INVESTIGATION OF THE USE OF TALO-PROCESS TECHNOLOGY IN SUGAR REFINING PROCESS

(A CASE STUDY OF DANGOTE SUGAR REFINERY APAPA LAGOS)

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FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA-NIGER STATE

A RESEARCH PROJECT SUBMITTED IN PARTIAL FULFILLMENT FOR THE AWARD OF BACHELOR OF ENGINEERING (B.ENG) DEGREE IN CHEMICAL ENGINEERING

NOVEMBER, 2004

DECLARATION

I, John Amaechi, do declare that this research project was solely done by me under the supervision of Engr. O.S. Azeez. And this project to my best of knowledge has not been submitted anywhere else.

22nd NOV, 2004.

John Amaechi **Date**

CERTIFICATION

This is to certify that this project "Investigation of the use of talo-process technology in sugar refining process (A case study of Dangote Sugar Refinery Apapa, Lagos)" was carried out by John Amaechi, of the Department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Minna, Niger state, Nigeria.

22nd Nov, 2004

Engr. Azeez O.S **Date**

(Supervisor)

Dr. F. Aberuagba **Date**

(Head of Department)

External Examiner **Date**

DEDICATION

I dedicate this research project to my late mum, Mrs. Virginia Amaechi who believed in me and ensured that my life has a meaning. You are always in my mind, I love you.

ACKNOWLEDGEMENT

My deepest thanks goes to God, Almighty through His son Jesus Christ fo His grace, kindness and mercy throughout this period of this research work.

I will like to express my profound appreciation to my project supervisor, Engr. O.S. Azeez whose guidance, assistance and supervision made this work a reality.

Also my sincere gratitude goes to the H.O.D. of Chemical Engineering Department, Dr. F. Aberuagba and the entire staff of the department, especially Engr. Abdulfatai Jimoh and Engr. A.S. Abdulkareem for their relentless efforts to impact knowledge on me.

My special thanks goes to mr. Adegoke Adetola, the assistance chief chemist of Dangote Sugar Refinery, Apapa; you are more than a friend, your practical help, assistance and encouragement in many ways were of immense benefit to me in doing this project.

To Engr. Kovo A.S. thanks for your assistance and corrections on this work.

I will not fail to express my gratitude to my good friend and brother Mekusproj for your encouragement, love and understanding as the work lasted, I love you, dearly.

To all casorites especially Eyam, Rachael and emah thanks for being my family in school. And my friends Godwin, Johnmary, Chigozie, Tosin, ifeoma, Opeyemi. Nancy, and Vivian, you all are wonderful people who have touched my life in no small measure. And to all my fellow course-mates cheer-up, the future holds great things for all of us.

Finally, my wonderful thanks to my family, especially my one and only Uncle, Mr. Ogwuru E.E., you are truly a father to me, your love, support , encouragement and more importantly, your efforts have brought me this far in life, I love you dearly sir.

ABSTRACT

This research work investigates the use of talo-process technology in sugar refining process (Dangote Sugar Refinery Apapa as a case study). The working conditions, operating parameters and the analysis of both the refined sugar and the by-product (scum) of the process were investigated. Operating parameters investigated were temperature, brix, chemical consumption rate, pH, colour and turbidity. Sucrose content, heavy metals, presence and quantity of waste generated in the process were examined.

The result obtained showed that percentage colour removal of the process was 45% compared to 25% of other conventional processes, low sugar loss (0.21-0.26%) was observed and small amount of scum was generated (0.559g/kg of the raw sugar refined). Investigation shown that chemicals used in the process could not be regenerated, and the amounts of heavy metals (iron (2110mg/kg), copper (10.02mg/kg), and lead (29.86mg/kg)) were also present in the scum. Small amount of iron (0.89mg/kg) was detected in the refined sugar, but other heavy metals were not detected in the product. The amount of heavy metals in the effluents of the process was too high and should be treated before being discharged into the environment. But, the amount of iron found in the refined sugar was satisfactory; thus, this process was better than other conventional processes used in raw sugar refining.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 General Overview

The brown colour of raw sugar (sucrose) processed from cane sugar or beet sugar is due to the impurities of both organic and inorganic matters that accompanied the raw sugar. Organic impurities such as Dextrins, gums, nitrogenous matter, organic acids (citric, glycolic and oxalic) and complex organic pigments (colourants) contaminated brown sugar (Brekham and Nesterenko, 1982). Also, large amounts of calcium, magnesium, silicon, iron, manganese and phosphate are the inorganic matters that add to the Impurities. These impurities make the raw sugar crystals quality and its colour to be poor, and of low demand domestically and industrially. Hence, the need for the raw sugar to be further refined.

Refined sugar has been produced in the past by different processes, such as double carbonation process, double sulphitation process and ion exchange process using granular (Kenana Sugar, 2000). And these processes have improved the quality of sugar produced, but not without their own problems. However, in 1919, Williamson first created a system to combine phosphatation and floatation (phosflotation); from then, many advances have been achieved and new floatation clarification processes have been developed in the sugar industry (Sugar Technology Rev, 1992). Since 1970's, a process combining phosflotation with either sulphitation or carbonation in which all precipitates are removed from the liquor by floatation. Though, this system has a higher colour removal, but it increased the capacity of sugar plant. One important advance made is the extensive application of highly efficient poly-acryl amide flocculants, which greatly increased the velocity and stability of the flotation-separation process (Bennett et al, 1973.)

After many years of research work into chemistry of refinery process, Tate and Lyle England developed a new process called talo-clarification process (Dangote Sugar, 1999). This advance in the application of special cationic surfactant called "talofloc" (aqueous dimethylammonium chloride) combines with the negatively charged colourants (major portion of colour) as well as other cationic impurities, and then precipitates together.

1.2 Objectives of the Research

The aim of this project is to investigate the talo-process technology in raw sugar refining to ascertain the following:

i. Its colour removal and clarification efficiency.

ii. The viability of this process to local sugar industry.

iii. Impact of this novel process to the environment.

1.3 Scope of the Research Work

The objectives shall be limited to the investigation of the wor conditions of the process (mode of operations), operating parameters and th analysis of both the product and by-product of this process. And only ra sugar of very high pol (VHP); that is 99.1 – 99.4% sucrose content shall used. Dangote Sugar Refinery Ltd, Apapa, shall be used as a case study.

1.4 Justification of the Research Work

This research work is initiated because of the need for the growing local sugar industry to develop a better refining technique, which is of better efficiency. And coupled with the fact that Nigerian Government has stopped the importation of refined sugar and sugar demand is high, and local production of sugar needs to be increased to meet this increasing demand. So this research work is important at this time to the nation.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Sources of Raw Sugar

Sucrose is the crystallised sugar used for domestic and commercial uses, which belongs to the class of sugar called disaccharide sugar ($C_{12}H_{22}O_{11}$). Sucrose is called 'sugar'.

Sugar occurs naturally in many plants and fruits like pineapple, carrots and sorghum. But, the main sources of sucrose are sugar cane and sugar beet.

2.1.1 Sugar Cane

Sugar cane is the chief source of processing sugar. It is grown in equatorial regions and similar regions of temperature and rainfall. But, where rainfall is insufficient for cultivation the cane is irrigated from rivers. Sugarcane approximately contains one tonne of raw sugar on every ten tonnes (Dangote Sugar, 1999). Raw sugar factories crush the cane to produce juice, which is chemically treated, evaporated and crystallised to raw sugar.

