PRODUCTION OF SWEETENERS FROM LOCUST BEAN PULP

BY

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CERTIFICATION

I hereby certify that I have supervised, read and approve this project work is adequate in scope and qualify for the partial fulfillment of the award of Bachelor degree in Chemical Engineering.

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_____ EXTERNAL EXAMINER _____ DATE AND SIGN

DEDICATION

This project is dedicated to God and humanity.

My mother Late Mrs. Omobola Aguebor, My father Festus Aguebor, my sisters, brothers and also to my Dove Sheila Peters. I love you all and God bless you all.

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Many Thanks

To Many People

For Many Things

In Many Ways.

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I am greatly indebted to the following people for which I believe God my way they are: Igbinigie O.J, Helen Akhimien, Gordon Asamah, Tina Anih thank you all for being there for me Gob bless you.

Finally my warmest thanks goes to my brother Rev. R.E Aguebor and his family for being a brother and a family to me. And other relatives and well wishers too numerous to mention here, whose prayer, love, moral and financial support contributed to the successful completion of my course and this project.

May the peace of the Lord, which passes all understanding, keep your heart and mind through Christ Jesus.

Phil 4:7

ABSTRACT

Production of Locust bean sweetener and the % purity was investigated using a batch method.

The production of Locust bean sweetener wa[illegible]ried out and it can be concluded that for a better quality. Sweetener to be obtained from a sample of locust bean pulp there is need for proper clarification and sh[illegible]g time. The various percentage yield obtained are 9.0, 8.0, 7.0% respectively for the corresponding addition of lime $Ca(oH)_2$ 0.25, 0.3, 0.35gm respectively. The corresponding percentage purity obtained was found to be 55.9, 56.2 and 56.6% respectively.

The results obtained showed that the best quality sweetener was obtained in the first experiment. The highest percentage purity of 56.6% was obtained at the third experiment.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 BACKGROUND OF STUDY

The Africa locust bean "Parkia Biglobasa" belongs to the Minosaceac. This family is a close relation to the papilionaceac and they both belong to the lugiminous group. The Africa locust bean is a large tree, with a very broad sometimes pendent crown, supported by a trunk usually short and thickset. They are two botanically related species Parkia Filicoide occurring in the Sahel and Parkia clappertoniana occurring typically in wet Savannah regions. However, the species are not differentiated in this section.

The leaflets are composed of many, simple, smaller leaflets about 1.5cm long by 0.5cm wide. The leaflets are sub-rectangular with rounded tips. The flowers, red in colour, like drooping balls appear in dry season after the leaf fall. The fruits are long, narrow pods and flattered, brown or black when ripe, they contain black seeds embedded in yellow pulp.

The yellow pulp of the fruits is rich in carbohydrate and is like flour. It is used in stews. The fermented seeds used to prepare a condiment rich in protein, this spice is called soucribala or mustard seed or dadawa. The black seeds of the locust contain 26% protein and are also rich in calcium. The dry yellow pulp which is very edible and is used to make a local drink. The African locust bean grows wild in the Savannah regions and is important in Northern Nigeria.

The locust bean is a light loving tree, it requires deep, well drained soil and can withstand drought reasonably well, once its firmly established. The tree is propagated from seed, though suckers are also used. The tree shades pastures. The leaves and pods provide valuable cattle feed. Growth is very slow, the seedling need a lot of attention. The tree comes into bearing between the eighth and the tenth year, but yields are low until the twenth year.

Sugars are synthesized by various plants with the suns energy using carbon-diode and water from the atmosphere. They serve as the principal energy food for human and enter into countless manufacturing sequences. (B.S Norris 1979).

Sweet foods are highly palatable and very popular. Many processed foods have sweeteners added and much of their success depends upon obtaining the right degree of sweetness.

Research into artificial sweeteners has tow been going on for many years. Initially, the aim was to find a suitable, cheaper alternative to sucrose and later, to find a low energy diets. (Anita tull 1996)

Saccharin was discovered in 1879 and in recent years it has been used in the manufacture of many foods. It is known that saccharin is 300 times sweeter than sucrose.

1.3 OBJECTIVE

The objective are as follows

(i) To obtain an alternative substitute for soluble sugar syrup

(ii) To design a process line for the production of sweeteners.

(iii) Concentrated locust bean into syrup by intensity the syrup sweetener

1.4 METHOD AND SCOPE

The Locust bean was de-shelled and the dry pulp mixed with water. This is then left to ferment. After which the soluble part of the pulp was leached out.

The aqueous mixture is treated with caott (lime) to precipitate non-sugar and neutralize organic acids. Then the aqueous mixture is evaporated under vacuum. To form a thick syrup.

The scope of this study is to try and get a suitable alternative to soluble sugar from the juice of locust bean pulp.

1.5 RELEVANCE OF STUDY

The increase demand for sugar around the world due to population explosion and the fact that it is needed also as a raw material doe other industries is a clear signal that absolute dependence on importation of sugar to meet our domestic need is not ideal. This has led to an investigation of new processes for utilizing local material for industrial purposes.

