REFINING OF LOCALLY PRODUCED BROWN SUGAR
(MARZAKWAILA)

BY

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CERTIFICATION

This is to certify that this work was done by Mr. D.S. Akinrinde (92/2751), and duly supervised and approved by me in partial fulfilment of the award of a Bachelor's Degree in Chemical Engineering.

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EXTERNAL EXAMINER (DATE)

DECLARATION

I hereby declare that this project work is my original work and has never to my knowledge been submitted elsewhere before.

....

AKINRINDE D.S.   (DATE)

DEDICATION

To the living memory of my late mother, Mrs Risikat Akinrinde.

ACKNOWLEDGEMENT

The horse is made ready for the day of battle, but victory rests with the Lord (Prov 21:31). I thank the gracious Lord for granting me success in my academic pursuits, and for being my Alpha and Omega always

I am sincerely grateful to my father, Mr. U.O. Akinrinde for his support. My thanks also go to my maternal grandmother, Madam Sabitiyu Okikiola for her love and support. I love you, Mama. This report will be incomplete without acknowledging the support received from my supervisor, Dr. K.H. Onifade in ensuring the successful completion of this project and for being a shining example of discipline and diligency to his students. I am also grateful to my sweetheart, Miss. R.M. Okunola, and Mr. A.O. Ajayi for their valuable advice and encouragement in the course of carrying out this work. My utmost thanks is also extended to Mr. Abdullahi A. Tajudeen for typing and printing this report

Too long.

ABSTRACT

The focus of this project work was the refining of locally produced brown sugar, mazarkwaila, through the removal of impurities from the brown sugar using different types of clarifying agents and activated carbon as the sole colour adsorbent.

From experimental results, it was confirmed that the higher the amount of calcium-phosphate precipitate removed after clarification, the higher the yield and the better the quality (colour tone and crystallinity) of the product obtained. However this fact depends on the mode of crystallization employed. Deductions made from results show that seeding mode gives better result than forced mode (see Table 4.4.1 - 4.4.7) of crystallization.

During evaporation process, it was discovered that boiling of the liquor at normal boiling point require some precautions such as continuous stirring, stopping boiling when super saturation is almost reached, and boiling in pan of large surface area instead of evaporating dish.

The highest yield was obtained in experiment 2B which as expected also gave the largest amount of mud removed (calcium-phosphate-impurities). Of all the clarifying agents used, (i.e. phosphoric acid, sodium hydrogen phosphate, sodium orthophosphate) phosphoric acid was found to be most suitable as it gives the highest average yield (28.48g) and the whitest product. It is followed by sodium orthophosphate with average yield of 27.35g and fairly white colour. However the results obtained when sodium hydrogen phosphate was used is discouraging especially in colour tone as no significant change in colour was noticed due to

decomposition of sugar during boiling

The average percentage yield for each clarifying agents phosphoric acid, sodium hydrogen phosphate, sodium orthophosphate are 56.98%, 50.35% and 54.71% respectively. Similarly the percentage purity obtained for each case are 57.55%, 48.12% and 53.74% respectively. Experimental results also show that the refractive index of the sugar has been improved by 5% (increased by 0.08), and the sugar confirmed to be dextrorotatory(+).

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CHAPTER ONE

INTRODUCTION

1.1 PROJECT AIMS AND SCOPE

In many countries of the developing world, go [illegible] exist to promote a variety of development objectives through research to improve products of local technology. These might be to promote self sufficiency in food production and to improve on the quality of the products to meet customers expectation among many other reasons.

This project is aimed at enhancing the quality of locally produced brown sugar called "Marzarkwaila" through the removal of soluble and insoluble impurities. This local product is very poor in quality due to the enormity of the impurities present in it. It is believed that if this product is refined and given a new identity, apart from enhancing its usage as food, it will also compliments sugar supply from mechanised sugar industries as feedstock to chemical industries. The demand for sugar as feedstock has continued to rise over the years. The recent development of "gasohol", a motor fuel (mixture of gasoline and ethanol) which is currently use in Kenya and Zimbabwe is expected to spread to other African countries including Nigeria. Hence the need to source for new areas to compliment sugar supply from large scale industries.

As a first step to understanding the source of the impurities in the local sugar, detail study of the local manufacturing process was carried out. This revealed the degree of impurity of the product on which the refining process was based upon ordinary sugar refining process. The operation responsible for the removal of impurities is called clarification or defecation. To a great extent this operation determines the level of purity of the final product. Hence for this project, much emphasis shall be placed on the clarification process. The basic reagent used for clarification is lime. Clarifying reagents put to use for the refining of this product are all phosphate based. Due to the dark colour of the brown sugar, it is expected that it must have undergone decomposition, due to continuous heating. Hence little heat should be used during refining to avoid further decomposition and phosphate treatment allows this. The reagents used are

1. Phosphoric acid
2. Sodium hydrogen phosphate
3. Sodium orthophosphate.

To avoid decomposition of the sugar, temperature is kept at 70°C for all operations except for evaporation and crystallization. For the decolorization of the brown sugar, powdered activated carbon is used. This is preferred because of the large surface area it possesses. The two modes of crystallization were tested i.e. seeding and forced mode, to determine the most suitable for the refining process. Evaporating dish which is normally used for boiling the sugar syrup in the laboratory has a very small surface area which might increase heating period, hence it will be replaced by an open aluminium pan which has a very high heat conductivity and at the same time high surface area. As a precaution, to prevent heat accumulation during boiling, continuous stirring is ensured so that the heat shall be evenly distributed throughout the boiling process. The pH of the sugar solution is considered very essential the bid to obtain a pure product. It is known that high acidity of sugar solution enhances the decomposition of sucrose to non recoverable monosaccharides: glucose and fructose. To curtail this, the pH of the sugar solution is constantly monitored before heating during clarification process.

By carrying out the refining process through the conditions stated above, it is expected that a well refined product which will not only be suitable for human consumption but qualifies also as feedstock to chemical industries will be obtained. This is the aim of this project, the result of which will help local producers of brown sugar to improve the quality of their products. The local demand for refined sugar in Nigeria is shown below in fig 1.1

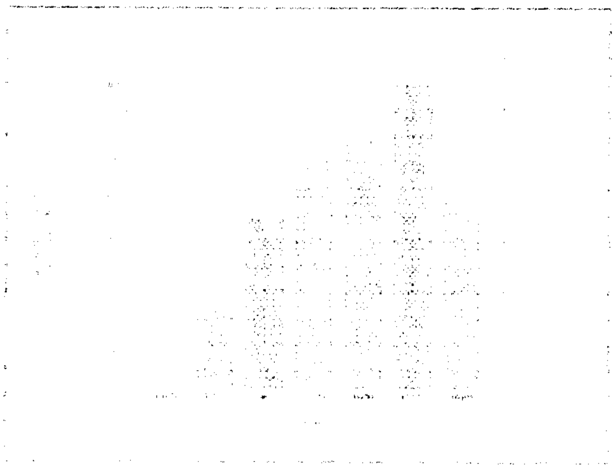

Fig 1.1 Local demand for refined sugar in Nigeria

1.2 ECONOMIC IMPORTANCE OF CANE SUGAR

1.2.1 CANE SUGAR AS FOOD ADDITIVE

Food additives are substances added to foods during processing to retain or improve desirable characteristics or quality. Cane sugar is one of the major food additives used in the food industry. Food additives may be added as a sweetener, thickener, emulsifier and as an [illegible] agents.

Many modern food processing plants controlled spoilage by adding some quality of sugar. High concentration of sugars helps preserve fruit jams and jellies, as well as canned and frozen fruits, against microbial spoilage. Common table salt serve similar role in the preservation of meat and fish. Sugar is also used domestically to add taste. Sugar is added to beverages and garri to add taste. Bakery products are hardly complete without the addition of sugar during its processing.

Although sugar is not the only major sweetener known to man, it is about the widely used in the world due to its availability. Sugar is however not good for use where a low calorie food is being manufactured, it has to give way to synthetic sweeteners such as saccharin, cyclamates etc.

