62	4915.98	-239.36	0.130	-0.064	0.000	-0.061	11
12	4179.10	4915.98	-736.88	0.130	-0.198	0.001	-0.187	5
13	4029.85	4915.98	-886.13	0.130	-0.239	0.002	-0.226	6

Appendices C

Model fitting to results of color for clarified filtrate juice

3. 1 ANOVA for results of color for clarified filtrate juice

Source		Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model		5.117E+006	4	1.279E+006	8.48	0.0056	significant
	A	3514.72	1	3514.72	0.023	0.8825	
	B	1.651E+006	1	1.651E+006	10.95	0.0107	
	A^2	1.647E+006	1	1.647E+006	10.92	0.0108	
	AB	1.815E+006	1	1.815E+006	12.03	0.0085	
Residual		1.207E+006	8	1.509E+005			
	Lack of Fit	9.265E+005	4	2.316E+005	3.30	0.1368	not significant
	Pure Error	2.804E+005	4	70094.44			
Cor Total		6.324E+006	12				

3. 2 Summary fit for results of color for clarified filtrate juice

Std. Dev.	388.41	R-Squared	0.8092
Mean	2031.80	Adj R-Squared	0.7137
C.V.	19.12	Pred R-Squared	0.3090
Press	4.370E+006	Adeq Precision	9.365

3. 3 Summary for coefficient estimate of color for clarified filtrate juice

Factor	Coefficient Estimate	DF	Standard Error	95% CI Low	95% CI High	VIF
Intercept	2328.70	1	140.28	2005.22	2652.17	
A-Flocculant	-20.96	1	137.32	-337.63	295.71	1.00
B-Limed FJ	454.33	1	137.32	137.66	770.99	1.00
A^2	-482.46	1	146.00	-819.15	-145.78	1.00
AB	-673.63	1	194.20	-1121.46	-225.79	1.00

Final equation in terms of coded factors:

$$C = 2328.70 - 20.96 \times A + 454.33 \times B - 482.46 \times A^2 - 673.63 \times A \times B$$

Final equation in terms of actual factors:

$$C = -60530.54135 + 9408.16734 \times A + 8250.69745 \times B - 120.61573 \times A^2 - 1122.7125 \times A \times B$$

3. 4 Summary for diagnostic statistics of color for clarified filtrate juice

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outlier t	Run Order
1	1075.43	739.24	336.19	0.598	1.365	0.554	1.458	12
2	1896.35	2044.57	-148.22	0.598	-0.602	0.108	-0.576	7
3	3204.13	2995.15	208.98	0.598	0.848	0.214	0.832	8
4	1330.54	1605.97	-275.43	0.598	-1.118	0.372	-1.139	9
5	1020.50	1393.41	-372.91	0.598	-1.514	0.681	-1.677	10
6	1646.28	1334.13	312.15	0.598	1.267	0.477	1.326	4
7	1974.28	1686.18	288.10	0.380	0.942	0.109	0.935	1
8	3439.21	2971.21	468.00	0.380	1.531	0.288	1.703	13
9	1932.48	2328.70	-396.22	0.130	-1.094	0.036	-1.110	2
10	2107.24	2328.70	-221.46	0.130	-0.611	0.011	-0.586	3
11	2618.58	2328.70	289.88	0.130	0.800	0.019	0.781	11
12	2038.49	2328.70	-290.21	0.130	-0.801	0.019	-0.782	5
13	2129.83	2328.70	-198.87	0.130	-0.549	0.009	-0.524	6

Appendices D

4. 1 Range of the constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Flocculant	is in range	4.00	8.00	1	1	3
Limed FJ	is in range	6.80	7.40	1	1	3
CJ pH	is in range	6.48	7.80	1	1	3
Turbidity	minimize	995.03	34129.30	1	1	3
Color	is in range	1020.5	3439.21	1	1	3

4. 2 Solutions of the optimization

Number	Flocculant	Limed FJ	CJ pH	Turbidity	Color	Desirability
1	4.23	6.85	6.62977	358.191	1110.19	1.000
2	4.52	6.87	6.71368	622.686	1359.02	1.000
3	4.40	6.84	6.6557	698.224	1175.03	1.000
4	4.35	6.87	6.67428	395.753	1250.07	1.000
5	4.45	6.93	6.74627	457.971	1483.91	1.000
6	4.35	6.91	6.70514	311.149	1360.78	1.000
7	4.05	6.97	6.69132	164.904	1404.57	1.000
8	4.67	6.95	6.81691	841.234	1676.5	1.000
9	4.29	6.88	6.66562	294.567	1230.75	1.000
10	4.21	7.01	6.77431	869.28	1657.12	1.000