2.1.2 Characteristics of Raw Sugar (Sugar Cane)

The raw sugar is of two types, very high pol (VHP), which has high sucrose content of range 99.1-99.4% pol. The low pol raw sugar of 96-98% pol which has to be affined before refining. The moisture content of raw sugar is 0.2 – 0.3% and of colour range of between 200 – 1400 ICUMSA (Kenana Sugar, 2000).

However, all solids other than sucrose are impurities that can be invert sugars, organic and inorganic impurities. The raw sugar quality is also influenced by cane quality, variety characteristics, boiling techniques and purging efficiency (Kenana Sugar, 2000).

2.2 Sugar Production in Nigeria

In the last two decades, Nigeria was producing sugar locally, in spite of the fact that there was sugar importation to augment the quantity produced. sugar industries like Nigerian Sugar Company Ltd, Bacita and Lafiagi in Kwara State produced about 160,000 tons of refined sugar annually and Savannah Sugar Factory and Refinery in Adamawa State produced about 140,000 tons of refined sugar annually (Jibrin, 1994)

Since 1995, these industries were either producing at low capacity or not in production at all. And this made sugar importation the chief source of sugar in Nigeria. However, in the year 2000, Dangote Sugar Refinery at Apapa was established to refine an average of 2000 tons of raw sugar per day. But, there is no sugar factory in Nigeria that could supply the quantity of raw sugar needed by the refinery. Hence, the company relies on raw sugar importation from southern America countries for its operation (Dangote Sugar, 2000)

2.3 Sugar Market Demand

The demand of sugar for both domestic and industrial purpose has ever been increasing yearly. Also, the market of sweetening of foods of which is the dominance of sucrose as a major carbohydrate sweetener has widened applications of sugar in food and pharmaceutical industries. Some of its industrial application is in:

i. Sweetmeat, bread, biscuits, chocolates, condensed milk, jams, jelly etc.

ii. Syrups in the pharmaceutical industry

iii. Beverages including aerated beverages and fruit juices.

Also the use of sugar domestically has remained high.

2.4 Sugar Refining Processes

2.4.1 Double Sulphitation Process

This is a process whereby raw sugar is clarified by addition of sulphur dioxide (SO_2) and lime (CaO). The liquor is heated to about 70°C, then limed and sulphited in a sulphiter. The treated liquor is further heated to about 105°Cthen gas and air is removed before the liquor enters a clarifier, where the settling of the mud floc ($CaSO_3$) take place. Accumulated mud from the mud boot is withdrawn by gravity flow. Passing it through sulphitation vessel further bleaches the liquor. This process has low colour removal and clarification efficiency and a large plant capacity. Also, large quantity of mud floc ($CaSO_3$) is formed.

2.4.2 Double Carbonation Process

Raw sugar is treated with lime and CO_2 at about 78-80^{0}C in a carbonator, before the liquor enters a clarifier where the mud flocs ($CaSO_3$), is formed and this is separated by method of sedimentation. The clear liquor is withdrawn from the upper regions through the overflow for further treatment. The liquor is further clarified through a saturator where more CO_2 is absorbed. This process is more effective in removing colorants and other impurities than sulphitation but precipitates heavier alkaline filter mud and its disposal is difficult because of pollution problems.

2.4.3 Phosflotation and Sulphitation Process

Raw sugar of brix (61 – 63^{0}BX) is heated to 60 – 65^{0}C prior to preliming (CaO) before the liquor is treated in a reactor with CaO, SO_2 and H_3PO_4 to a P^H of 8.0 – 8.8. The treated liquor is heated in a heater to 75-80^{0}C then passed through an aerator to a mixer where flocculants is added. The $CaSO_3$ precipitate formed in the liquor can be removed together with calcium phosphate ($Ca_3(PO_4)_2$) formed by flotation, and both function as colour removal by SO_2 and $CaSO_3$ (based on absorption) can be utilised giving a higher decolourisation. The precipitates ($CaSO_3$) are heavier and more difficult to float and there is residual and more difficult to floats and there is residual of SO_2 in the clarified liquor.

2.4.4 Phosflotation and Carbonation Process

This system consists of low-level carbonation and phosflotation; which involves two steps of treatment and flotation unlike sulphitation process that is of one step. The CO_2 used in this process is either obtained from alcohol fermentation or flue gas. A tabular reactor is used for CO_2 absorption.

However, the operating conditions of the process are similar to that of phosflotation and sulphitation process; except, that there is two steps of treatment and flotation that is involved. But, it is of higher decolourisation efficiency and the amount of lime added is reduced considerably which reduces the amount of filter mud ($CaCO_3$ and $Ca_3(PO_4)_2$) produced.

2.5Talo-Process Technology in Dangote Sugar Refinery

2.5.1 Talo-Clarification Process

This is a process in which anionic high molecular weight colourant and other cationic impurities are precipitated by addition of special talo floc additive, a cationic surfactant (aqueous dimethyl amine polymer). The precipitated colourants and all other insoluble impurities are flocculated by phosphatation treatment and then separated by floatation using talo-flote flotation aid a co-polymer of Acryl amide and sodium acrylate (Dangote Sugar, 1999).

2.5.2 Chemical Used in the Process

1. Phosphoric acid (H_3PO_4): The concentration of the acid is 85% (food grade) with a specific gravity of 1.689. Its active ingredient is P_2O_5, which is 61.6% content of the acid.
2. Lime Sucrate: This is a mixture of 25% milk of lime ($Ca(OH)_2$), 25% water and 50% unclarified liquor (at 65^0 BX). The milk of lime is prepared at 10^0Be mol (18^0BX) and contains active ingredient of 94gm CaO per litre.
3. Talo-floc 'L': This is a cationic surfactant, a quaternary ammonium compound derived from fatty acids, an aqueous dimethyl amine polymer. It is a decolourising agent and non-hazardous with a specific gravity of 1.16.
4. Talo-flote '100':A free flowing powder flocculants, co-polymer of acryl-amide and sodium acrylate. It gives a strong adhesion to calcium phosphate precipitate. And it is prepared as a 0.1% solution