As a preservative, sugar inhibits microbial growth by reducing the water activity of food and raise the osmotic pressure of the solution and thus cause plasmolysis of microbial cells.

1.2.2 NON CARBOHYDRATE SWEETENERS

With the commercial introduction of aspartame in 1974 by G.D. Searle[2], the term non nutritive sweetener no longer is descriptive. Aspartame is a nutritive sweetener equally as calorific as sugar on a gram for gram basis but possessing intense sweetness when used at low concentrations in foods.

Saccharin[3] presently is the only non carbohydrate sweetener being used commercially in foods as the sole sweetener. Aspartame commercialisation died down due to delayed approval. Artificial non nutritive sweeteners began one day in 1879 when a young German Chemist, [illegible]

Fahlberg[1] was working in the laboratory of Johns Hopkins University. He discovered by accident the sweetness of a compound they had synthesised known as saccharin [illegible] Fahlberg ate a piece of bread without washing his hands and traced the unknown sweetness to a chemical o-sulfobenzimide.

FIG 1.2

Saccharin (1): Glucide

Molecular Weight [illegible]

Aspartame was discovered in 1965 in the laboratory of G.D. Searle & Company while scientists there were hunting for new drugs to treat ulcers[1]. Aspartame is the generic name for a compound of the protein constituents L-aspartic acid and L-phenylalanine methyl ester from which the sweetener is synthesized.

Other sweeteners include Acesulfam being developed by Hoechst, a German Pharmaceutical firm. The product is derived from acetic acid.

1.2.3 SYNTHETIC SWEETENERS AND CANE SUGAR : A COMPARISON

In recent years, cane sugar have had to face competition with synthetic sweeteners. The first of these was saccharin, which is about 300 times as sweet as sucrose. This perceived sweetness is dependent to a large extent on formulation, temperature of the food when served and the like. Unlike cane sugar, solutions of saccharin are acid to litmus paper.

in the use of sucrose as feedstock

Although adjustments have to be made to the carburettor system of cars, there are major cost savings. Gasohol was developed in Brazil, but it is used also in Kenya and in Zimbabwe. Other countries in Africa could follow their example. It has been suggested that there will be a change in emphasis in the use of sugar, molasses and bagasse. Bagasse is the fibrous residue left after the sugar has been extracted from the cane. At present sugar is used for food, industrial chemistry, fermentation and fuel, in that order. In the next 20 years or so it is likely that the order will become fuel, fermentation, industrial chemistry and food.

CHAPTER TWO

2.1 SUGARCANE : ORIGIN, HUSBANDRY AND PHYSIOLOGY

2.1.1 ORIGIN:

Sugarcane (Saccharum Officinarum L.), a member of the grass family originated in New Guinea about 8000 - 15,000 B.C. and was later moved by primitive man westward into southeast Asia and India and eastward into polynesia(1). The original sugarcane, so called "noble" canes, have 2n = 80 chromosomes, although there are some exceptions. S. officinarum hybridized with wild species especially S. spontaneum L, in Asia, giving rise to new types that were variable in morphological and ecological tolerance and that extended the distribution of sugarcane into the subtropics. Two additional species are recognised in botanical literature.

Most current commercial varieties are interspecific hybrids involving primarily two or more of the following species: S. officinarum, S. robustum and S. spontaneum. Extensive breeding programmes in the tropics where sugarcane flowers under natural conditions and in the subtropics in climate-controlled structures, sponsored by public and private research agencies provide a continuous supply of new varieties adapted to major sugarcane producing areas of the world.

Sugarcane is one of the crops which serve as a source of sucrose (sugar of commerce). Sugarbeet is another sucrose source. In Nigeria, sugarcane is the major source of raw material for sugar production. The primary sugar, glucose is a product of photosynthesis and occurs in all green plant, and through chemical union, diverse sugars and starches are elaborated and become the major reserve food in storage organs, fruits, and sap of plants. Other sugar crops are sweet sorghum, sugar maple, sugar palm, honey, and corn sugar.

2.1.2 STRUCTURE OF SUGARCANE

Every new variety of sugarcane begins as a single shoot (stalk) that develops from a seed. The cylindrical stalk is divided into nodes and internodes with lateral bud at each mode. The lateral buds occur alternately on opposite sides of the stalk, and there is an apical bud A root band at each node includes several rows of root primordia. The internodes are covered with wax and are filled with parenchyma or storage cells (pith) and the vascular bundles. The leaf consists of a sheath attached to the stalk at the node, an auricle, a ligule, a dewlap and a blade which tapers gradually from the base to the tip and is supported by a midribs extending almost its entire length. Edges of leaves of most varieties are serrate. The inflorescence is a silky panicle at the apex of the stem bearing many small spikelets which are arranged in pairs on the branches.

2.1.3 SUGARCANE PHYSIOLOGY : SUCROSE BIOSYNTHESIS

Sucrose is formed as a result of photosynthetic carbon assimilation in virtually all higher plants. Its importance as the most abundant sugar in plants and as the world's principal sugar is exceeded only by its physiological status as a reserve constituent in cell respiration. Energy liberated via respiratory processes is utilized for numerous metabolic activities. Of these the synthesis of amino acids, proteins, fats, and other organic compounds rank among the most important. Yet the manner by which sucrose itself is synthesised has long eluded plant scientists. In recent times, the discovery that sucrose is the principal form of transient carbohydrate, plus the fact that it is not the form most readily accumulated in cellular storage compartments, has shown that a sucrose molecule might be synthesised, inverted and resynthesised several times before fulfilling its physiological destiny. On purely technical grounds, the synthesis of sucrose is such a complicated matter that enormous strides in the field of plant physiology were required before the natural mechanisms could be seen in anything like a correct perspective.

It has been noted that for many years physiologists were uncertain as to whether a simple sugar, the disaccharide sucrose, or starch was the primary product of photosynthesis. This seemingly insurmountable problem lend to many researches carried out by great plant scientists. The discovery of uridine diphosphate glucose (UDPG) in 1949 by Leloir and his co-workers in the sugarcane storage tissue led to a good understanding of the synthesis of sucrose in sugarcane. UDPG was recognised as an active metabolite of photosynthesising green leaves, and at various times has being used in the enzymatic synthesis of sucrose and sucrose phosphate. Researchers have also discovered other enzymes involved in the synthesis of sucrose.

Hatch et. al. reported in 1962 that enzymes having direct and indirect bearing on sucrose synthesis had been found in storage tissue preparation from S. officinarum and S. spontaneum varieties. Many of the essential enzymes were identified, among these are sucrose synthetase, UDPG pyrophosphorylase etc.

2.1.4 QUALITY AND PROCESSING OF SUGARCANE

Sugar producing factories are quite aware of the importance of the sugarcane quality used in their production. Scientists have carried out many researches to determine the best species of cane for the production of sugar. Such qualities of the specie could be:

- resistance to diseases.
- low fibre content.
- sucrose content
- rind tissue softness etc

Plant scientists employed the generic make up of the species to determine the characters that are favourable. Such methods like cross breeding, intergeneric crossing, and controlled crossing were used to obtain good seedlings for sugarcane production. Sugarcane rich in sucrose and that possesses low fibre content are desirable for the production of sugarcane.

The processing of sugarcane starts with the removal of the juice from the cane by mechanical pressure. The top 4.7cm of the cane stalk is removed because it contains waxes and resins which make clarification of the sugar in the plant difficult. Hand harvesting is still practiced in Nigeria. The pressed cane is sprayed with hot water and run through another set of rollers to extract additional juice. The remaining fibre cane materials, known as bagasse, is removed at about 30% wet basis, moisture content. The bagasse can be used for fuel for the plant operations, structural materials and paper. It has a heating value of about 19,800 KJ/Kg on a dry weight basis.

The cane juice is strained, then treated chemically and moved through the process for the manufacture of sugar.