The research work was therefore aimed at producing a sweetener from locust bean pulp to serve as alternative sugar source and also to broaden the application of the farm product into an ever-increasing tide of raw materials.

CHAPTER TWO

2.0 LITERATURE REVIEW

For many people food was, and is eaten mainly to fill the stomach and to provide the energy needed to sustain a particular way of life. But as science has been applied to Agriculture, it has been possible to develop food, which are not only filling but also pleasing to the palate. One of the ways in which food is made more palatable is to sweeten it. [R.S Shallenberger 1982].

The sensation experienced upon tasting pure sucrose is sweetness. In the same reference mode, sodium chloride tastes salty, quinine sulphate tastes bitter and dilute solution of hydrochloric acid tastes sour. The foregoing definition is to be regarded as an absolute definition of sweetness that by passes the argument as to whether or not sweet, bitter and sour are in "chemical sense" primary taste (H.G Baker).

The use of sugar as a sweetener probably began less than 3000years ago, when the dry sap of the cane was extracted. But although sugar was an effective sweetener it remained a rare, and therefore expensive, substance until quite recently.

All green plants make sugar and these can be stored in their fruits, roots, bulbs, stems or flowers. At different times people throughout the world have used the sap or juice extracted from plants and especially their stems to obtain sweet syrups.

The date palm, sorghum and the sugar marple are notable examples. Today, however just two plants supply most of the world's sugar. The sugarcane (Saccharum Officinarum) which is related to the garden but root. The refined sugar (sucrose) produced from both these sources is identical and though sugarcane has supplied the world for much longer. Sugar beet now provides nearly half the commercial supply.

The first large-scale production of sugar took place in the Caribbean, where plantations were developed only because of the extensive use of slave labour. Slaves were brought over from Africa, mainly from the West Coast to work for the European plantation owners. The Caribbean is still an important sugar producing area with Cuba the largest single producer.

Sugar as a whole is much maligned and generally regarded as "bad for us" but it is really only the quantity that many westerners consumes that has health experts worried. We need sugar to maintain body temperature and provide energy, but most of our essential is metabolized by the body itself from carbohydrates. We consume such as starch much of which is contained in quantity in cereal grains and edible roots. Sugar are also present naturally in small amount [in form of fructose in most green leaves and fruits and glucose found especially in grapes and vegetables] in many of the food we eat.

Although, the method of production of sugar has improved since about the middle of the eighteenth century. The changes to take place have occurred in last century. One of these has been the gradual change in emphases from sugarcane to sugar beet, both have the same sugar content of about 15%. The sugar is sucrose, which has the molecular formular [C_{12} H_{22} O_{11}].

However, sucrose is not the only substance called sugar. For example glucose and fructose, which are found in fruits and honey and which have the same molecular formular [C_6 H_{12} O_6] are also sugars. They may be obtained by the hydrolysis of sucrose with dilute mineral acids or with enzyme invertase in yeast. Water splits the sucrose molecules into simple glucose and fructose molecules.

$$[\ C_{12}H_{22}O_{11}(s) + H_{20} \longrightarrow C_6H_{12}O_6\,(aq) + C_6H_{12}O_6\,(aq)\ ]$$

SUCROSE GLUSOCE FRUCTOSE

2.1 SOURCES OF SWEETENERS

2.1.1 SUGAR CANE (SACCHARIUM OFFICINARUM)

Recent studies by Brandes and others (SPENCER MEADE) indicates that sugar cane originated in New Guinea rather than in India a previously believed.

The Arabs were responsible for the spread of the cane culture around the Mediterranean in the middle apes.

The Arabian influence establish the art of refining sugar in Egypt about 1000AD for several centuries the product continued to be a costly delicacy to which marvelous curative power were ascribed.

Although sugar from cane grown by the Arabs was in demand in Europe by the twelfth century as an expensive luxury, it was not until the establishment of plantations in Brazil and the West Indies early in the sixteenth century (following the introduction of the plant to the new world) by the exploitation of generations of slaves slipped from Africa. Entire countries were given over to sugar cane cultivation and large areas of tropical forest destroyed to make a way for this new cash crop. Though processing methods are now greatly updated and scientifically controlled, the cane crop supply itself is still very much labour intensive. Much of the world supply (around 95million tonnes of sugar) comes from Cuba, Hawaii, Puerto Rico, Brazil and India where cutting of the sugar cane by hand is common.

Raw sugar contains many of the chemicals (such as protein and fat) found in the sugar-cane stalks molasses the excess sticky brown liquid which is separated out from the newly formed raw sugar crystals by means of a centrifuge is sometimes re added to white sugar to make it brown.

2.1.2 SUGAR BEET (BETA VULGARIS)

The Romans grew sugar beets as vegetables many centuries ago, but their use as a commercial source of sugar dates back to the beginning of the nineteenth century, following the investigation of a German chemist who had extracted 6.2% of the sugar from the root of a white variety.

Today improved varieties and modern method of extraction have boosted the figure to about 20% and large quantities of sugar beet is now grown in Russia, France, Germany, Poland, U.S.A. one of the earlier promoters of sugar bet was Napoleon Bonarparte who encouraged the supply of sugar from cane grown in British dominated colonies.