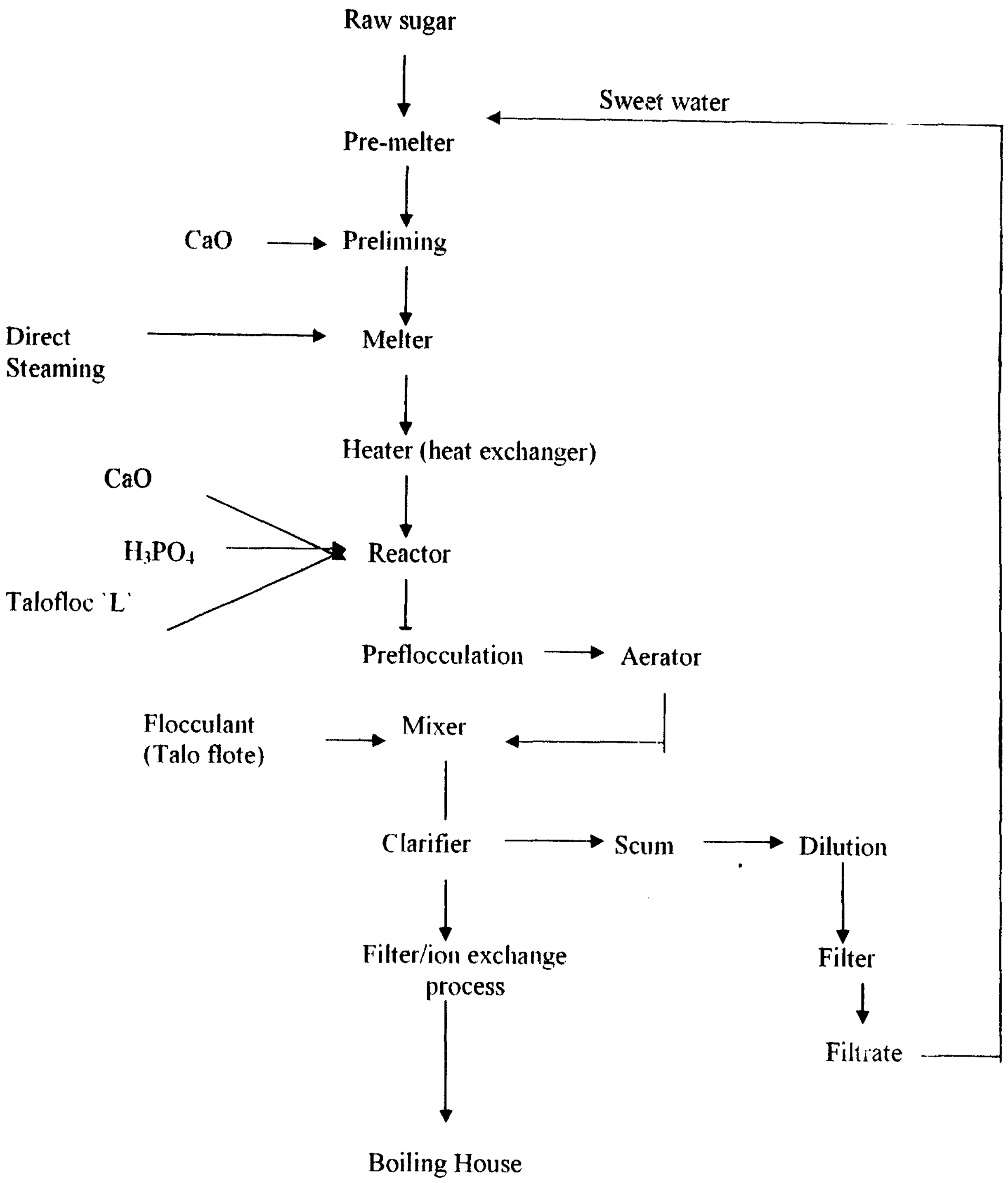

Fig2.1 Flow Sheet of Talo-Clarification Process

2.5.3 Mechanism of Flotation

Flotation process is a highly efficient technology for separating solid particles in liquids to remove them through addition of suitable air bubbles and flocculent. This process works on the principle of forming low aggregates of particles and bubbles, and the lower the density, the more quickly the float. The attachment of solid particles firmly to sufficient air bubbles is determined by physio-chemical factors and hydrodynamic parameters.

Physio-chemically, the properties of the solid particles surface is divided into; hydrophobicity: particles surface repel water from surfaces and tend to adhere to air bubbles by their own. So they can become firmly attached to bubbles and rise together spontaneously. While hydrophilicity: particles surface have affinity for water; and almost all naturally occurring solid particles and n inorganic chemical precipitates are hydrophilic and are invariably unflotable of which calcium phosphate is one of them (Svarovsky, 1981).

When talofloc, a surface agent is added to the sugar liquor before aeration, the air bubbles would attach to the solid particles and cause them to·float together. This is because of the adsorbed surfactant molecules on the surface of $Ca_3(PO_4)_2$, whereby the hydrocarbon chains of the surfactant make the surface of particles hydrophobic. Calcium phosphate is very effective in removing suspended matter (including chemically inert particles) in that the particles can trap minute air bubbles forming floccules that have a lower density than the liquor. They float different velocities depending on the size of the floccules and the amount of air occluded (Sugar Technology Rev. 1972). The floccules are coagulated by the flocculants to for a scum.

2.5.4 Air Bubbles Parameter

Air bubbles provides the lifting force for flotation of solid floccules, their sizes and numbers have great influence on the stability and velocity of flotation. A basic physical mathematics analysis is given below;

The force (F_1) causing a body to float in the liquor is:

$F_1 \quad = V(D_2 - D_1)$ -------------------------------------- 1

Where

V - the volume of the body (sphere)

D_2 - the density of the liquor

D_1 - the density of the body

The resistance force (F_2) to a body in motion is given by:

$F_2 \quad = CD_2 AV_2/ 2g$ --- 2

Where

C - the coefficient of motion resistance

A - the sectional area of the body

V_2 - the moving velocity of the body

And g - the acceleration due to gravity

The resistance coefficient,

$C \quad = 24 / Re$

Where: Re - the Reynolds number of the system examined. (Alan et al 1979)

For achieving good flotation, the following conditions are essential:

1. Air bubbles must be of microscopic size; sizes smaller than 30microns are preferable and sizes smaller than 50microns are acceptable (Sugar Technology Rev., 1972).

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1. Air bubbles must be of microscopic size; sizes smaller th 30microns are preferable and sizes smaller than 50microns ar acceptable (Sugar Technology Rev., 1972).

2. The quantity of bubbles should be sufficient but not too much, as the floccules can only enmesh a certain quantity of bubbles; excessive bubbles are useless and harmful.

2.5.5 Method of Aeration

Many aeration methods have been used in the flotation process in the sugar processing. In the early years, the aeration of liquor was due by injecting compressed air into it, or pumping either the whole or a part of the liquor through an injector to suck air in, but these methods formed many large bubbles (Sugar Technology Rev., 1972). The aerator (cavitation type) used in this process is o multi-knife style consisted of a high speed (about 2900rpm) inside a cylindrical shell (Kenana Sugar, 2000). The knives are machined to make their edges ver sharp, and both to increase the cutting effect on the bubbles, the shell form hundreds of small trough, with sharp edges on the inner surface. The liquor with airflows through the passage formed by clearance of rotor and shell and in cut b knives and ground by the shell; producing numerous minutes bubbles. All larges bubbles are broken down and eliminated.

2.5.6 Reaction Tank

The reaction tank consists of three compartments:

(i) Reactor

(ii) Primary flocculation chamber

(iii) Aeration chamber

(i) Reactor: The reactor has a stirrer in it; and the following reactions occur:

For the lime $CaO_{(aq)} + H_2O_{(l)} \rightarrow Ca(OH)_{2(aq)}$

For the phosphoric acid: $P_2O_{5(aq)} + 3H_2O_{(l)} \rightarrow 2H_3PO_{4(aq)}$

Overall reaction is given below:

$3Ca(OH)_{2(aq)} + 2H_3PO_{4(aq)} \rightarrow Ca(PO_4)_{2(aq)} + 6H_2O_{(l)}$

(ii) Primary Flocculation Chamber: Talo floc (surfactant) is added to form the floccules and the liquor overflow to the aeration chamber (Dangote Sugar, 1999)

(iii) Aeration Chamber: Air is introduced into the treated liquor to aid flotation ole clarification using a cavitation type aerator.

2.5.7 Talo-Clarifier

The aim of the flotation process is to achieve high quality clarified liquor with high separation rate and short retention -- time in conjunction with a small volume of concentrated scum and this depends on the previous treatment before the clarifier and the working of the clarifier.