2.3 PESTS AND DISEASES OF SUGARCANE

Sugarcane is subject to many diseases of which few of them are causing serious losses in susceptible varieties. Diseases become of particular importance in sugarcane because of

1. the use of the stalk (vegetative part) for commercial plantings, a practice that spreads disease into new plantings, if the propagative stalks are diseased.
2. the relatively small number of varieties grown in a country, which exposes large areas to a disease if the dominant variety is susceptible; and
3. the production of several crops from one original planting, which may result in the accumulation of disease in a field.

The important diseases of sugarcane are caused by viruses, bacteria and fungi. Viruses cause mosaic and Fiji disease. Ratoon stunting disease, gummosis and leaf scald are caused by bacteria. Red rot, smut, downy mildew and root rot are caused by fungi. Except for root rot, all diseases are carried in the seed piece (stem node), and are spread in the field by insect vectors or mechanical means such as harvesting or rain.

Resistant or tolerant varieties provide the most satisfactory means for controlling diseases. At present ratoon stunting disease is controlled by immersing cane in hot water at 50°C for 2 hours before planting or by hot air treatment at 58-59°C for 8 hours.

The Nigerian Sugar Company at Bacita, where sugarcane is been grown for several years on a large scale has been undergoing series of pest and nematode attacks ranging from stem borers, and [illegible] of recent termites attack.

Attempt to solve this problem using pesticide such as [illegible] aldrin and gamma BHC have so far failed to achieve real [illegible] success.

2.1.6 SUGAR TRADE AND PRICE POLICY: A NIGERIAN PERSPECTIVE

Trade in agricultural products constitutes a very large proportion of the total world trade volume. World trade in sugar is characterised by special marketing arrangements between exporting and importing countries. Until recently, quite an appreciable amount of sugar needs of Nigerians were imported. With the advent of sugar production in the country, import has been reduced to a certain degree. In most sugar producing countries, tariffs on sugar import are imposed to protect their domestic producers. The government of Nigeria since in the 1970's had been making efforts to improve sugar production in Nigeria so that Nigeria could cope with the high sugar demands of her economy and with time become a major exporting power in the sugar trade. The withdrawal of a great power, in sugar trade, Cuba in [illegible] led to an increase of about 50% and this motivated many countries with potentials to start massive sugar production and by 1965, the price had fallen below the 1961 level.

The growth of sugar manufacturing in Nigeria can be traced back to 1957 when the Nigerian Sugar Company went into production. In this particular year, the total value of sugar importation into the country dropped by 25%. To protect the seemingly budding sugar industry, the the government of the country imposed import duty on sugar importation. Import duty on raw sugar was also revised upward in [illegible], and although the import duties are relatively low, even up to today, very large amount of sugar is still imported into the country for the large scale production of sugar in the country. However the quality of the sugar produced need to be improved upon especially the refining process.

2.2 CHEMISTRY AND TECHNOLOGY OF SUGAR PRODUCTION

2.2.1 INTRODUCTION

Most usually, sucrose, the common sugar of commerce that occurs universally throughout the plant kingdom in fruits, seeds, flowers and roots of plants. Sugars belongs to the class "carbohydrate" and as the name implies they are hydrate of carbons. This group of compounds plays a vitally important part in the lives of plants and animals, both as a structural element and in the maintenance of functional activity. Plants are unique in that they alone in nature have the power to synthesize carbohydrates from carbondioxide and water in the presence of the green plant chlorophyll through the energy derived from sunlight, by the process of photosynthesis.

Sugar belongs to the disaccharide, $(C_{12}H_{22}O_{11})$ group of carbohydrate and upon hydrolysis splits into two monosaccharides, or simple sugars. Sugarcane and sugarbeets are the chief sources for the preparation of sugar on a large scale. Other sources of sugar are [illegible] corn, Maple plant and honey.

2.2.2 STEREOCHEMISTRY OF SUCROSE

Sucrose is an oligosaccharide, D glucopyranosyl β-D fructofuranoside, also known as saccharose, cane sugar, or beet sugar. The structure is shown in the illustration below

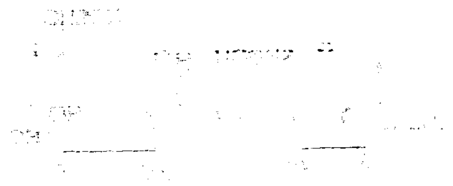

FIG. 4: STRUCTURAL FORMULAR FOR SUCROSE

On hydrolysis, sucrose splits into two simple sugars, glucose and fructose.

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

Sucrose Water Glucose Fructose

Sucrose rotates the plane of polarised light to the right as does glucose, but fructose is so strongly laevorotatory that it overcomes the effect of glucose. This hydrolytic reaction is called inversion of sugar. This reaction is of immense benefit to the manufacturers of sugar so that adequate measures will be taken during evaporation process to prevent sugar inversion.

The specific rotation of sugar is $[\alpha]_D^{20}$ is +66.5 and melting point (M.P) is 186°C. It is non reducing and is not fermentable by yeast. Sucrose is very soluble in water and crystallizes from thin solution in an anhydrous form. In plants sucrose is synthesised from uridine diphosphate D-glucose and D-fructose or D-fructose 6 phosphate. In the latter case the resulting sucrose phosphate is hydrolysed by an enzyme, phosphatase, to yield free sucrose.

The stereochemistry of sugars has been a subject of debate and researches among scientists in the recent past. Notable among researches carried out in the field [illegible] projection formulas which is based on the convention that tetrahedrons, representing individual asymmetric carbons of a sugar, viewed as though supported by their carbon to carbon bonds with the hydrogen and hydroxyl groups extending outward are projected upon the depicting plane. This formulor is quite convenient because the relationship between various sugars is readily demonstrated. In 1927 W.N. Haworth suggested that the five atom ring sugars should be regarded as derived from furan and the six atom ring configuration from pyran.

2.2.3 NUTRITIONAL VALUE OF SUGAR

Sucrose, has in the past been attacked by some nutritionists on the ground that it provides only "empty calories", without proteins, minerals, or vitamins. The argument lost much of its force when it was shown that all the vitamins and minerals recommended by the Research Council can be obtained by consuming any of a great variety of foods in amounts that yield a total of only one-half of the calorific requirements of an average person.

The problem of sugar and tooth decay is not so easily disposed of, however, and continues to be the subject of active research. It is currently believed that dental plaque is a dextran, formed from sucrose and important in the etiology of caries. If this theory is confirmed, the systematic application of dextranases should be effective in preventing tooth decay in humans as it has been in animals.

Another charge against sugar is that it may be involved in the etiology of atherosclerosis. Certainly there is a strong correlation between sugar consumption in a population and the prevalence of atherosclerosis. Research is actively under way on the relation of diet to atherosclerosis with the prospect of definitive results in the near future.

2.2.4 SUCROSE AND SWEETNESS

Not all sugars are sweet. Among the oligosaccharides sucrose is the sweetest. Milk sugar (lactose) is nearly void of sweetness and Gentiobose taste bitter. The variable sweetness of the sugar is therefore directly related to their stereochemistry, and the sugars contained within their structure information that leads to developing an understanding of the initial chemistry of the sweet taste response itself.

Sweetness can be defined as the sensation experienced upon tasting pure sucrose. Of all the sugars, fructose is the sweetest when sucrose is used as the standard for the sensation sweetness, there is no other compound that possesses all of the sensory features associated with the taste of sucrose. These include the impact or onset time, the intensity of the sensation, and its duration.

Other sweeteners which mostly are synthetic are in active competition with sucrose for industrial use. Such synthetic sweeteners include saccharin, which is about 300 times sweeter than sucrose, and cyclamates which is 30 times as sweet as sucrose.

2.2.5 DIGESTION AND ABSORPTION OF SUGAR

Sucrose has a complex structure and to be absorbed by the human body, it needs to be broken down into simpler molecules. Sucrose is non-reducing but due to its unique carbonyl to carbonyl linkage, it hydrolyses readily in acidic condition forming a mixture of D glucose and D fructose, called "Invert Sugar". The complete breaking down of this complex molecule (sugar) into simpler ones and breakdown of large food particles into smaller ones is known as digestion.