The sugar is contained in the whitish conical roots of the plants, which are harvested, with an average weight of about 1kg. To extract the sugar the roots are first shedded and then heated in the running water. After the removal of impurities the clear

liquid obtained is concentrated and crystallized to give a sugar distinguishable to that made from sugar cane. Molasses is also produced from sugar beets.

The pulp that remains after the beets have been processed (along with the rosette of leaves from each plant) makes excellent food for cattle.

2.1.3 SUGAR MARPLE (ACER SACCHARUM)

In North America however, Indian groups had long used the sugar marple as a source of sweetness. After slashing the bark of the trees in the spring of the sap collected was boiled down to make a thick syrup.

The practice is continued on a commercial basis today and marple syrup has become a famous Canadian export and tourist attraction.

The sugar content of sugar marple syrup is much lower than of sugar cane, it takes 40 gallons of sap to make one gallon of syrup and the price remains high.

2.1.4 PALMS AND MARPLES

The sucrose content of sap from palm trees is extensively variable but is often exceeds 10%. This is a higher level that can be obtained from any other tree family. Sucrose is produced in parts of the tropical Africa and Asia by evaporating water from the sap of a variety of palm species.

2.1.5 PALM SPECIES WHICH SERVE AS SUCROSE SOURCE

The palm family (Palmae) consist of 2700 species. The coconut palm (cocos nucifera) is widely distributed but others have a limit natural rage.

Most palms are trees and in tropical areas they are second only to grasses in economic importance. Among the many foods and fibre products that are obtained is palm sugar also known as Jaggery. This is unrefined sucrose, obtained by boiling water from in some cases, tree sap and others the sweet juice obtained from unexpounded blossoms.

2.1.6 MARPLE TREES AS SOURCE OF SUCROSE

The sugar marple and the black marple (Acer nigrum Michx) are the only two of the thirteen-marple species native of North America, which are used to produce marple syrup. These species are favoured because their sap is much sweeter (higher in sucrose) than the sap from other species.

2.1.7 MINOR SUGAR SOURCE

Sugar is also produced from various cereal plants. Certain varieties of sorghum are grown for sugar production the sap being pressed out of their stan as in sugarcane. More important, however is the production of sugar by hydrolysis of the starch in maize grains. This process is carried out under the influence of dilute acid, produces so-called corn syrup. Their difference here actually is the fact that corn syrup is rich in glucose in contrast to all other commercially important plant sugar products, which consist entirely of sucrose.

2.1.8 SYRUPS

Syrups, which are sweeteners in liquid from usually of high viscosity, have been used as food sweeteners since the early days of mankind e.g honey. (Marie S. 1991).

2.1.8.1 SORGHUM SYRUPS

Sweet sorghum (sorghum bicolar) is a giant grass, somewhat similar in appearance, but able to withstand cooler climate. In mid-West and parts of the U.S.A the juice of sweet sorghum is heated, clarified by skimming and concentrated into a syrup. Sorghum juice tends to be higher in invert sugar than juice, it is therefore difficult to crystallize sweet sorghum sugar and syrup is the product of choice.

2.1.8.2 CANE SYRUP

The term "cane-syrup" for food industry use, are produced at sugar cane factories or at refineries where a blend of brown and golden coloured streams are combined to produce syrups. Cane syrups are dark golden brown in colour, with medium flavour intensity. A factory evaporator syrup is often completely inverted and mixed with univerted syrup.

2.1.8.3 CORN SYRUP

This is the purified concentrated aqueous solution of nutritive saccharides obtained from edible starch and has a dextrose equivalent of 20 or more.

2.1.8.4 MARPLE SYRUP

Marple syrup sweeteners made by the concentration of the sap of the sugar marple tree. Heating of the sap courses flavour and colour development as well as concentration, and the characteristics marple flavour of the syrup and sugar.

Physical properties of marple syrup are similar to those of sucrose syrups, making the product suitable for baking and confectionery, but, because of its cost, most marple syrup is sold directly.

The major sucrose producing palm species

Sugar palm (Borassus flabellifer)

Date palm (Phoenix sylvestris)

Coconut palm (Cocos nucifera)

Sago palm (Caryota urens)

2.2 SWEETENERS TYPES

There is a scarcely any area of food habit today that does not in some way involve sweetness, our use of sugar and sweeteners has increased and the diversity of natural and fabricated products available commercially and the use of sweet testing additives promises to increase even more (Horace I.S).

In assessing the relative sweetness attribute of a substance, it needs to be recognised that no two substances can have the same taste. When sucrose is used as a standard for the sensation of sweetness there is no other compound that possess all the sensory features associated with the taste of sucrose. These include the impact or onset time the intensity of the sensation and its duration.

Sucrose whether from sugar cane or sugar-beet has faced much competition in recent years because of the introduction of liquid sweeteners made from other sources chief among the rivals are corn syrup which contain glucose (made from hydrolised maize starch) and fructose and invert sugar syrups made by treating this same starch with enzymes or acids that convert it to glucose molecules. These syrups are widely used in place of sugar in many processed foods especially soft drinks and alcoholic drinks and baked goods, where quick fermentation is required.

