# **PRODUCTION AND REFINING OF SHEA NUT OIL**

## BY

# MUSA ASHIFA TORI (98/7074)

# DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY FEDERAL UNIVERSITY OF TECHNOLOGY MINNA.

# NOVEMBER, 2004.

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# DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY FEDERAL UNIVERSITY OF TECHNOLOGY MINNA.

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

NOVEMBER, 2004.

## **CERTIFICATION**

This is to certify that this research project "production and refining of sheanut oil" is the original work of Musa Ashifa Tori (98/7074) and has been prepared in accordance with the regulations governing projects in chemical engineering department.

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Engr. Mohammed Alhassan Project supervisor

Dr. F. Aberuagba H.O.D.

External examiner

23/11/004 Date

Date

Date

## DECLARATION

I, Musa Ashifa Tori declares that the this research work was carried out to the best of my ability under close supervision of Engr. Mohammed Alhassan.

Musa Ashifa Tori.

Date.

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## **DEDICATION**

I dedicate this work to Almighty ALLAH who has spared me to date, to my late parents Mr&Mrs M.T Mahamud. May their souls rest in peace (amin). To my little hommies, Awwal, Saifideen, Ibrahim, Abubakar, Nana and Khadijah.

All praise belong to Allah (SWT), the first and the last, the creator in whose hands lies the soul of every being, the cherisher and sustainer who has guided me through all aspects of life.

A project a heavy undertaking that requires utmost input to achieve set goals. Numerous indeed are those whose contributions yielded the expected result.

My heartfelt gratitude goes to Mr&Mrs Usman Ibrahim (N.E.P.A) whose parental love, care and endless support saw to my success. In this regard, I say thank you very much.

Special thanks goes to my project supervisor Engr. Mohammed Alhassan, whose advice, corrections and wonderful supervision made this work a reality. To my HOD Engr. Dr F Aberuagba, Aunty Lizzy for her motherly care and love, all the lecturers of xcal engineering department who have been very cooperative and helpful through out my five year stay in the dept.

My deep appreciation goes to Group Capt & Mrs A G Sabir, Lt Col & Mrs A Alkali, Lt Col & Mrs A M Dikko, Capt I Usman, SP Mohammed Wali, for there strong words of encouragement and support that gave me the zeal to carry on. May the sky be your limits in your various professions (amen). Not forgetting the entire staff of Islamic Affairs Depart. HQ 1Mech. Infantry Division Kaduna.

Worthy of remembrance at this hour are good friends who stood firm and gave me the encouragement to push forward. They are Festus Oyelowo (festooloye), Helenatu, Muri, Danbaba (nepa), Holyman, Anthrax, Pa (the great), to mention a few. Not forgetting colleagues such von Smith, Kolo, Elsud, Tovi, Arewa connection, O'oduwa connection, without your friendship life could have been unbearable.

## ABSTRACT

The extraction of shea nut oil from shea nut seed was carried out using hexane as suitable solvent. The oil obtained was milky in color with a pleasing odor. The produced oil was degummed, neutralized, decolorized and deodorized to make it suitable for use. The saponification value, iodine value, FFA, acid content, specific gravity and oil contents were gotten to be 179, 66.20, 1.57, 3.15, 1.13 and 58% respectively. These shows that the oil is a good raw material for soap manufacturing industries, it has a long shell life, and of great yield.

In designing a prototype extractor, an expected percentage yield of oil to be produced can be estimated from the graph of weight, time and concentration against percentage of oil produced (graph A and B).

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#### **CHAPTER ONE**

#### **I.0 INTRODUCTION**

#### 1.1 BACKGROUND OF STUDY

A fat that is liquid at room temperature is called oil. That, which is solid or semi-solid, is a fat. "Fats and oils irrespective of how they are gotten represents the highest source of energy per unit weight that man an consume".(weiss1983).

Shea nut oil is a fat extracted from shea nuts obtained from shea nut tree (butyrospernum parkii), which is of great economic importance and has variety of uses (edible and non-edible). The tree grows wild in the drier part of equatorial belt. It also exists naturally in the savannah zones. (Agubas 1999).