Digestion is both physical and chemical. The physical process involves the breakdown of large food particles into smaller ones and the chemical process also called "inversion" involves the breakdown of larger molecules ($C_{12}H_{22}O_{11}$) into smaller ones ($C_6H_{12}O_6$). Each stage of digestion involves inversion process supported by hydrolysis and catalyzed by a hydrolysing enzyme.

The simple sugars i.e glucose and fructose are absorbed into the bloodstream at different rate. Glucose has a faster absorption rate than fructose. Glucose is transported into the bloodstream not by passive diffusion as in case of fructose but by active transport.

2.2.6 DISORDERS RELATED TO SUGAR METABOLISM

A OBESITY

This disorder has been with man for centuries and in many societies it was associated with affluence and wealth. As a result of excess sugar in the bloodstream due to shortage of insulin, the excess sugar is converted to fat thereby leading to accumulation of excess body fat. This indicates that energy intake from food exceeds energy expenditure. Obesity therefore results from a positive energy balance which in turn may result from a number of causes.

According to recent evidence the obese person is also more affected by external cues with regard to regulation of food intake than his lean contemporary. An obese person is mostly restricted to taking formula diet which may not be adequate to satisfy his hunger. As these person usually find it difficult to partake in physical exercises which reduces energy expenditure hence leading to further accumulation of calories.

Prevention and treatment rests on avoidance of positive calorie balance and production of negative calorie balance, respectively. This result can be reached by either increasing physical activity or decreasing calorie intake.

B. DENTAL PLAQUE

Currently, dental plaque is believed to be a dextran, formed from sucrose and important to the etiology of caries. The non ionic character of sucrose serves as a facilitator for the relatively free passage of sucrose into the deeper layers of dental structure of man.

Vanhoute and Saxton[5] studied the morphology of human dental plaque using an electron microscope. In the inner part of the plaque, many microorganisms exhibited unusually thick cell walls and contained large amounts of intracellular polysaccharides. The organisms starved of sugar had thin walls and less plaque was formed[5].

To prevent tooth decay, the systematic application of dextranases should be effective as it has been in animals.

2.3 CANE SUGAR TECHNOLOGY

2.3.1 History and Development of the Technology

The word "shaka" was used by the persians in ancient times to describe a sweet spice which passed through their markets on the caravan route from East to west. Hindu literature would have made us believe that it originated in north-eastern India around 1,000BC, while others maintain that the Chinese knew of it many centuries earlier.

It was made from a cane with grass like leaves which grew twice as tall as a man and, unlike the bamboo, could be chewed in the mouth and yielded a pleasant sweet taste.

but if it were allowed to grow for too long and became hard or [illegible] uneaten after being cut, it would soon lose much of its flavour and [illegible].

People in colder places who had never seen sugar cane came to know this rare spice as a delicacy not unlike honey but purer in taste and much easier to carry about. This was worth money, perhaps gold, to the traders along the way, and so a demand was created and a manufacturing industry was born. Their first concern was to achieve a consistent product. It was made in batches, and sometimes it would turn out black and evil smelling while at others it would be a reasonable light brown. Then just occasionally, one batch might appear almost golden and very attractive indeed. Possibly someone found out that one of the workers who cleared away ashes from the fire place had let some drop in the tray of fresh juice standing ready to be boiled the next day. The addition of a little wood ash to slightly warm juice—allowing it to stand for some hours — had a "clarifying effect". This became one of the many techniques of purification that were to turn sugar boiling into an art. As time went by, those who knew kept this knowledge as secrets and passed them guardedly.

Each new attempt to boil sugar in a different place would be met with fresh difficulties in controlling the appearance of the final material. Various treatments would be tried out on the juice before the best one was found. Among the substances used were plant extracts, animal's blood and some [illegible] essentially unchanged [illegible] boiling [illegible] with [illegible].

As wealth and prosperity grew [illegible] entrepreneurs on both sides of the oceans to fill the demand for sugar crushers could be built in less time than it took to propagate the cane, but finding the expertise to operate them was becoming more difficult. The traditionally secret arts of the sugar boilers had to be rationalized so that more people could be trained to boil a good sugar. Even though it was a seller's market, the better the appearance of the product the higher the price it would fetch. Among many who sought to make their fortunes, the more astute could foresee that a certain quality standard would sooner or later become crucial to their continuing in business.

Fortunately for them, the preceding century had seen the foundation of the science of inorganic chemistry. The concept of acid and alkaline solutions and what could be expected to take place in certain conditions when known substances were brought together in water had been tried and tested and the results published. This could provide the key to controlling the process of purification and securing good and consistent product appearance. The emergence of the sulphitation process superseded, almost universally, the traditional methods of juice purification. It involved the burning of sulphur and passing the fumes through the juice while adding lime in a sufficient quantity to counteract the resulting acidity. This caused a large proportion of the mineral impurities to precipitate and to sink to the bottom of the tank. The complete sequence of operations for the production of sugar in this way is now called the open pan sulphitation (OPS) technology.

The refining industry, which was growing quite large in European and American Atlantic coast seaports, could charge a high premium for [illegible] sugars and they [illegible] experimenting with loaf sugar. This was made by pouring massecuite into inverted cone-shaped moulds, and when it has set they would pour some of the original clear liquor the cones to wash away the coating of yellow syrup from the crystals. It was far too costly a process to be applicable to the thousands of tons of unrefined sugar that were being produced for shipment as general cargo across the sea.

The emergence of the vacuum pan technology signalled a new era in sugar technology. The industry due to this technology have been dominated by larger plants. However there is still a need to encourage the OPS technology so that small scale plants could thrive. It may take five years, it may take ten, but at some time a transition is likely to be needed from OPS to VP technology. This process will be accelerated if labour costs rise as the prosperity of the area grows

2.3.2 Introduction to Processing Techniques

The process of converting sugarcane into white sugar can be conveniently divided into six stages and into a "front end" and a "back end" as shown below.

Crushing	"Front end"(juice extraction)
Clarification Boiling Crystallization Centrifuging Drying	"Back end" (juice processing)

The vacuum pan (VP) factory has a greater efficiency in both front and back ends than the open pan sulphitation (OPS) plant. A very rough idea of the differences can be gained from table below

Table [illegible]

Process	[illegible] (per cent)	Refining Losse[illegible] [illegible]	[illegible] per [illegible]
[illegible] (high)	[illegible]	[illegible]	[illegible]
[illegible] (low)	[illegible]	[illegible]	[illegible]
[illegible] (high)	[illegible]	[illegible]	[illegible]
[illegible] (low)	[illegible]	[illegible]	[illegible]

The higher [illegible] figures [illegible] approximately [illegible] pending [illegible] figure [illegible] rather than [illegible] these sugars, the low figure corresponds [illegible] condition where only one [illegible] sugar only.

All the figures [illegible] are indicative and depend on the quality of the cane [illegible] the factory [illegible] sugar content and other [illegible]. This will vary with, among other things, the [illegible] size, the climate [illegible] content of [illegible] process [illegible] imposes [illegible] removing [illegible] of many [illegible] of [illegible] and [illegible] [illegible] of the [illegible] extremely [illegible]

The [illegible] process [illegible] largely [illegible]

Table [illegible] [illegible] where [illegible] during [illegible]
and [illegible] the [illegible]
[illegible]
[illegible]

Inversion: High temperatures and acid conditions lead to the chemical decomposition of sucrose into simpler sugars. By boiling under carefully controlled conditions, the VP process reduces inversion of sucrose.

Crystallization: In a VP plant the crystallization process is a very carefully controlled. This ensures maximum yield of sucrose from the massecuite.

In addition to having a higher sugar recovery, the VP process is more energy efficient than the OPS process.