Talo-flote is added into the liquor entering the clarifier In talo-clarifier, the liquor enters from the bottom through a gate stirrer, which plays the role of ensuring good flocculation, bubbles entrapment and eliminating turbulence in the feed liquor. The liquor is then distributed over the whole area of the clarifier. The floccules float to the surface and then gradually concentrate into scum, which is scraped off by a low – speed rotating scraper into an annular chute and discharged. The clarifier liquor flows towards the outer circumference of the clarifier and passes through annular pipes with small holes to be discharged into the weir box. This box controls the level of liquor in the clarifier, which is slightly lower than the overflow surface so that the scum has a suitable time (30 mins) for concentration to reduce its volume. The clarified liquor flows to the deep bed filters where the carry over flocs is removed.

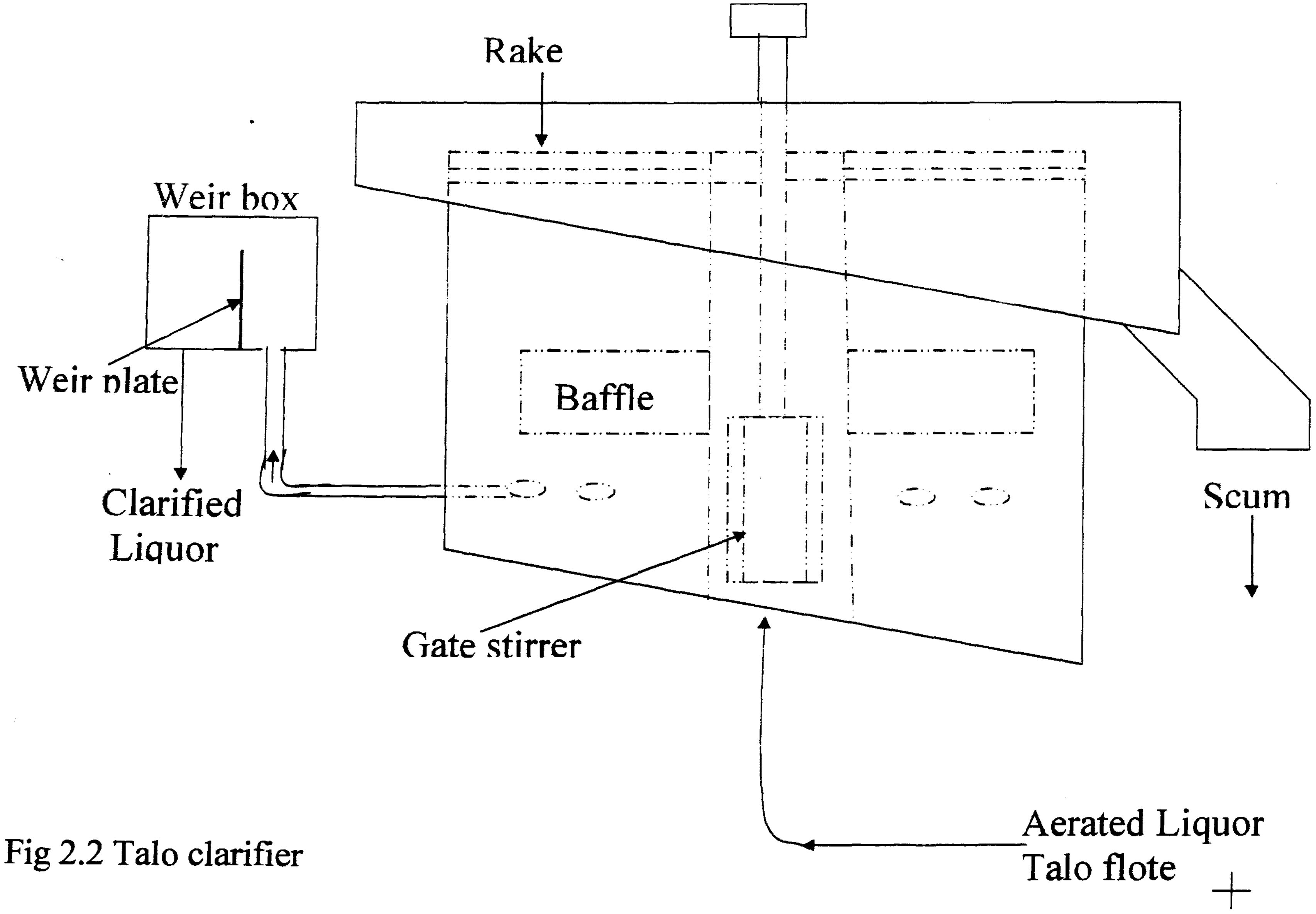

Fig 2.2 Talo clarifier

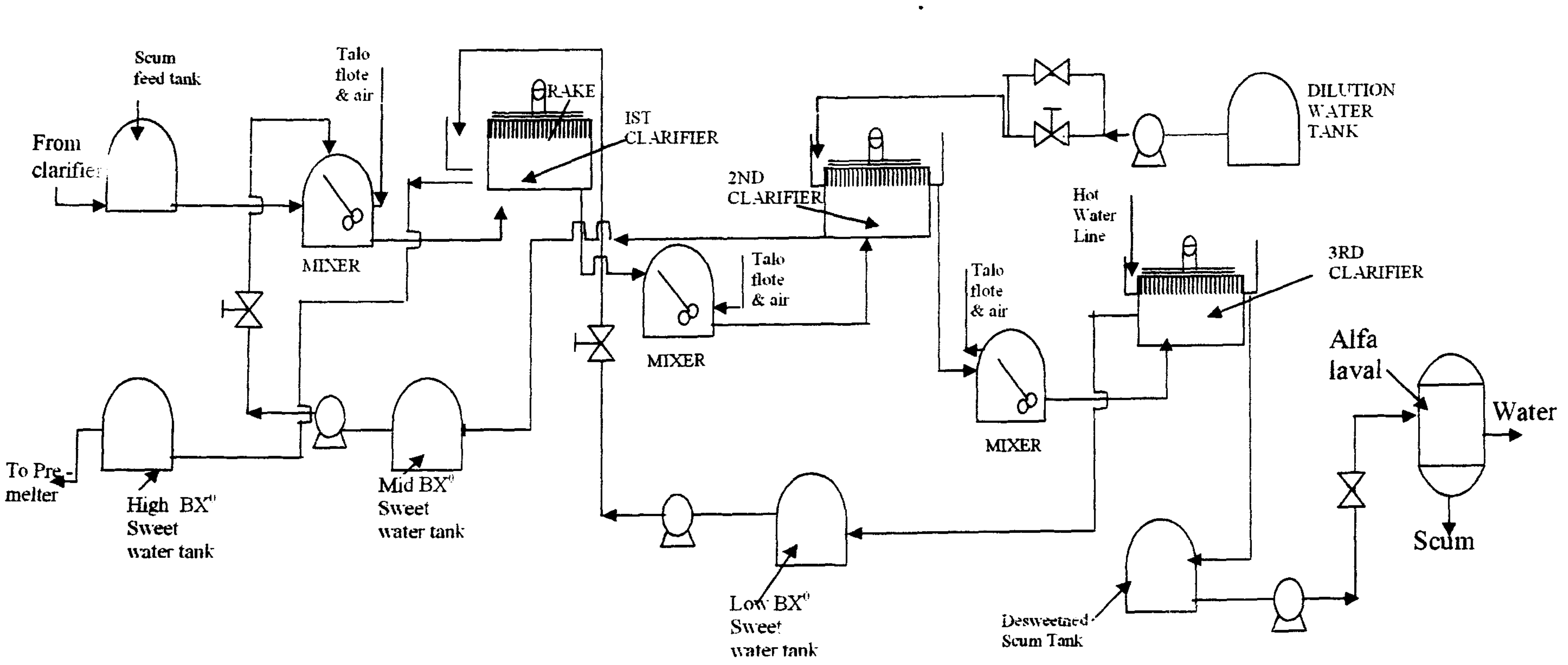

Fig 2.3 Scum Desweetening Flow Diagram

2.6 Scum Desweetentening Process

Scum produced in the clarifier undergoes a desweetening process to recover sugar through further clarification process. This process is a three consecutive stage counter – extraction process that involves aeration and more flocculants addition, followed by flotation in the clarifiers. The desweetening scum is pumped to Alfa-lava where water is extracted from the scum, and then, the semi-solid scum is discarded. However, the, sugar recovered in this process which is in solution form (sweet water) is used in raw sugar melting process at the pre-melter.