Some interesting alternatives to sweet syrups and sugars exist as natural component of other plants. Potentially much safer than the very low calorie synthetic sweeteners already developed (such as saccharin which is made from petroleum, cyclamates whose use is now largely banned and aspartame formed by the unnatural bonding of two amino acids the source of which is kept a close guided secret, three plants in particular all form tropical West Africa have aroused much scientific interest.

The first of these, a herbaceous plant that grows up to 3m high (Thanmatococeono Danielli) has produced what is probably the sweetest substance known to man up to 4000 times sweeter than sucrose, crimson fruit which develop just above the soil surface contains the substance in the soft jelly like aril that surrounds the black seeds.

In humid rainforest of the native West Africa (Thaumetin Danielli) is well known to local people, they use is broad flexible leaves as a disposable plate or for wrapping food prior to cooking, and the long thin leaf stalks are harvested for met making, children suck the sweet arils of the fruit and these are also used as a source of sweetness in food preparations.

The protein (Thaumatin) now extracted for commercial use from the fruits is effectively non-calorific and therefore very suitable for dieters and diabetics. It is also sold as a flavour intensifier, since it will enhance a variety of sweet and savoury tastes while suppressing any bitterness. Also the bright red fruits of the serendipity berry (Dioscorophyllum cumminsii) which are only about one centimetre long and grow in grape like cluster of 50-100 fruits are also intensity sweet. The above principle, a protein called nonelin is around 3000 times sweeter than sucrose. The fruit of a third West African plant (Synsepelum dulcificum) have the unusual power in the word of the kew bulletin to change the flavour of the most acid substance into a delicious sweetness. The taste of a lemon or some other sour substance become wonderfully sweet after chewing just one plum like fruit for a short time. The glycoprotein responsible (aptly named miraculin) which has been extracted from the fruits has undergone much investigation for use as a food supplement in the West.

The Japanese are the pioneers of another plant derived sweetener "Sterioside" a white crystalline powder 250-300 times as sweet as sucrose refined from the leaves of (Stevia rebaudiana). The plant a member of the compositac family has long been used for sweetening drinks by the Guarani Indians of Paraguay who call it caa-ehe.

Approximately Relative Sweetness of Different Chemicals Compound to Sucrose.

CHEMICAL	RELATIVE SWEETNESS
Sucrose	1
Glucose	0.7
Fructose	1.3
Cyclamate	30
Chloroform	40
Glycyrlizin	100
Aspartame	200
Aeesulhame k	200
Dulcin	200

Saccharin	300
Sterioside	300
Sucralose	700
Alitame	2000
Thaumatin	2000
Perillartine	2000
Monellin	1500-3000
Neohesperidine dihydrochalcone	2000-3000.

2.2.1 ARTIFICIAL SWEETENERS

Artificial or synthetic sweeteners are those intensity sweet substance which do not normally occur in nature. They are as a result of specific chemical reaction and their structures are very diverse. The sweetness of compound such as chloroform, dulcin and saccharin has been known for well over a hundred years. At present only few substance have received government approval for use and these vary depending upon the country. Saccharin, cyclamate and aspartame are perhaps the most widely used sweeteners today (TNO 1991). Each artificial sweetener has its own physical and chemical characteristics which dictates how it is best used to achieve the desired result. The artificial sweetener industry is so well established now that it can be cheaper to use artificial sweeteners in foods rather than sucrose or other carbohydrate sweeteners (HOUGH CAM, PARKER KJ, VAJ. 1981).

2.2.2 NATURAL SWEETENERS

Natural sweeteners may be defined as those chemicals processing intensive sweetness which are derived from plant components roots, leaves, bark. Such substances have been used throughout history for sweetening food. The Aztec people as far back as 1570s used Lippia dulcis (verbanaceae) or "sweet herb" as a sweetening agent.

Thaumatin, another intensively sweet natural product 2000-2500 times as sweet as sucrose is a protein extracted from the fruit of the West African plant Thaumatococcuo danielli. Sterioside is an intensively sweet diterpene glycoside rebaudiana bertoni herb. It is about 250-300 times as sweet as sucrose and has a slightly estringent and bitter taste, with a mild after taste.

Although natural intensive sweeteners may have potential for commercial widespread use, few are actually used. Glycyrrhizin is the most widely used food flavour, it application are in the industries where low concentrations can impart desirable physical and sensory properties without the undesirable liquorice aftertaste observed with higher concentrations.

2.3 PHYSICAL AND CHEMICAL PROPERTIES OF SWEETENERS

Sweeteners all have PH values on the acid side minimum 3.5-5.5, to minimise colour and flavour development.

The reducing sugars characteristics of glucose, fructose and maltose allow perticipation in Maillard or browning. Syrup content is always expressed on a solid basis.

Sweeteners because of their solids content, have collective properties similar to those of sucrose invert syrups as regard boiling point elevation and freezing point depression. They have very high viscosities. Their hygroscopic properties vary with their carbohydrate composition.