2.3.3 The Refining Technology:

Cane sugar refining is classified as the "back ends" of the production techniques. It forms the major parts in obtaining a good yield as well as a pure sugar fit for truman consumption. Impurities such as waxes lipids, gums, pectin etc. have to be removed to give the desired quality of sugar.

The technology of removing these impurities has remained basically unchanged for many years, but recently some progresses have been made in the refining industry. Such improvements in the refining process include the clarification process where phosphoric acid or a soluble phosphate and lime are used as clarifying agents rather than the former mechanical filtration. Activated carbon is now the major adsorbent used for decolorisation of the syrup. Inversion of sucrose which forms the major problem in the boiling process have been tackled by the introduction of vacuum boiling technique. Some of the major operations carried out in refining process are:

A. Affination: This process involves the removal of the film of molasses from the surface of the raw sugar. The sugar crystals are essentially pure sucrose. The crystals are washed with hot water to effect further purification. The washed crystals are dissolved in about half their weight in water in melters. Heat is applied in order to accelerate dissolution of the sugar.

B. Clarification: The raw washed sugar liquor from the melter contains some insoluble materials, such as bagacillo, clay, or sand, and an appreciable amount of fine suspensions and dispersoids, as well as gums, pectins, and other true colloids that have escaped the clarification of the raw house or have been formed in the subsequent process of manufacture. The raw liquor is also acidic.

Clarification can be said to be a chemical process designed for the treatment of the raw liquor with certain chemicals and heat to render the solution suitable for filtration. The phosphate - lime treatment with heat first neutralizes the organic acids present in the raw sugar and then forms tri-calcium phosphate. The heating and defectants (lime and phosphate) cause the coagulation and precipitation of organic non-sugars. The precipitates occludes much colloidal matter, including an appreciable amount of colour.

C. Decolorization: This is one of the major steps in the refining process. Any colorant in the sucrose is removed during this process. This is done by adsorption. The adsorbent is powdered activated carbon. The colouring matter are adsorbed physically on the surface of the activated carbon pore surface.

These carbons are regenerated at intervals in order to maintain their effectiveness. Other adsorbents that are in use worldwide include bone char, ion exchange resin.

Crystallization and Finishing: In the production process, water is used mainly as the solvent and it accounts for about 30-35% of the decolorised solution. Vacuum pans are employed in the evaporation of water and later the resulting sucrose solution is crystallized. After the crystals are formed, they are washed and then transferred to centrifuges for removal of all free liquor. The moisture content of sugar leaving the centrifuge is too high for convenient handling and storage. To prevent the growth of micro organisms which will cause fermentation, the sugar is dried. This also prevents loss of sugar on storage.

2.3.4. POLLUTION CONTROL IN SUGAR INDUSTRY

The effect of pollution as a result of industrial operations on ecological balance is generating a lot of discussion among environmentalist and people as a whole. The ability of pollutants to alter the state of the environment has added a new dimension to the task of the engineer. To meet this demand, engineers have devised different means of controlling pollution either at the design stage of the plant or introducing a new approach at production stage to curtail the pollution.

The federal government of Nigeria through legislation set up conditions (law) to guide environmental pollution. These laws are enforced by environmental protection agencies. These regulations are set to check the activities of the process industries and to ensure the safety of lives and properties

In the sugar manufacturing industries, waste products of the production process constitute hazard to the environment hence steps are taken to ensure that this trend is corrected. To control this, some factories use some of their by products as energy source. By products like bagasse are burnt to generate steam, hot water and for boiling process. Other pollutants that are associated with the sugar producing factory are the waste solution of molasses from crystallization process and waste water used for treatment of the cane. If the liquor and waste water are not treated before releasing into the river system it will alter the ecologically balance of the aquatic life. To treat this, an effluent treatment plant will be needed to treat this water before it is disposed off. Effluent treatment plants are recently being built by many process industries in their bid to control water pollution as in case of the sugar manufacturing industry.

The small scale sugar producing factory burns most of their by products in their power plants to generate heat energy for boiling of water and the evaporation process proceeding crystallization. Ultimately, the sugar producing industry has witnessed a good pollution control system which can be improved upon.

2.4 LOCAL MANUFACTURING TECHNOLOGY OF MARZAKWALLA

The local production of brown sugar (marzakwalla) in marzakwalla local government area of Kaduna state is crucial to understand the source of impurities in the final product. Though from research, there is not much difference between the local and the modern technology, hygiene and the fact that the clarification process is omitted in the [illegible] technology play an important role in determining the quality of the product. The production of [illegible] is found to include the clarification process responsible for the removal of soluble and insoluble impurities. The environment under which [illegible] is being produced is unhygienic to the extent that sands and clay are other components of the final product.

The manufacturing process are outlined below.

A. Juice extraction: Juice extraction by crushing the cane between massive rollers is the first step in producing [illegible]. The mill consists of three roller mechanism through which the crush cane or bagasse successively passes. The rollers are rotated by a horse running in a circle and controlled by a man. Unextracted sugar, woody fibre, and 40 - 50% of water are major content of bagasse.

B. Evaporation: The extracted juice containing about [illegible] water is transferred into the boiling house to concentrate the juice batchwise until the solution is saturated with sugar. Under atmospheric pressure concentration at normal boiling point is largely responsible for the discolouration of the sugar among other reasons. Drops of groundnut oil are poured into the boiling juice to reduce the surface tension when foaming begins. Some of the impurities coagulate and float on the surface as scum and these are scooped off from time to time with a large metallic spoon. This is the major heat consumption process in the production process.

C. Crystallization and moulding: The second step of the crystallization involves a sharp change of temperature to produce condition of lower solubility and consequent superation and crystallization.

The super saturated solution is quickly transferred into an open day pot placed on a cold surface where it is continuously agitated with a wooden stick. The reason is that crystallization, especially in such a viscous solution, is a slow process. Sudden cooling of such a solution will produce a temporary conditions of supersaturation simply because the system is slow in adjusting itself to its equilibrium conditions. Agitation of the solution will hasten this adjustment.

The super saturated solution is then seeded with powdered potash (commonly called kanwa), the rate of addition being varied as required. The potash acts as nuclei for crystallization. This seeding operation is known as graining. The crystallization process depends mainly on the skill and experience of the person carrying it out in the local sugar industry, unlike in the modern industries where it is regulated by equipment/instruments i.e. the degree of super-saturation within suitable limits.

The mazarkwaila produced are finally given a definite shape by pouring the thick syrup into tin moulds where they are allowed to solidify and dry. The manufacturing process is shown diagrammatically thus

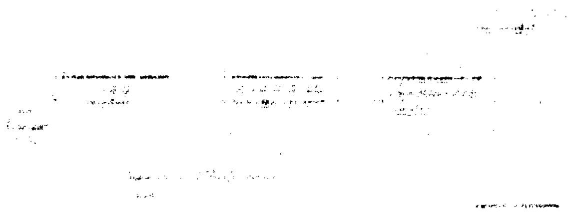

Fig. 2.[illegible] Manufacturing process of [illegible]

CHAPTER THREE

EXPERIMENTAL WORK

3.1 MATERIALS AND APPARATUS

The underlisted materials and apparatus are used for the practical work carried out in the Chemical Engineering Laboratory of Federal University of Technology, Minna.

Apparatus

i. Measuring cylinders
ii. Electric stove
iii. pH meter
iv. Thermometer
v. Weighing balance
vi. Filter papers
vii. Mortar and pestle
viii. Stirrer
ix. Open pan (aluminium)
x. Beakers
xi. Funnels
xii. Refrigerator

3.2 CHEMICAL REAGENTS

The following reagents are used for the refining process.

i. Powdered activated carbon – colour adsorbent
ii. Lime
iii. Phosphoric acid
iv. Sodium hydrogen phosphate } 0.1M solution each
v. Sodium orthophosphate }
vi. Manzakwaila samples
vii. Icing sugar
viii. Distilled water

[illegible].2 PROCEDURE

Batchwise method was adopted for this experiment. The experiment consists of two runs A and B. Distilled water was used as solvent to enhance high purity of product. The sequence of experimental work carried out are outlined below:-

A. 100ml of hot distilled water (70°C) was used to dissolve 50g of the sample in a clean beaker.

B. The raw sugar liquor was weighed, filtered and the pH noted.

?