2.7 Talo-Process Technology Operation

The refinery plant is designed to operate for 24 hours in a day. And the talo – clarification process has a capacity to clarity an average of 2500 tons of raw sugar of very high pol (VHP) per day.

2.7.1 Operating Efficiency of the Process

Tate and Lyle England designed talo process for high % colour and insoluble impurities removal in raw sugar refining process. And this leads to high decolourisation efficiency when compared to other clarification processes that have lower efficiency of 25% (Kenana Sugar, 2000). While the Pol content in the scum after desweetening process is 0.1 – 0.4 %. (Dangote Sugar, 2003).

2.7.2 Operating Parameters

The efficiency of the Talo-process depends on the operating parameters of the process. These parameters determine how effective and reliable this technology will be. These parameters are listed below:

(i) Brix

(ii) Temperature

(iii) Chemical consumption

(iv) pH

(v) Flow rate

2.7.3 Operating Conditions

(a) Brix

(i) 66 -68^{0}BX of sugar slurry, in the pre – melter

(ii) 64.5 – 65^{0}BX of sugar liquor in the melters

(iii) 63.5 – 64^{0}BX of sugar liquor, in the reaction tank and Talo – clarifier.

(b) Temperature

(I) 40-45^{0}C in the pre – melters

(ii) 64 – 65^{0}C in the melters

(iv) 82 – 85^{0}C in the reaction tank.

(v) 80 -82^{0}C in the Talo-clarifier.

(c) Chemical consumption

(i) Lime Sucrate

<500 parts per million (ppm) of hydrated lime (CaO) on sugar solids

(ii) Phosphoric acid

200 -420 parts per million (ppm) of P_2O_5 on sugar solids.

(iii) Talo floc 'L'

100- 300 ppm on sugar solids

(iv) Talo flote

8 - 13 ppm on sugar solids

(d) pH

(i) 7.5 – 9.0 pH of sugar slurry in pre-melter

(ii) 7.0 – 7.5pH of liquor in melter

(iv) 6.9 – 7.2pH in the reaction tank.

(vi) 6.9 -7.2pH of the clarified liquor should be attained in the clarifier.

(e) Flow rate

The flow rate of the process should not be more than 200m^3/hr

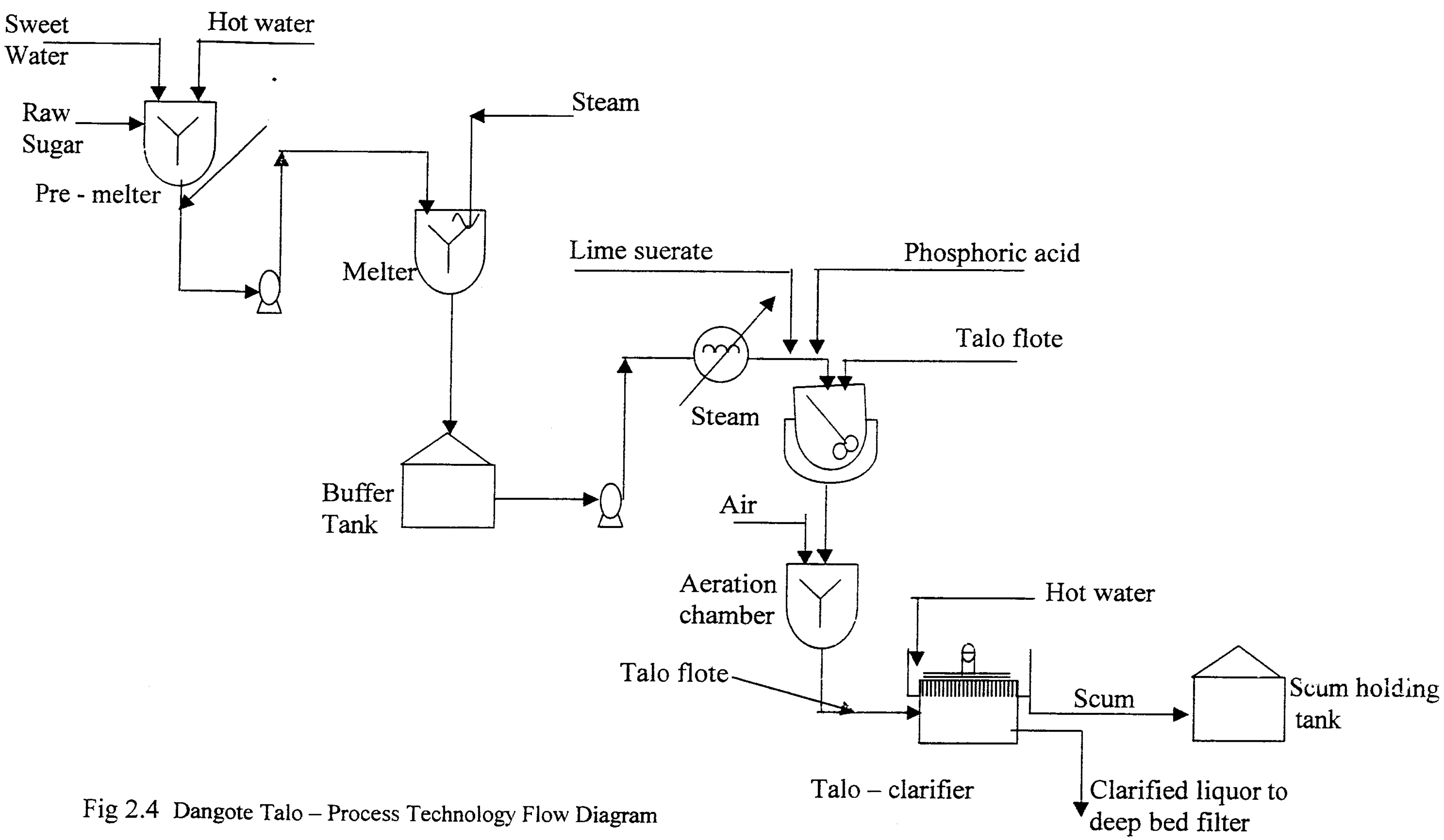

Fig 2.4 Dangote Talo – Process Technology Flow Diagram

CHAPTER THREE

3.0 EXPERIMENTAL WORK

The effectiveness of this process is determined by investigating the working conditions of the process, and examining the operating parameters of the clarification process does this. Since, the parameters involved are also used to determine the colour removal and clarification efficiency, they should be carefully investigated. The parameters are listed below:

(i) Brix

(ii) Temperature

(iii) Chemical consumption

(iv) pH

(v) Colour

(vi) Turbidity

Also the analysis of scum is done to ascertain the implication of the following parameters on the environment:

(i) Moisture content

(ii) Sucrose content

(iii) pH

(iv) Heavy metals analysis

(a) Lead

(b) Copper

(c) Iron

3.1 Materials Required

3.1.1 Materials and Equipment for Liquor Analysis

(A) Brix

Equipment

- i. Stirrer
- ii. Spatula
- iii. Beaker
- iv. Refractometer (Brixometer)
- v. Cotton wool

Materials

- i. Distilled water
- ii Liquor (pre-melt, melt and clarified)

(B) Temperature

Equipment

- i. Beaker
- ii. Stirrer
- iii. Thermometer

Materials

- i. Distilled water
- ii. Liquor (melt, and clarified)

(C) Chemical Consumptions

Equipment

- i. Chemical dosing indicators at the Talo-process panel
- ii. Chemical dosing chart
- iii. Reaction tank

Materials

i. Reaction tank liquor

(D) pH

Equipment

i. Beaker

ii. Stirrer

iii. pH meter

Materials

i. Liquor (pre-melt, melt, clarified)

ii. Distilled water.

(E) Colour And Turbidity

Equipment

i. Beaker
ii. Electronic weighing balance
iii. Spatula
iv. Stirrer
v. Refractometer (brixometer)
vi. Vacuum pump membrane
vii. Cellulose filter paper
viii. Conical flask
ix. Cotton wool
x. Dilute brix factor table

Materials

i. Distilled water
ii. Liquor (pre-melt, melt and clarified)

3.1.2 Scum Analysis

(A) Moisture Analysis

Equipment

i. Moisture Analysis
ii. Electronic weighing balance
iii. Spatula
iv. Petri dish

Materials

i. Scum sample

(B) Sucrose (Pol) Content

Equipment

i. Beaker
ii. Electronic weighing balance
iii. Kolrhaush flask (200ml)
iv. Funnel
v. Stirrer
vi. Spatula
vii. What man # 91 filter paper
viii. Propol polarimeter

Materials

i. Scum sample
ii. Distilled water
iii. Filter aid (celite)

3.1.3 Heavy Metals Analysis in Scum and Refined Sugar

(A) Ash Content

Equipment

i. Petri dish
ii. Spatula
iii. Hot plate
iv. Electronic weighing balance

Materials

i. Scum and refined sugar samples

(B) Digestion of Ash Content

Equipment

i. Conical flask

ii. Beaker

iii. Stirrer

iv. Measuring cylinder

v. Hot plate

vi. What man # 91 filter paper

vii. Funnel

Materials

i. Ashed scum and refined sugar samples

ii. Conc. HCl analaR grades

iii. Conc. HNO_3

iv. Distilled water

(C) Heavy Metal Presence

Equipment

i. 50ml beaker

ii. Measuring cylinder, $100cm_3$

iii. 10ml pipette

iv. Iron, copper and lead hollow – cathode lamp

v. Atomic Absorption Spectrophotometer (AAS) (filter with air / acetylene burner).

Materials

i. Digested refine and scum filtrate samples

ii. Pure iron, copper and lead samples

iii. Distilled water

(D) pH

Equipment

- i. Beaker
- ii. Electronic weighing balance
- iii. Measuring cylindering
- iv. Spatula
- v. Stirrer
- vi. pH meter

Materials

- i. Scum sample
- ii. Distilled water

3.2 Experimental Methodology

3.2.1 Liquor Analysis Procedures

Twelve days were taken to investigate the performance of the operating parameters of the process in the refinery. The time interval of 2 hours was adopted for the investigation and an average parameter performance was taken after 8 hours each day.

i. Brix Analysis Procedure

Samples of liquor from pre -melter, melter and clarifier were poured into different beakers and thoroughly stirred. Small portion of each sample were put on the refractometer lens and their readings taken.

ii. Temperature Analysis

Samples of liquor were taken from melter and clarifier put into different beakers and stirred. And immediately thermometer was inserted into the sample to take the temperature readings.

iii. Chemical Consumption Analysis

Chemicals consumed were obtained from the chemical dosing indicator at the talo-process panel in litre per hour. And the corresponding values in parts per million (ppm) were obtained from chemical dosing charts.

iv. pH Analysis

Samples of liquor were taken from pre melter; melter and clarifier put into different beakers and each sample was stirred. The pH reading was taken using pH meter.

v. Liquor Colour Determination

30g of sample of liquor was taken from pre melter and melter, and 50g from clarifier respectively. Then, the brix of each sample was measured with refractometers. The weight of each sample was made –up to the value of its measured brix using distilled water. The diluted brix samples were obtained before the diluted brix values were filtered using vacuum pump with membrane cellulose filter attached. The filtrate absorbance was measured in a talameter at 420nm. The colour of the sample was determined with the equation below:

Colour = Absorbance / (Cell size x diluted brix factor)

vi. Turbidity Determination

The same procedure was used as in colour determination above (3.2.1.5); except that the absorbance of the diluted brix samples were measured before and after filtration with talameters at 420nm. The turbidity of each sample was determined as below:

Turbidity:
$$\frac{A_1 - A_2}{(\text{Cell size x diluted brix factor})}$$

A_1 – absorbance before filtration

A_2 – absorbance after filtration.

3.2.2 Scum Analysis Procedures

In this research work, 3 samples of scum were taken in an interval of 4 day to determine the concentration of heavy metals, its moisture content, pH and sucrose content (pol).

i. Pol Determination

50g of scum was weighed in a 100ml of beaker and dissolved with distilled water. All measured content was transferred into a 200ml of kolrhausk flask. The sample was made up to its 200ml marks, with distilled water and thoroughly shaken. The sample was filtered with the aid of filter aid (celite), while the filtrate pol was determined using propol polarimeter at 26 setting.

ii. Moisture Content Determination

20g of scum in a petri dish were weighed on a weighing balance. The sample was well spread on the dish for uniform heating; then, put in analysed until no weight difference existed on the sample in the analyzer. The moisture content value on the digital display was recorded.

iii. pH Determination

10g of scum sample was weighed and 100ml of distilled water was added to the sample, and the mixture thoroughly stirrer then, pH value of the mixture was taken using pH meter.

3.2.3 Heavy Metals Analysis Using Atomic Absorption Spectrophotometer (AAS)

a) Scum Sample Analysis

i. Ash Content Analysis

3g of scum samples were weighed in 3 different Petri dishes and heated for 6 hours at 700^0c.and the samnles were cooled and weighed. The percentage ash content was determined with the equation below:

$$\% \text{ ash content} = \frac{\text{Sample weight after cooling x } 100\%}{\text{Initial sample weight.}}$$

ii. Digestion of Ash Sample

2ml of conc. HCl and 3ml of conc. HNO_3 were added to 2 different ash samples, and they were heated on a hot plate for a while, before small quantity of distilled water was added. Then, the sample was further heated to reduce the volume of the solution and filtered. The filtrate was diluted to 25ml in standard flask using distilled water.

iii. Atomic Absorption Spectrophotometer (AAS) Analysis.