2.4 PRODUCTION OF SWEETENERS

2.4.1 CLARIFICATION

Clarification may be defined as a chemical process designed for the treatment of raw liquor with certain substance and heat to render the solution suitable for filteration. The oldest clarification process is the use of phosphoric acid or lime and heat. These

process neutralizes organic acids present in the aqueous mixture and also precipitate non-sugars.

2.4.2 DECOLORIZATION

The next step is the decolorization of the solution. This is an important step due to the fact that sugar must be colourless before it is actually marketed carbon products are the most commonly used absorbent for the removal of colorants in sugar solutions within the refinery. The colouring materials are absorbed physically on the surface of the activated carbon pore surface.

In addition to the use of carbon, ion exchange resins are becoming widely applied in sugar refining (HARRY M.P, W.R JUNK 1979).

2.4.3 CONCENTRATION

The next operation consist of removing most of the water so that sucrose separates from the solution by crystallization, this separation is continued until the dissolved material also begins to crystallize at which point the operation is heated.

The main purpose of this unit operation is to reduce the water content of the dilute solution until they are close to the point of incipient crystallization.

2.5 USES OF SWEETENERS

It is used in the food industries for the production of canned food.

It is used as sweeteners for confectioneries like sweet, chewing gums etc.

It is used in the manufacture of drugs in pharmaceutical industries.

It is used for calorie reduced diet.

CHAPTER THREE

3.0 EXPERIMENTAL

3.1 SAMPLES AND SAMPLING

3.1.1 RAW MATERIALS

The mature pods of the African Locust bean Parkia Biglobasa occur in large bunches. The mature pod contains a yellow dry pulp (Powdery) (Dorowa Hau). Most of the beans are collected by individuals from their farms. It is commonly found in Kaduna villages.

3.2 MATERIALS AND REAGENTS

3.2.1 MATERIALS

(i) Weighing balance

(ii) P.H. materials

(iii) Oven

(iv) Filter pulp

(v) Funnel

(vi) Filter paper

(vii) Desicator

(viii) Kheldahl flask

(ix) Bowls

(x) Furnace

(xi) Measuring cylinder

(xii) Beakers

(xiii) Heating mantle

(xiv) Soxhlet extractor

(xv) Spectular wire guage

(xvi) Petri-dish

(xvii) Water bath

(xviii) Markham semimions N still.

3.2.2 REAGENTS

(i) Animal charcoal

(ii) Ethanol

(iii) CaoH. (lime)

(iv) Mixed catalyst

(v) NaOH

(vi) Boric Acid

(vii) HCL

(viii) Mixed indicator

(ix) Dye

(x) Sulphuric acid

(xi) DNS

(xii) Phenol red indicator

3.3. CHEMICAL ANALYSIS OF THE PULP

3.3.1 DETERMINATION OF ASH

Ash was determined by holding a clean flat bottom silica dish in a lot bunsen-burner flame for one minute after washing with clean water, it was then transferred to a desicator, cooled and weighed W_1 – 5g of ample was taken into the dish and weighed W_2 so that weigh of the sample would be W_2-W_1. Silica dish and sample was then heated on the heating mantle in a fume cupboard until smoke ceased. It was then transferred into a muffle furnace heated 500^0c. Heating continued until all the carbon were burnt. Silica dish was taken out immediately and placed inside a desicator cooled and weighed W_3. The ash content was calculates as:

$$\text{Ash \%} = \frac{W_3 - W_1}{W_2 - W_1} x100$$

The proportion of the sample which got burnt is organic matter and is thus calculated:

$$\text{Organic matter \%} = \frac{W_2 - W_3}{W_2 - W_1} x100$$

3.3.2 FAT OR LIPID DETERMINATION

FAT was determined base on About 5g of the sample was put on a thimble of known weight W1. The weight of the sample inside the thimble was measured W_2. The thimble and the sample in it were placed inside a soxhlet extractor. 300ml of normal hexane was used as the organic solvent, and dissolved lipid continued to fall back into the flask. The extraction continued for 6hrs.

The thimble and content were then removed, cooled in a desicator and weighed W_3.

$$\text{Calcu. Lipid \%} = \frac{W_2 - W_3}{W_2 - W_1} x100$$

3.3.3 DETERMINATION OF CRUDE PROTEIN

The semi-micro Kjeldehl method was used.

2.5g of the sample was weighed into a clean dry Kjeldehl flask. 6ml of sulphuric acid, mixed catalyst and few glass bios where added, the sample was carefully digested over an electric heater with low flame until frothing subsides and then at higher temperature until the content were cleared. Digestion continued for 5hours after which the heat was put off. The tube was emptied into a measuring cylinder after cooling but leaving the glass bids. The solution was then increased to 100ml with distilled water. 10ml of 4% NaoH solution was mixed with 10ml of the digest and then transferred to Markhan semi-micro nitrogen still. Steam distil, ammonia was liberated into 5ml boric acid solution containing 4 drops of mixed indicator taken in the conical flask, solution turned to green and distillation continued for 2 more minutes. Distillate was removed and then titrated with standard hydrochloric acid, the end point reached when the indicator changed from green through grey to definite pink, and the amount of acid consumed was noted.

$$\text{Nitrogen \% in the sample} = \frac{\text{corrected litre (ml)}}{10 \text{ x sample weight (g)}}$$

$\therefore$ Crude protein content % = 6.5 x Nitrogen %

3.3.4 DETERMINATION OF CARBOHYDRATE

⟶ [100- (PROTEIN + ASH + LIPIDS]

This gives the amount of carbohydrate present in the sample.