C. A known volume of 0.1M clarifier was dissolved in 100ml of distilled water and added to the sugar liquor, after which 50/100ml of lime solution was added to complete clarification. The lime solution was used to adjust the pH of the liquor to acceptable level (between 6.5 – 7.5).

D. The clarified liquor was allowed to settle until a clear solution is obtained. The precipitate was removed from the surrounding liquor by subsidence and decantation.

E. Decolorisation was carried out with the use of activated carbon of known weight and type.

F. To recover the adsorbent, the decolorised liquor was filtered.

G. The decolorised liquor was boiled in an open aluminium pan to vaporise the water content. Continuous stirring was ensured to enhance even distribution of heat.

H. At the reach of sugar saturation, the liquor was seeded with a small quantity of icing sugar to enhance crystal growth in the mass ([illegible]).

I. Crystallisation was continued by transfering the liquor [illegible] (super saturated) into a plate placed on cold water or into a freezer to fast cool. Constant agitation was ensured to hasten adjustment to equilibrium conditions. It was then allowed to cool and finally crystallize.

J. The refined sugar was weighed after drying.

K. The above sequence was followed for different samples of mazarkwaila.

L. 5grams of each refined sample was dissolved in 10ml of hot distilled water (3.[illegible]). 5ml of the sugar solution was then poured into the polarimeter tube to determine the degree and direction of the rotation of the plane of polarized light of the sugar.

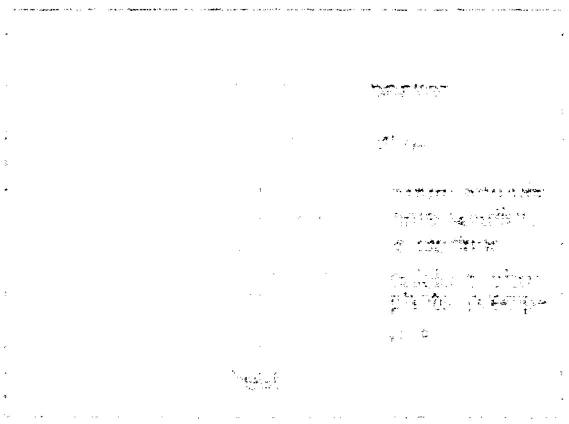

Fig. 3.1 Diagrammatical representation of clarification exercise.

CHAPTER FOUR

RESULTS AND ANALYSIS OF RESULTS

4.1 Codes for experimental results

Colour codes

white	W
fairly white	W-
light brown	B
brown	B+
insignificant colour change	C

1. Mode of crystallisation

Forced crystallisation	FC
Seeding crystallisation	SC

2. Clarification temperature for all samples is 70°C

4.2 Table of results : The results obtained from the experimental work are presented in tables 4.2.1 - 4.2.7

TEST RUN 1 USING 0.1M PHOSPHORIC ACID AS CLARIFIER

TABLE 4.2.1

DESCRIPTION OF PARAMETERS	SAMPLE RUNS	
	A	B
Weight of sample used (g)	50.00	50.00
Volume of water used to dissolve samples (ml)	100.00	100.00
Weight of sugar solution (g)	140.00	47.[illegible]
pH of raw sample liquor.	5.70	5.70
Volume of phosphoric acid dissolved in 100ml of distilled water (ml)	0.50	[illegible]
Weight of lime dissolved in 100ml distilled water (g).	.00	[illegible]
Weight of mud removed after clarification (g)	20.14	24.25
pH of clarified liquor.	6.50	6.20
Weight of adsorbent used for decolorisation (g).	10.00	10.00
Weight of decolorised liquor (g).	261.89	258.[illegible]
Mode of crystallisation.	CD	[illegible]
Weight of refined sample (g)	28.25	27.73
Pol	0.20	0.20
Brix	0.36	0.35
Purity (%)	56.15	56.8[illegible]
Recovery (%)	56.66	55.46
Colour code	W	[illegible]

EXPERIMENT USING [illegible] PHOSPHORIC ACID AT CLARIFICATION

TABLE 4.2.3

DESCRIPTION OF PARAMETERS	SAMPLE CODE	
	A	B
Weight of sample used (g)	50.00	50.00
Volume of water used to dissolve samples (ml)	100.00	[illegible]
Weight of sugar solution (g)	140.50	[illegible]
pH of raw sample liquor	5.70	[illegible]
Volume of phosphoric acid dissolved in 100ml of distilled water (ml)	0.20	[illegible]
Weight of lime dissolved in 100ml distilled water (g)	10	[illegible]
Weight of mud removed after clarification (g)	26.13	25.77
pH of clarified liquor	7.00	7.[illegible]
Weight of adsorbent used for decolorisation (g)	2.00	[illegible]
Mode of crystallisation	[illegible]	[illegible]
Weight of refined sample (g)	10.[illegible]	[illegible]
Pol	0.21	[illegible]
Brix	0.16	0.[illegible]
Purity (%)	50.04	54.02
Recovery (%)	58.3[illegible]	12.[illegible]
Colour grade	W	[illegible]

EXPERIMENT TWO: USING 0.1M PHOSPHORIC ACID AS CLARIFIER

TABLE 4.2.2

DESCRIPTION OF PARAMETERS	SAMPLE RUNS	
	A	B
Weight of sample used (g).	50.00	50.00
Volume of water used to dissolve samples (ml).	100.00	100.00
Weight of sugar solution (g).	140.57	142.05
pH of raw sample liquor.	5.70	6.00
Volume of phosphoric acid dissolved in 100ml of distilled water (ml)	0.30	0.30
Weight of lime dissolved in 100ml distilled water (g).	[illegible]	[illegible]
Weight of mud removed after clarification (g).	[illegible]	[illegible]
pH of clarified liquor.	6.70	7.00
Weight of activated carbon used for decolorisation (g).	[illegible]	[illegible]
Weight of decolorised liquor (g).	[illegible]	[illegible]
Mode of crystallisation.	[illegible]	[illegible]
Weight of refined sample (g).	[illegible]	[illegible]
Pol	[illegible]	[illegible]
Brix	[illegible]	[illegible]
Purity (%)	[illegible]	[illegible]
Recovery (%)	55.86	[illegible]
Colour code	W	W

EXPERIMENT THREE: USING SODIUM HYDROGEN PHOSPHATE ([illegible]) AS CLARIFIER

TABLE 4.2.4

DESCRIPTION OF PARAMETERS	RUNS	
	A	B
Weight of sample used (g).	50.00	50.00
Volume of water used to dissolve samples (ml).	100.00	100.00
Weight of sugar solution (g).	144.33	140.73
pH of raw sample liquor.	6.0	[illegible]
Volume of phosphoric acid dissolved in 100ml of distilled water (ml).	0.75	1.00
Weight of lime dissolved in 50ml distilled water (g).	1.00	[illegible]
Weight of mud removed after clarification (g).	21.33	21.52
pH of clarified liquor.	5.7	5.9
Weight of activated carbon used for decolorisation (g).	10.00	10.00
Weight of decolorised liquor (g).	157.62	147.08
Mode of crystallisation.	DD	DD
Weight of refined sample (g).	30.30	24.00
Pol	0.13	0.17
Brix	0.25	0.36
Purity (%)	52.22	47.24
Recovery (%)	52.72	48.00
Colour code	[illegible]	[illegible]