4 standard solutions of 2 ppm, 4ppm, 6ppm, 8ppm and 10ppm of lead (pb), iron (fe) and copper (cu) were prepared respectively. Then, the atomic absorption spectrophotometer was adjusted and the hollow – cathode lamp emission line selected at 248.3nm, 217.0nm and 324.8nm for iron, lead and copper respectively. Distilled water was aspirated into the flame and the absorbance scale was zeroed. Each sample of scum solution and standard solution were aspirated, rinsed with distilled water between each one and absorbance reading in each case was recorded. Aspirating with distilled water again checked the zero readings and duplicate readings were obtained from the standard and sample solutions. Standard curves of absorbance verse the metal concentration was plotted to obtain a straight line. And the metal content of the original scum samples was calculated and expressed as mg/kg.

b) Refined Sugar Sample Analysis

Composite sample of refined sugar for the period of twelve days of investigation was used. Sample of 3g of refined sugar was made from the composite sample. However, other procedures followed during the analysis were the same with that of the scum sample described in section 3.2.3.1.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION OF RESULTS

4.1 Liquor Analysis Results

Table 4.1.1 Liquor Analysis Result

		Day 1	Day 2	Day 3	Day 4
Raw Sugar Melted (Tons)		2.747	2.766	2.755	2050
Pre-Melt Liquor					
pH		7.6	6.9	7.3	7 4
Brix		67.4	67.6	67.5	66
Melt Liquor					
pH		7.2	7.0	6.7	7.1
Brix		65.5	66.1	65.5	65.5
Colour (Icumsa Unit)		493	407	477	495
Turbidity		803	667	780	809
Temperature (^{0}c)		65	64.5	65	64
Clarified Liquor					
pH		7.2	7.3	7.1	7.2
Brix (Conc.)		64.5	65.0	64.2	64.5
Colour (Icumsa Unit)		263	223	254	28[illegible]
Turbidity		56	52	55	60
Temperature (^{0}c)		81	82	83	81
% Colour Removal		46.7	45.2	46.8	42.2
Chemical Consumption (ppm)					
	LIMIT				
Hydrated Lime	< 500 ppm	326	383	402	426
Phosphoric Acid	< 420 ppm	293	365	279	349
Talo Floc	< 300 ppm	237	225	236	245
Talo Flote	< 13 ppm	12	12	14	12
Scum Made In Tons (Estimated)		1.3	1.4	1.4	1.2

	Day 5	Day 6	Day 7	Day 8
Raw Sugar Melted (Tons)	1883	2095	2,493	2,136
Pre – Melt Liquor				
pH	7.1	7.5	7.9	7.6
Brix	66.6	67.3	66.5	67.1
Melt Liquor				
pH	6.7	6.9	7.5	7.4
Brix	65.1	64.8	64.5	65.4
Turbidity	879	812	820	948
Temperature (^{0}c)	64.5	65	64.5	65
Clarified Liquor				
pH	7.1	7.2	7.2	7.2
Brix	64.0	64.4	63.1	63.4
Colour (Icumsa Unit)	287	337	250	301
Turbidity	61	66	56	60
Temperature (^{0}c)	81.5	83	82	81.5
% Colour Removal	468	32.3	50.4	48.2
Chemicals Consumption (ppm)				
Hydrated Lime	489	334	494	420
Phosphoric Acid	335	214	387	331
Talo Floc	261	180	243	259
Talo Flote	13	11	16	16
Scum Made In Tons (Estimated)	1.2	1.3	1.3	1.3

	Day 9	Day 10	Day 11	Day 12
Raw Sugar Melted (Tons)	1,927	2,121	2,538	2,396
Pre – Melt Liquor				
pH	7.9	6.8	7.4	7.2
Brix	67.0	66.8	66.7	67.1
Melt Liquor				
pH	7.5	7.2	7.2	7.3
Brix	65.6	64.7	64.8	64.6
Turbidity	571	529	480	48.8
Temperature (^{0}c)	921	836	782	790
Clarified Liquor				
pH	7.1	7.0	7.1	7.1
Brix	64.1	64.2	63.4	63.3
Colour (Icumsa Unit)	295	385	245	258
Turbidity	62	92	54	58
Temperature (^{0}c)	82.5	81	82	82
% Colour Removal	48.3	27.2	49.0	47.1
Chemicals Consumption (ppm)				
Hydrated Lime	443	368	433	437
Phosphoric Acid	389	204	304	390
Talo Floc	256	181	265	244
Talo Flote	15	12	14	14
Scum Made In Tons (Estimated)	1.2	1.3	1.4	1.2

Table 4.1.2 Average Liquor Analysis Result per Day

Raw Sugar Melted (Tons)	2326
Pre-Melt Liquor	
pH	7.4
Brix	67.0
Melt Liquor	
pH	7.1
Colour (Icumsa Unit)	505
Turbidity	821
Temperature (^{0}c)	64.8
Clarified Liquor	
pH	7.2
Brix	64.1
Colour (Icumsa Units)	280
Turbidity	61
Temperature (^{0} C)	81.9
% Colour Removal	44.6 %
Chemical Consumption (ppm)	
Hydrated Lime	399
Phosphoric Acid	317
Talo Floc	234
Talo Flote	12
Scum Made (Tons Estimated)	1.3
Scum Made (Per Kg Of Raw Sugar Melted)	0.559 (g/kg)

4.2 Scum Analysis and Refined Sugar Results

4.2.1 Scum Analysis Results

Table 4.2.1 Scum Analysis Results

Test Performed	Result:
Appearance	Dark Brown Semi Solid
Odour:	Characteristics of Sand

	1st Sample	2nd Sample	3rd Sample
pH (10%)	7.6	7.3	7.5
Moisture (% W/W)	76.40	79.80	80.60
Sucrose (%)	0.26	0.25	0.21
Ash (%)	11.60	9.48	10.40
Lead (Mg/Kg)	11.00	40.97	37.62
Copper (Mg/Kg)	16.00	5.83	8.24
Iron (Mg/Kg)	1700	2400	2230

Average heavy metals in scum (mg/kg)

Lead – 28.86

Copper – 10.02

Iron – 2110

Table 4.2.2 Guideline For Interim Uniform Effluent Limit In Nigeria For All Categories Of Industries (mg/kg).

Discharged To Surface Water	Land Application
Iron (Fe) <20	-
Copper (Cu) < 1	-
Lead (Pb) < 1	-

(FEPA, 1991)

4.2.2 Refined Sugar Analysis

Table 4.2.3 Results Of Refined Sugar Analysis

Test Performed	**Result**	
Appearance	White crystalline sugary	
Taste	Sweet, characteristic of sugar	
Odour	Characteristic of sugar	
		Limit
Iron (mg/kg)	0.89	-
Copper (mg/kg)	Not detected	2.0 (max)
Lead (mg/kg)	Not detected	0.5 (max)

4.3 Discussion Of Results

4.3.1 Liquor Analysis

From the result obtained in the table 4.1.1; the investigation showed that the operating parameters listed below were not influenced by the varying colour of the raw sugar.

(1) Brix

(2) Temperature

(3) pH

These parameters were independent of the colour of the raw sugar, at every stage of the process. And this showed that for the process to be efficient and effective the range of these parameters as established in the literature must be maintained.

However, rate of chemical dosage in the process was observed to be dependent on the magnitude of colour of the raw sugar. As the colour of the raw sugar increased, the rate of dosage increased as well. The results in table 4.1.1 justified the fact above as indicated on day 1 and day8. As shown in table 4.3.1 below:

Table 4.3.1.Liquor analysis result of day 1 and day 8

	Day 1	Day 8
Melt Liquor Colour (icumsa)	493	581
Chemical Dosage (ppm)		
Hydrated Lime	326	420
Phosphoric Acid	292	331
Talo Floc	237	259
Talo Flote	12	16
% Colour Removal	46.7%	48.2%

In table 4.1.1 above, when the rate of chemical dosage was low, the colour removal efficiency of the process was very poor. As shown in day 5 and day 10 in table 4.1.1, where % colour removal were 32.5% and 27.2% respectively. This was due to low chemical dosage that led to improper reaction in the reaction and bad clarified liquor quality at the clarifier.