3.3.5 DETERMINATION OF REDUCING SUGAR

Reducing sugar was determined by the most widely used (DNS)) reagent method.

DNS reagent was prepared by dissolving with warm 10g 3.5 DNS in 200ml 2M-NaoH, also 300g of Rochelle. Salt was dissolved in 500ml of water, these two solutions were mixed with constant stirring and diluted to 1 litre.

Sugar solution was prepared by dissolving 0.2g of conc.sample in 100ml of distilled water and hydrolysed and unhydrolysed sugar were prepared as follows:

(A) Hydrolysed sugar: 5ml of sugar solution was mixed with 1ml of 2moles HCL and heated in a boiling water bath for 30minutes. It was then cooled and 1ml of 2mole NaoH was added, the mixture was then diluted to 10ml and mixed.

(B) Unhydrolysed sugar: The following tubes were set

	Standards				Unknown
Table number	1	2	3	4	5
	Blanks	S1	S2	S3	
5M glucose / mfructose	0.0	0.4	0.8	1.2	0.0
Umoles sugar present	0.0	2UMF	4UMF	6UMF	
Sugar solution	0.0	0.0	0.0	0.0	0.2
Water	2.5	2.1	1.7	1.3	2.3
DNS	2.0	2.0	2.0	2.0	2.0

The mixture inside the test tubes were mixed well and heated on a boiling water bath for exactly 5minutes, cooled in a beaker of tap water and diluted to 20ml with distilled water. The absorbance was noted down and the total sugar content was calculated.

The process was repeated for the hydrolysed sugar solution prepared M(A).

3.4 PRODUCTION PROCESS

3.4.1 PREPARATION AND PROCEDURE

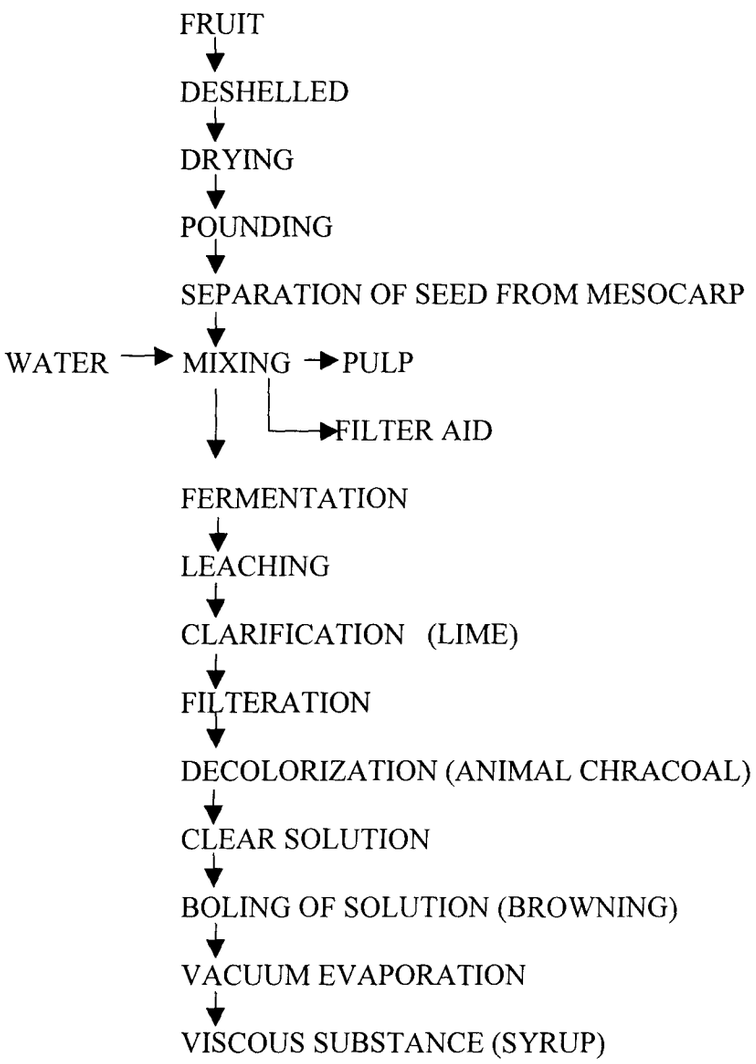
The method used in the initial preparation was based on the local method commonly employed in Kaduna villages. The sample were obtained from a yellow dry powdery pulp.

The pulp was first subjected to a mechanical action that is deshelling by hand. The separation of the mesocap from the seed was carried with the aid of the mortar after a second drying process.