Shea butter has various domestic and industrial uses contributing significantly as a substitute for edible oils. The oil is a good raw material for soaps production, medical ointment etc. the most widely used method of producing the oil is the local production method,(the cold and hot water extraction methods). Recent advancement embraces extracting the oil using solvent extraction method, which is more efficient. In assessment of the fat, greater emphasis will be laid on the oil color and odor as past

Observations have shown that the fat is usually neglected cause of its objectionable odor.

The difference between fats and oil as mentioned earlier is the ability of being a solid or liquid at room temperature. It was observed that the more hydrogen contained in the oil molecule the thicker the oil becomes. "shea nut oil forms one of the major export commodities in the first and second quarter of the 20<sup>th</sup> century"(keay 1989). In Nigeria for instance between 1909 and 1933 record of export trade shows that shea oil production contributed immensely to the Nigerian economy. But between 1993and 1997 the export value has totally being forgotten, because the oil production was left in the has of the local producers alone.

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#### **1.2 OBJECTIVE OF STUDY**

The main objective of this work is to:

- Extract shea oil from shea nut using the available production methods (continuous extraction and the local means).
- Determine the chemical and physical properties of the oil produced using suitable analytical methods.
- To suggest suitable methods of extracting the oil from its nuts.

## **1.3 SCOPE OF STUDY**

Shea butter oil was first extracted using the solvent extraction method with water as the solvent. In this work the same method of extraction was employed but with the use of hexane as solvent. This method gave the highest yield with minimum heat consumption. However effect of technological characteristics such as concentration, temperature, particle size, and time on the rate of extraction was also looked

Into and the characteristics of the oil extracted was also carried.

## **1.4 RELEVANCE OF THE STUDY**

The demand for qualitative edible oils in our society has continued to increase therefore the need to bring to lime light the gradually disappearing shea nut oil is necessary. Shea butter oil contains high percentage of essential nutrients that are useful domestically and industrially. But being a fat at room temperature has lead to its neglect. This work is meant to produce and refine the oil, also to eliminate odor and make it more suitable for use.

There is no doubt that the out come of this finding shall be of immense importance in designing and fabricating a prototype extractor for commercial use.

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#### **CHAPTER TWO**

#### 2.0 LITERATURE REVIEW

#### 2.1 HISTORICAL BACKGROUND OF SHEA NUT TREE

Shea nut tree is a deciduous tree 40m high with leaves clustered at the end of branches. It is a perennial tropical tree (parkii specie) named after the scotish surgeon who discovered the tree during his travels to the interior part of Africa in 1970.(DM DALZIEL 1979).

The shea nut tree grows wild in savannah region of West Africa particularly in northern Nigeria and Ghana. Though the tree has been put into several traditional uses but no attempt has been made to propagate the plant and make research on its validity for food and other uses until after its discovery by Mungo park (TA PHILIPS1989).

#### 2.2 CULTIVATION

The habitat of the tree is the savannah woodland. It occurs widely spread in nature therefore it is not abundantly cultivated. But in the areas where the tree is sparsely populated attempt has been made to cultivate and protect the plant. Experimental planting of the tree has shown that fresh seedlings develop long taproots, which renders transplanting of the tree difficult or risky. The planted seed germinates readily but the tree grows slowly (RWJ KEAY 1989).

## 2.3 MATURITY

The tree becomes fully matured when up to about 40m high. At this stage the bark of the tree becomes dark grey, rough and square pieces containing abundant white latex. The leaves are clustered at the end of stout twigs up to 9 (nine ) inches long and three and half inches wide. It is made up of about thirt (30) pairs of lateral nerves spreading almost at right angles.

Flowering of the tree varies from place to place. In some places it starts flowering around January to February. In some other places flowering occurs from December to March. The flowers are of fragrance, long stalked and clustered at the end of leafless twigs. The sepals are covered with pinkish brown hair and the petals are white. Fruiting begins around May to august in Nigeria when the tree is about 12-15 years of age and remains in full bearing of fruits for about 20-25 years. The fruits are yellowish and ellipsoid about two inches long with one, two, or three seeds sometimes. The fruits when fully ripe fall to the ground. The seed is ovoid with hard bonny testa and shield shaped scar nearly as long as the seed.