When the rate of chemical dosage of the process were high, chemicals were wasted because the large quantity of chemical dosed did not result to higher % colour removal, as shown in table 4.3.2 below:

Table 4.3.2 Liquor analysis result of day 1 and day 4

	Day 1	Day 4
Melt Liquor Colour (icumsa)	493	495
Chemical Dosage (ppm)		
Hydrated Lime	326	426
Phosphoric Acid	292	349
Talo Floc	237	245
Talo Flote	12	12
% Colour Removal	46.7%	42.2%

- In the table 4.3.2 above, though, more quality of chemicals were dosed to melt liquor in day 4 with colour of 495 icumsa, compared with liquor in day 1 with colour of 493 icumsa, the % colour removal of day 1 liquor was higher than that of day 4, so chemicals were wasted in day 4.

However, the overall talo – process technology had high efficiency of colour removal as shown in table 4.1.2

Melter liquor colour / day – 505 icumsa

Clarified Liquor Colour / Day - 280 icumsa

% Colour removal / day - 44.6%

The colour removal efficiency of this process (44.6%) was higher than that of the other conventional clarification process (25%). This showed that the process was highly efficient. Hence, it yielded sugar liquor of low colour (280 icumsa) and sparkling appearance.

In table 4.1.2, waste produced/kg of raw sugar melted was 0.559 g/kg, thus, the amount of waste generated in the process was very small compared to the quantity of raw materials processed (2326 tons of raw sugar / day). And this reduced the problem of pollution the process would have created to the environment.

Finally, the rate of chemical dosages / day that yielded % colour removal of 44.6% were economical and within the chemical consumption limit of the process, as shown in tables 4.1.1 and 4.1.2

4.3.2 Scum Analysis

In table 4.2.1 the sucrose content in the scum was low (0.21 – 0.26%) which showed that the clarification process was efficient in that small amount of sucrose was lost in the process.

The investigation also reviewed the presence of toxic heavy metals in high proportion in the scum generated. Though, such toxic metals (iron, lead and copper), where not directly added during different stages of the process, but the impurities indicated were a product of accumulation of these elements during industrial processing and the origin of the raw sugar. (Brekham and Nesterenko, 1982). And there might be traces of copper and lead in refined sugar but their quantities less than 1mg/kg respectively (Brekham and Nesterenko, 1982).

Scum generated had average content of iron (fe) – 2110 mg/kg, copper (cu) 10.02mg/kg and lead (pb) – 29.86mg/kg; was discarded into the lagoon untreated and the values of these heavy metals in the scum were higher than effluent limit for industries to be discharged into water bodies as shown in table 4.22.

4.3.3 Refined Sugar Analysis

In table 4.2.3, no trace of copper and lead was found in the refined sugar as the result reviewed. But, the refined sugar contained 0.89mg/kg of iron. This result was satisfactory; thus, the refined sugar obtained from talo process technology was good for human consumption and other industrial purposes.

4.4 Research Findings

i. Chemical used in this process are costly and cannot be regenerated after use. Also, they are not sourced locally and need to be imported. And so it the rate of chemical dosage is not efficiently controlled, the cost of sugar refining using this process will be high.

ii. In this process, a decolourisation rate of 45% can be achieved compared to 25% in conventional clarification process (Kenana Sugar, 2000).

iii. This process allows installation of smaller decolourisation station of lesser capital investment for equipment and produces a small amount of scum (0.559g/kg); and, thus reduces pollution problems.

iv. Small size clarifiers (retention time 30 minutes) used is optimum and has the requirements for filtration and decolourisation processes that resulted in less sugar loss (0.21 – 0.26%).

v. The level of heavy metals (iron – 2110mg/kg, copper – 10.02mg/kg and lead -29.86mg/kg) in the effluent of the process is high enough to be treated before being discharged to the environment.

vi. Trace of toxic heavy metals (Copper and lead) is not found in the refined sugar produced with this technological process.

CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The colour removal and clarification efficiency of talo- process technology was 45% compared to 25% of other conventional clarification process. Also, the sugar loss in this process was very low (0.21 – 0.26%). The amount of scum generated per kg of raw sugar melted was 0.559 (g/kg) and this would allow installation of small decolourisation station of less capital investment for equipment.

Chemicals used in the refining process could not be regenerated after use. And the amount of heavy metals (iron – 2110 mg/kg, copper -10.02mg/kg and lead – 29.86mg/kg) in the by – product (Scum) being discharged untreated into the environment was too high. However, only 0.89mg/kg of iron was detected in the refined sugar, the other heavy metals were not detected.

5.2 Recommendations

i. Talo – process Technology of Sugar refining process should be used in raw sugar refining in Nigeria. This is due to its high colour removal and clarification efficiency, its low sugar loss and its reduced pollution problem because small amount of scum (0.559 g/kg) was generated.

ii. To reduce the cost of refining, the rate of chemical dosage in the process should be optimized.

iii. The amount of heavy metals (iron, copper and lead) in the by – product (scum), which is the effluents of this process is too high; and should be treated to reduce them to their threshold limit values (TLV), acceptable in the country before being discharged into the environment.

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APPENDIX

Amount of Waste Product Generated

Average waste produced / raw sugar melted (g/kg)

1 ton = 1016.0469/kg

Average of 1.3 tons of scum was made / day =

1.3 x 1016.04691 = 1320.860983kg

1 kg – 1000g

Scum made in g/day = 1320.860983 x 1000

= 1320860.983g

Average Raw sugar melted per day (tons) = 2326 tons

Average raw sugar melted / day (kg) = 2363325.113kg

$$\text{Average waste produced/day (g/kg)} = \frac{\text{waste produced in grams}}{\text{Raw sugar refined in kg}}$$

$$= \frac{1320860.983\text{g}}{2363325.113\text{ kg}} = 0.559\ (\text{g/kg})$$

0.559 g of scum was produced in 1 kg of raw sugar refined.

GLOSSARY

Affined sugar	spun sucrose from the affination centrifuges.
Affination	process by which syrup is removed from and around raw sucrose crystals
Alfa – lava	a decanter centrifuge separator used to separate water and scum.
Ash	mineral impurities in sugar.
Brix (BX)	percentage by weight of soluble materials in a sugar solution.
Clarification	the process by which solids are separated from the liquid phase and clarity of liquid phase is of prime concern.
Colour	complex molecules of impurity.
Desweetening	the removal of sucrose from waste product by ashing washing with water.
Floc	very fine particles formed during raw sugar clarification process.
Icumsa	International Commission for Uniform Measurement of Sugar Analysis.
Invest sugar	Mixture of glucose and fructose.
Liquor	solution of sucrose before crystallization.
Melting	dissolution of sucrose in water.
Phosflotation	a process of raw sugar clarification that combines phosphatation and flotation mechanisms.

Pol content	the amount of sucrose in a substance expressed in percentage.
Raw sugar	a product of raw sugar clarification that have between 96 – 99.5% sucrose content.
Scum	final waste product generated in raw sugar clarification process.