3.4.2 PROCEDURE

1. 750g of the collected dry pulp was mixed with 400m of water.
2. 200g of guinea corn shaf in the semi-solid form was used as filter aid.
3. The mixture was then fermented under the sun for 12 to 15 hours. The fermented mixture was stirred and a container with a perforated base was used to leach out the soluble part of the fermented mixture.
4. A continuous collection of the product was achieved at a rate of 3g per minute
5. The resulting aqueous mixture was now taken to the laboratory where it is filtered and heated.
6. CaoH (lime) is added to precipitate non sugar and neutralize organic acids
7. The resulting scum was removed and animal charcoal was used to decolourize the juice.
8. The juice is then concentrated and evaporated under vacuum to produce a viscous liquid syrup which is our sweetener.
9. 50m of the aqueous mixture at 50^0c is then poured into the polarimetre tube to determine the degree and direction of the rotation of the plane of polarized light of the sweetener.
10. The refractive index of the refined sweetener solution was also measured with a refractometer.

FLOWSHEET FOR PREPARATION OF SWEETENERS



3.5 ANALYSIS OF THE PRODUCT

3.5.1 (i) Determination of fat

(ii) Determination of protein

(iii) Determination of reducing sugar

(iv) Determination of carbohydrate

(v) Determination of ash

All these tests were carried out using the same methods as the ones used for the syrups before production of the product.

The PH of the pulp and the product were tested. Using PH meter (KENT ECL 7020 England) .

CHAPTER FOUR

4.0 DISCUSSION OF RESULTS

4.1 RESULTS

TABLE 4.1

EXPERIMENTAL DATA OBTAINED IN EXPERIMENT

SAMPLES	A	B	C
Weight of aqueous solution (gm)	100	100	100
PH of raw liqour	2.75	2.75	2.75
Weight of $Ca(oH)_2$ used (gm)	0.25	0.30	0.35
Weight of animal charcoal used (gm)	0	0.5	0.5
Treated melt PH	3.65	3.90	3.96
Boiling temperatures (^{0}c)	50	45	35
Weight of sweeteners obtained (gm)	4.5	4.0	3.5

4.1.1 CHARACTERISATION OF THE PULP/ PRODUCT

4.1.2 CHARACTERISATION OF THE PULP

COMPONENT	AMOUNT (g/100g)
Carbohydrate %	81.5
Ash %	3.5
Crude protein %	6.5
Fat %	14.0
Reducing sugar %	70.4

4.1.3 CHARACTERISATION OF THE PRODUCT

COMPONENT	AMOUNT (g/100g)
Carbohydrate %	59.2
Ash %	1.05
Crude protein %	18.0
Fat %	Trace
Reducing sugar %	76.2

4.1.4 CHARACTERISATION OF THE PULP/PRODUCT AS COMPARED TO NUTRITIONAL COMPOSITION OF HONEY

COMPONENT	AMOUNT (g/100g)
Carbohydrate %	30-40
Ash %	0.18
Crude protein %	15-35
Fat %	Trace
Sucrose %	2.5
Water%	17.6
Reducing sugar %	71.9

Data from contessi A (1983) Le Api.

TABLE 4.2

ANALYSIS OF DATAS CONTAINED IN TABLE 4.1

EXP	Yield %	Amount of Ca(OH)2 used in gms	Weight of aqueous solution in (gm)	Weight of sweeteners obtained	Refractometer	Polarimeter	Purity
A	9.0	0.25	100	4.5	1.68	94	55.9
B	8.0	0.30	100	4.0	1.67	94	56.2
C	7.0	0.35	100	3.5	1.67	94	56.6

4.2 DISCUSSION OF RESULTS

4.2.1 ANALYSIS OF THE PULP

During the experiment on the pulp it was discovered that the pulp contained a very high percentage of carbohydrate which was about 81%. Another component that was also very high on the scale was the amount of reducing sugar, which was about 70%. The other components like ash content, crude protein and lipid content were also determined and they conformed appreciably well to standards.

4.2.2 ANALYSIS OF THE PRODUCT

Simple inspection of the liquor clarified with lime supplied ample evidence not only of colouration (browning) which reappears after a certain of about 30-50^{0}c, but also of lack of crystallization of the clarified liquor. The lack of crystallization was due to the effect of excessive invert sugar which hinders crystallization of any kind.

From table 4.1 the product of the first run was observed to be brown, though it was not decolourized, while those of run 2 and run 3 were decolourized with the colouration returning at a temperature range of 30-50^{0}c. From the first run we can deduced that the weight of the sweetener obtained was higher than those of run 2 and 3 respectively, and it was also observed that there were sediment in run 1 making the product look rough.

The 2nd and the 3rd runs, as observed from the table, yielded almost the same result except for the amount of calcium hydroxide used in run 3 which was 0.5g and at reduced temperature. It was also vacuum dried. These differences led to run 3 being the most viscous among the products. The weight of the products differ accordingly: run 1 (4.5g), run 2 (4.0g), run 3 (3.5g).

From the analysis of the data obtained in table 4.1, weight of the sweetener decreased with increase impurity.

Result of the polarimetric analysis showed that the samples at a concentration of 1g per 20ml of water were levorotatory (-) ie the angle of rotation about the plane of polarized light was anticlockwise.