4. 3 Number of Starting Points for the optimization

Flocculant	4.63	4.52	5.80	5.15	7.70	4.15	7.93	7.26	7.57	5.01
Limed FJ	7.09	6.87	7.37	6.99	6.84	7.16	7.25	7.21	7.04	7.16

Appendices E

6. 1: Photo of flotation filtrate juice clarification plant

6. 2: Photo of reaction tanks in flotation system

6. 3: Photo of sample of the filtrate juice

6. 4: Photo of measured flocculant powder

6. 5: Photo of prepared flocculant solution

6. 6: Photo of the heated filtrate juice at 95^0C

6. 7: Photo of the clarified filtrate juice at 6 ppm of flocculant and LFJ of 7. 10 pH

6. 8: Photo of the clarified filtrate juice at 6 ppm of flocculant and LFJ of 6. 68 pH

6. 9: Photo of the clarified filtrate juice at 3.17ppm of flocculant and LFJ of 7.10 pH

6. 10: Photo of insoluble solid filtered from 10grams of lime

Appendices F

Sugar Engineering Terminology

Bagacillo: - Fine fraction of bagasse obtained by screening or pneumatic separation, generally used as a filter aid in filtration.

Bagasse: - Cane residue leaving mills after extraction of juice.

Brix: - Measure of dissolved solids in sugar juice, liquor or syrup using a refractometer, otherwise referred to as refract metric dry solids. For solutions containing only sugar and water, Brix = % sugar by mass. Spindle Brix is determined using a hydrometer, but is now seldom used.

Clarifier: - Apparatus for the separation by sedimentation of suspended solids from a turbid sugar solution.

Clarified juice: - Juice from clarifiers, also referred to as clear juice.

Color:- Attenuation index, determined by absorption of light under defined conditions. Generally measured using the ICUMSA method at 420 nm, and referred to as ICUMSA units or IU.

Dissolved solids: - Dissolved materials are all solute material which is in solution, including sucrose, monosaccharaides, ash and other organic impurities.

Evaporator effect: - One of a system of evaporators operates in series as a multiple effect system (e.g. the first effect, second effect). Condensates and vapors are labeled correspondingly (e.g., first condensate and vapor one condensate and vapor from the first effect, respectively).

Extraction: - The proportion of sugar extracted from cane in the extraction plant; equals mass of sugar in raw juice as a percentage of mass of sugar in cane.

Extraneous matter: - All cane leaves and tops, mud, soil, roots, rocks, stones and tramp iron delivered with the cane.

Filter cake: - Material retained on the filter screens and discharged from the filters after filtering clarifier muds.

Filtrate: - Liquid passed through the screens of the filters.

Flocculant: - Polyelectrolyte in solution added to juice to assist clarification.

Imbibition: - The process of adding imbibition water for the extraction plant to increase extraction. Sometimes incorrectly referred to as maceration (steeping cane in juice). Water added is called imbibition water.

Massecuite: - The mixture of crystals and mother liquor is resulting from the crystallization process. Massecuite are classified according to purity as A, B, or C massecuite.

Molasses: - The mother liquor separated from the crystals by centrifuging. A, B or C molasses is derived from the corresponding massecuite. C molasses are also referred to as final molasses.

Non sucrose: - Dissolved solids contained in any process stream other than sucrose.

Non sugar: - A common overall term for dissolved solids other than sugar contained in any process stream.

Pan or vacuum pan: - Vacuum evaporative crystallizer used in the sugar industry to crystallize sugar from liquor, syrup or molasses.

Phosphatation: - Clarification using phosphoric acid and lime, in which certain non-sugar components are removed by flotation.

Polarization (or pol):- The apparent sucrose content expressed as a mass percent measured by the optical rotation of polarized light passing through a sugar solution. This is accurate only for pure sucrose solutions.

Purity: - The true purity is the sucrose content as a percent of the dry substance or dissolved solids content. The solids consist of sugar, plus non sucrose components such as invert, ash, and colorants. Apparent purity is expressed as polarization divided by refractometer Brix, multiplied by 100.

Reducing sugars: - Generally referred to and/or interpreted as invert sugar, determined by measuring, reducing substance content by laboratory analysis.

Re melt: - Syrup made from centrifuged low-grade sugar, which is dissolved or re melted and returned to the high grade boiling.

Sucrose:- Pure chemical compound $C_{12}H_{22}O_{11}$ known as white sugar, generally measured by polarization in pure solution or by GC or HPLC in impure solution. The chemical term is β-D-fructofuranosyl @alpha;-D-glucopyranoside.

Sugar: - A term for the disaccharide sucrose and products of the sugar industry essentially composed of sucrose.

Syrup: - The concentrated juice from the evaporators.

Trash: - Cane tops, leaves, dead stalks of cane and any other vegetable matter delivered with the cane.