EXPERIMENT FOUR: USING 0.1M SODIUM ORTHOPHOSPHATE AS CLARIFIER

TABLE 4.2.F

DESCRIPTION OF PARAMETERS	SAMPLES RUNS	
	A	B
Weight of sample used (g).	50.00	50.00
Volume of water used to dissolve samples (ml).	100.00	100.00
Weight of sugar solution (g)	141.76	140.07
pH of raw sample liquor.	5.70	6.00
Volume of 0.1M sodium orthophosphate dissolved in 100ml of distilled water (ml).	0.05	0.50
Weight of lime dissolved in 100ml distilled water (g).	0.50	0.50
Weight of mud removed after clarification (g).	20.50	27.70
pH of clarified liquor.	6.50	6.70
Weight of activated carbon used for decolorisation (g).	10.00	10.00
Weight of decolorised liquor (g).	245.25	220.06
Mode of crystallisation.	SD	SD
Weight of refined sample (g).	26.17	29.22
Pol	0.18	0.18
Brix	0.35	0.35
Purity (%)	51.03	50.75
Recovery (%)	52.14	51.34
Colour code.	W.	[illegible]

EXPERIMENT FIVE: USING SODIUM ORTHOPHOSPHATE AS CLARIFIER
[0.1M SOLUTION)

TABLE 4.2.6

DESCRIPTION OF PARAMETERS	SAMPLES	
	A	B
Weight of sample used (g).	50.00	50.00
Volume of water used to dissolve samples (ml).	100.00	100.00
Weight of sugar solution (g).	140.54	143.[illegible]
pH of raw sample liquor.	6.00	6.00
Volume of 0.1M sodium orthophosphate dissolved in 100ml distilled water (ml)	0.35	0.35
Weight of lime dissolved in 100ml distilled water (g).	0.50	0.50
Weight of mud removed after clarification (g).	28.33	30.02
pH of clarified liquor.	7.00	7.00
Weight of adsorbent used for decolorisation (g)	10.00	15.00
Weight of decolorised liquor (g).	160.23	162.02
Mode of crystallisation.	[illegible]	[illegible]
Weight of refined sample (g.	27.93	23.[illegible]
Pol	0.20	0.16
Brix	0.36	0.35
Purity (%)	56.22	45.60
Recovery (%)	55.85	46.74
Colour code.	W	[illegible]

EXPERIMENT SIX: USING 0.1M SODIUM ORTHOPHOSPHATE AS CLARIFIER

TABLE 4.2.7

DESCRIPTION OF PARAMETER	SAMPLE	
	A	B
Weight of sample used (g).	50.00	50.00
Volume of water used to dissolve samples (ml).	100.00	100.00
Weight of sugar solution (g).	141.37	141.12
pH of raw sample liquor.	6.00	6.00
Volume of 0.1M sodium orthophosphate dissolved in 100ml of distilled water (ml).	0.35	0.35
Weight of lime dissolved in 100ml distilled water (g)	0.50	0.50
Weight of mud removed after clarification (g).	30.13	29.26
pH of clarified liquor.	7.00	7.00
Weight of adsorbent used for decolorisation (g).	12.00	10.00
Weight of decolorised liquor (g).	[illegible]	[illegible]
Mode of crystallisation.	SD	SD
Weight of refined sample (g).	[illegible]	29.37
Pol	[illegible]	0.21
Brix	0.35	[illegible]
Purity (%)	59.38	59.27
Recovery (%)	58.47	58.74
Colour code.	W	W

Table 4.2.8 ANALYSIS OF RESULT

CLARIFIER USED	RUNS	VOL OF CLARIFIER DISSOLVED IN 100ML OF WATER (ML)	WEIGHT OF LIME DISSOLVED IN 100/50ML OF WATER (g)	MODE OF CRYSTALLIZATION	YIELD(%)	AVERAGE YIELD(%)	POLARIMETER READING
	Test Run A	0.50	1.00	SD	56.46		[α] + 8
				B			
Phosphoric acid (0.1M)	1A	1.00	1.00	SD	55.46		[α] + 6
	1B	0.50	1.00	SD	58.32	56.98	[α] + 6
	2A	1.00	1.00	FC	53.86		[α] + 7
	2B	0.50	1.00(50ml)	FC	55.86		[α] + 13
		0.50	1.20(50ml)	SD	61.92		
Sodium hydrogen Phosphate (0.1m)	3A	0.75	1.00(50ml)	SD	57.72		[α] + 8
						50.36	[α] + 6
	3B	[illegible]	1.00(50ml)	SD	58.00		
Sodium ortho Phosphate (0.1m)	4A	0.50	0.50	SD	52.34		[α] + 8
	4B	0.50	0.50	SD	56.64		[α] + 9
	5A	[illegible]	0.50(50ml)	SD	55.86	54.71	[α] + 7
	5B	[illegible]	0.50(50ml)	FC	46.24		[α] + 5
	6A	[illegible]	0.50(50ml)	SD	58.42		[α] + 15
	6B	[illegible]	0.50(50ml)	SD	56.74		[α] + 12

The Refractometer reading were constant at a value of 1.42

Initial Refractive index of sample = 1.34

4.3 DISCUSSION OF RESULTS

Locally produced brown sugar "mazarkwaila" apart from having a very poor colour also contains a high percentage of soluble and insoluble impurities which include waxes, fat, gums argument etc. The results obtained from experimental work show a great deal of improvement in the quality of the sugar as shown by the improved colour tone and removal of impurities. Of utmost value to the refining process is the defecation (clarification) and decolorisation reagents used. The basic reagent used for defecation process is lime but due to the crude nature of mazarkwaila some precipitating reagents have to be introduced as clarifier. Three clarifying agents were adopted for this process to determine the most suitable. Activated carbon is used as the sole decolorisation reagents.

The three clarifying agents used are:

(i) phosphoric acid

(ii) sodium hydrogen phosphate

(iii) Sodium orthophosphate.

Phosphoric acid is the most popular clarifying agent used in the sugar industry. From results obtained, phosphate treatment gives an average of 58percent colour removal (table 4.[illegible], 4.[illegible]) by removing polyphenols and iron that gives sugar its brownish colour. However the determination of the quantity of lime to be added depends on the phosphoric acid content is needed for pH control. According to Staffle's plan, high temperature and acidic condition enhances the decomposition of sugar hence to reduce the inversion of sucrose (cane sugar), an optimum formular for lime phosphoric acid combination has to be worked out. From the results obtained 0.5ml of phosphoric acid dissolved in 100ml distilled water is sufficiently neutralised by 1.2g of lime dissolved in 50ml distilled water. This combination gives the best clarification results. Of the three clarifiers used, phosphoric acid gives the highest yield and at the same time, best colour tone. The product of the test run gives fairly good result, however the brown colour associated with sample 3 is due to the acidic condition of the liquor evaporation which lead to "inversion of sugar" characterised by brownish colour. A good pH control and boiling the liquor under vacuum will definitely minimise the loss of sugar due to inversion and at the same time give a pure white sugar crystals. It is also discovered from experimental results that the higher the volume of phosphoric acid added to the sugar solution during defecation process, the higher the tricalcium phosphate precipitate obtained but this is limited by the pH control as more phosphoric acid in sugar solution means increased acidity.

The workability of sugar solution depends on the amount of impurities removed from it which in turn depends on the concentration of lime and acid used. Phosphoric acid gives clarified liquor a high working quality compared to the other clarifying agents i.e. sodium hydrogen phosphate and sodium orthophosphate. From Table 4.1 & 3, it shows that the highest impurities removal is 33.17g and this give the purest, whitest and highest recovery if the sugar crystals. An improvement is expected on this record if the sugar liquor is boiled under vacuum. The defecation process is temperature dependent, the higher the temperature, the higher the degree of clarification. However, the temperature used for each experiment is kept at an average of 70°C as used in sugar industry. This apart from minimizing decomposition of the reducing sugars, will also give room to determine the actual effect of the clarifying agents on the sugar solution. In essence the rate of formation of the calcium phosphate precipitate solely depends on the lime - acid formular used for defecation. Temperature, however, does not have a direct influence on the yield but certainly on the formation of precipitate.