From the characterisation carried out on the pulp and that of the product obtained, it was observed that nutritional composition conformed well to that of honey.

One major component in the pulp and also the product was the reducing which actually was 70.4% in the pulp and 76.2% (gm per 100g) in the product and that of honey from literatures is 71.9% (g per 100g).

This observed increase in the level of reducing sugar is due to an enzymatic action of the enzyme invertase which catalyses the conversion of sucrose to fructose and glucose. It can be also be observed that the boiling time also has an effect on the colour of the refined sweetener obtained, because some compounds in the locust bean pulp which are normally responsible for the colouration becomes more prominent under the influence of long time boiling.

Also from the table, the percentage yields were 9.0, 8.0, 7.0 percent respectively. It could be deduced that increase in clarification resulted in better quality of the product. The purity also increased with increase in clarification. The percentage purity were 55.9, 56.2, 56.6 respectively.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

It was observed that at different temperature 50^0, 45^0 and 35^0c with the same concentration of clarifying agents for run 2 and 3 the same amount of precipitate was observed but at different rates, with the rates increasing in temperature.

The production of locust bean sweetener was investigated and it can be concluded that for a quality yield of sweetener to be obtained from a given locust bean pulp there is need for proper clarification and a short boiling time.

The best quality sweetener was obtained in the third experiment, while the least quality was obtained in the first experiment.

The highest percentage purity of 56.6 percent was also obtained at the third experiment.

5.2 RECOMMENDATION

The production of locust bean sweetener offers a method to utilize locust bean pulp as a form of sweetener. Otherwise the locust bean are inedible. The locust bean sweetener can successfully be absorbed into the sweeteners shelf and it also serves as a source of calorie. It can easily be substituted for honey and even any of the various natural sweeteners.

In order to increase the acceptability of the locust bean as a source of sweetening agent, it is essential to improve the production process and present the product in the market. Besides more research is needed to develop the production of the locust bean sweetener to the same standard as sorghum and corn syrups. Future research should also be aimed at translating the research knowledge into developing small scale technology appropriate for rural areas. Besides, better clarifying agent like activated carbon could enhance the production of quality locust bean sweetener. It is also necessary to study other parameters like crystallization which will help in better marketing of the product.

REFERENCES

(1). B. Shreve Norris (1977). Chemical process industries.

(2) R.S Shallenberger (1982). Advanced sugar chemistry and principle of sugar stereochemistry.

(3) H.G Baker Plants and Civilization (3rd edition).

(4) Spencer Meade. Cane sugar handbook (9th edition).

(5) Horace I. Sipple and Kristen W. Mcnutt. Sugar in nutrition.

(6) Anna Lewington. Plants for people.

(7) J.R. Steward and P.J Towse. Chemical Technology in Africa.

(8) Bishop Carter. Crop science and food production.

(9) Anita Tull (1986). Food and Nutrition.

(10) Helen Andrews Guthrie. Introduction to Nutrition (4th edition).

(11) Richardson & Coulson Vol (II). Particle technology and separation process.

(12) N.R Reddy PHD et al (1986). Legume based fermented foods.

(13) TNO (1991). Focus on artificial sweeteners vol 8.

(14) Hough CAM, Parker KJ and Vlitos AJ (1981). Development in Sweeteners.

(15) Marie S. and Piggot J.R (1991). Handbook on sweeteners.

(16) Johnson J.M and Harris CH (1989). Cereal chemistry.

(17) Lee CK (1987). The chemistry and biochemistry of the sweetness of sugars Advances in carbohydrate and biochemistry PP 45: 199-351.

REFERENCES

(1). B. Shreve Norris (1977). Chemical process industries.

(2) R.S Shallenberger (1982). Advanced sugar chemistry and principle of sugar stereochemistry.

(3) H.G Baker (1978) Plants and Civilization (3rd edition PP. 87-94)

(4) Spencer Meade (1980). Cane sugar handbook (9th edition PP. 39-42)

(5) Horace I. Sipple and Kristen W. Mcnutt (1980) . Sugar in nutrition PP

(6) Anna Lewington (1990). Plants for people PP.77-80

(7) J.R. Steward and P.J Towse (1984). Chemical Technology in Africa.PP. 99

(8) Bishop Carter (1986). Crop science and food production PP 312-327.

(9) Anita Tull (1986). Food and Nutrition. PP 90-112

(10) Helen Andrews Guthrie (1986). Introduction to Nutrition (4th edition PP.155).

(11) Richardson & Coulson Vol (II). Particle technology and separation process.

(12) N.R Reddy PHD et al (1986). Legume based fermented foods. PP. 245-271

(13) TNO (1991). Focus on artificial sweeteners vol 8.

(14) Hough CAM, Parker KJ and Vlitos AJ (1981).Development in Sweeteners. PP. 82-99

(15) Marie S. and Piggot J.R (1991). Handbook on sweeteners.PP.24-60

(16) Johnson J.M and Harris CH (1989). Cereal chemistry. PP.76, 79

(17) Lee CK (1987). The chemistry and biochemistry of the sweetness of sugars Advances in carbohydrate and biochemistry PP 45: 199-351.