The formation of precipitate as shown by table [illegible] shows a low precipitation yield, about 10.34% lower than that of phosphoric acid treatment. This could be attributed to the fact that sodium hydrogen phosphate is highly acidic and hence requires large amount of lime for neutralisation. An inspection of the results obtained for sodium hydrogen phosphate treatment will clearly reveal that to obtain [illegible] sugar, quite a large amount of liming will be needed for pH control which may not be economical in the long run. The percentage recovery as shown in table 4.4 is invalid as these seemingly tolerable values [averagely 50.18%] could be attributed to presence of large amount of impurities not removed during defecation process. Hence it is advisable to discard the use of this clarifier in sugar refining as it is uneconomical and time consuming. The products obtained from sodium hydrogen phosphate is characterised by insignificant colour change due to inversion of sugar which leads to the production of non recoverable monosaccharides: glucose and fructose. Similarly the workability of the sugar solution is greatly reduced due to the low formation of the calcium phosphate yield. During the filtering process, [illegible] is notable that liquor is either saturated which is not comparable when clarification is carried out by phosphoric acid and sodium orthophosphate.

A rundown of results obtained during clarification by sodium orthophosphate (Table 4.4.5 - 4.4.7) reveals an average yield of 51.71% which is about 7.4% less than that obtained from phosphoric acid treatment. Sodium orthophosphate is slightly alkaline, hence small amount of liming will be required to bring the pH to the accepted level (7.00). In Table 4.4.5, the formation of calcium phosphate precipitate compares with that obtained for phosphoric acid treatment. Contrary to expectation, less acidic sample B clarified liquor, gives a slightly brown sugar while sample A liquor which is more acidic gives a fairly white crystals. This could be due to the heating of sample B upto the point of saturation. Table 4.4.7 shows the best result obtained for clarification by sodium orthophosphate. Calcium phosphate formation is moderate as shown in Table 4.4.7 as well as yield (58.53%ave). [illegible] (Sample A). The advantage that phosphoric acid treatment has over this is that the [illegible] structure of sugar is enhanced by the [illegible].

More often than not, phosphoric acid is used in preference to other clarifiers. This is due to advantages derived from its usage such as high yield, high precipitation formation and low cost.

The efficiency of the mode of crystallization on the growth of crystals is found to be, from comparison of results, very crucial to the refining process. Crystallization by seeding is found to be suitable for the production of white sug[illegible], the [illegible] crystallization [illegible] liquor, [illegible] results in a low sugar yield [illegible]. According to [illegible], seeding by grains of crystal enhances the formation of crystals and hence the crystallisation rate. This is supported by the results obtained from the experiment (see Table 4.1.1 - 4.1.7). Forced crystallization also results in low yield (Table 4.1.1, [illegible] 4.1.7), this is as a result of excessive seeding.

Generally, experimental results are related with the expectation [illegible] refinement. The [illegible] efficiency [illegible] of this process depends on the degree of significant [illegible] increased by the results obtained from the experimental work carried out on the refining of locally produced brown sugar "[illegible]", a higher yield and whitest product was obtained ([illegible] experiment [illegible]). These achievements are traceable to the degree of purification. In this experiment, a high proportion of impurities, [illegible] gums, waxes, fats, pectins which reduce marketability are removed from the [illegible] sugar which gives [illegible] and improves the [illegible] marketability of [illegible].

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Generally, experimental results conform to the general opinion found in different literatures. The use of phosphoric acid as the clarifying agent is found to be more effective when compared to other clarifying agents used i.e sodium hydrogen phosphate and sodium orthophosphate.

To reduce decomposition of sugar as a result of excessive heat, continuous stirring of the liquor is ensured and boiling is stopped when super saturation is almost reached. These two precautions help in no small measure to obtain a good yield and a good colour tone for the refined sugar. To fasten grain growth, it is advisable to use aluminium pan of large surface area for crystallization process. It is also discovered from the lab that crystallization by cooling is more effective than forced crystallization as it enhances grain growth.

The average yield and purity percentage for the clarifier used are shown below:

Clarifiers	Average yield (%)	Average Purity(%)
Phosphoric acid	77	[illegible]
Sodium hydrogen phosphate	30.36	[illegible]
Sodium orthophosphate	8.71	[illegible]

It could be rightly concluded that, high yield of products (refined sugar) is gotten from highly purified liquor. The higher the purity of the clarified liquor, the higher the yield of refined crystals.

5.2 RECOMMENDATION

From research work carried out and experiments, the following recommendations are made to help local sugar manufacturers to improve on the local technology which will have a positive influence on the marketability of the local sugar for human consumption and as feedstock in industries:

A. The workability of a sugar solution depends on the existence and efficiency of a good clarification process which is omitted in the local technology due to "illiteracy". Efforts should be geared toward educating the local producers on the need of the process. An appropriate package of a well designed clarification process that will fit into the already existing local technology should be presented to them through practical demonstration. This form of programme has been adopted in countries like Kenya and Tanzania.

B. The cost implication of refining unclarified finished product (sugar) such as marzakwaila on the cost of production should be carefully analysed so that the pricing of the refined product can compete favourably with the of white sugar produced through modern technologies. By doing this, cost-reduction techniques can be suggested to reduce the cost of production and at the same time maintain a reasonable quality level. This is the principle of optimization of product quality at the cheapest possible cost.

C. Although, the enhancement of product quality yield and purity through pH control, mode of crystallisation and selection of suitable clarifiers is effective, vacuum boiling to reduce the decomposition (inversion) of sugar to non-recoverable monossacharides is considered most essential. Further work on this project should incorporate this process in refining marzakwaila. This will definitely give a better refined product which will be able to meet minimum standard requirements.

D. The quality of sugarcane as a raw material for the production of sugar plays a major role in determining the quality of the final product. Sugarcane hybridization researches should be encouraged to enhance the quality of sugar cane through cross breeding, inbreeding and selection. This programme will improve the juice content of the plants as well as their yield. Sugarcane grown under drought condition often contain large amount of wax which could prove difficult to eliminate during processing, therefore it is advisable to avoid the use of this kind of sugar cane as much as possible.

E. Further work on this project should make use of open aluminium pan as a tool for boiling and crystallising sugar syrups as this will reduce heating time, and also increase the crystal surface area for faster grain growth. Aluminium pan should replace evaporating dish due to its high conductivity of heat.

F. Efforts should be made by scientists to improve on the ability of activated carbon to absorb the brown colour of a viscous sugar liquor. Improved adsorbent structure and adsorptivity will surely enhance the colour purification of brown sugar like mazarkwaila. The use of filter aid in the filtration process should also be looked into as mazarkwaila solution poses a serious problem during filtration as a result of it's high viscosity

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APPENDIX

1. Pol

To calculate Pol = $\frac{\text{weight of sugar in solution}}{\text{weight of sugar solution}}$

e.g. experiment one, Pol = $\frac{29.16}{140.56}$ = 0.21

2. Brix

To calculate Brix = $\frac{\text{weight of sugar and non-sugars in solution}}{\text{weight of sugar solution}}$

e.g. experiment one, Brix = $\frac{50.00}{140.56}$ = 0.36

3. Purity (%)

To calculate purity = $\frac{\text{Pol}}{\text{Brix}}$ x 100

e.g. experiment one, purity = $\frac{0.21}{0.36}$ x 100 = 58.04%

4. Recovery (%)

To calculate Recovery(%) = $\frac{\text{weight of refined sugar}}{\text{weight of sample (unrefined)}}$ x100

e.g. experiment one, Recovery = $\frac{29.16}{50}$ x 100

= 58.32%

GLOSSARY

Bagasse : The by-product of crushing of cane sugar. The residue obtained by crushing cane in one or more mills.

Molasses : The heavy, dark viscous liquid discharged by the centrifugals after no more sugar can be separated from the final massecuites.

Mud (filter cake) : The residue which settles at the bottom of the liquor after clarification.

Brix degree : An arbitrary scale for conversion of saccharimeter reading of sugar solution into its specific gravity. It can also be expressed as Brix percentage.