## 2.4 VARIETIES

Though different varieties of shea nut tree exist based on the district in which they are found, three main varieties are recognized. They are:

- Butyrospernum parkii, variety magnifolum. Usually found in upper Senegal and middle Niger to the shari basin.
- Butyrospernum parkii, variety poissonni. Found predominantly in yhe northern part of Nigeria.
- Butyrospernum parkii, variety nilotrains. Found in baharel ghazel.(keay 1989).

Of the above types of varieties, the most suitable for the production of shea butter oil is the variety poissonni, which is found predominantly in the northern part of Nigeria.

### 2.5 NUT STORAGE

The kernels are usually stored in dry places before processing begins. They are usually left in well ventilated rooms or parked in sacks under

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conducive temperature. The embryo of the seed is usually killed by either fermentation or by boiling to prevent the seed from germinating. Improper storage will affect the level of oil produced.

## 2.6 USES OF SHEA NUT OIL

Apart from being used as cooking oil, it is widely used for other purposes, that is the food purposes. They include:

- The residue after extraction of butter can be used animal or poultry feed preparation. (Adgizi 1999).
- As an illuminant: The oil having good combustion characteristics is used as a substitute for bush lamps and found to be very economical (Burseglove 1984).
- As medical ointment: The fat is known to be used in treatment of fractures, dislocation, swelling and proved to be highly effective (Okafor 1980).
- Women for hair dressing and plaiting also use the oil.
- Industrially the fat is used in the manufacture of soaps, candles and cosmetics.
- It has an export market value and can be used as a means of foreign exchange earnings. (Momodu 1987).

The following tables from FAO statistics shows that shea nut oil has a foreign exchange value, which is not being maximized in Nigeria.

## Table 4.8

#### Shea nut exports 1993-1998

1993	1994	1995	1996
340	2590	1520	5846
1070	2223	1400 ·	1400
1601	1973	793	793
137	764	788	1274
	340 1070 1601	340     2590       1070     2223       1601     1973	340         2590         1520           1070         2223         1400         .           1601         1973         793         .

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B. faso	500	500	847	847
Nigeria		1500		-
Mali	150	150	150	150
Uk		45	37	9
Others	6	9	38	19
Total	3,523	9,382	6,733	10,329

# Source FAO statistics

Table 4.9

# Shea nut production 1994-1997

Country / yr	1994	1995	1996	1997
Benin	15500	15000	15000	15000
B faso	70100	75700	70000	70000
Cote d,voir	19785	20000	20000	20000
Ghana	57000	56000	55000	55000
Mali	85000	85000	85000	85000
Nigeria	353000	384000	345000	385000
Togo	7000	8520	2504	6500

## **Source FAO statistics**

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#### 2.7 FAT PRODUCTION

Vegetable oils are produced and processed from oil bearing seeds either for edible or non-edible purposes. Within the decade to early eighties, production and consumption of oil is on the increase and almost twice that of sixties. (weiss 1983). In the production of shea nut fat, two (2) methods were employed. The first being the local means of producing the fat and the second is a laboratory work known as solvent extraction process. (Continuous extraction method).

### 2.7.1 LOCAL METHOD

The processes involved in this method are boiling the nut, the boiled nuts are then sun dried, cracked and further dried to increase the shell life and also to reduce the moisture content in the seed. The dried seeds are then pounded to a coarse brown paste, which is now roasted. The roasted nuts are now ground to a greasy mass. Addition of water makes the fat to float on top and can then be collected. (RMRDC 1998).

## 2.7.2 EXTRACTION:

This is a process in which a compound in a liquid mixture is selectively transferred to a liquid in contact with it. Extraction also involves transfer of solute from one solvent to the other. There are several means trough which oil can be extracted. Some of the methods include:

- Hydraulic pressing: This is a process by which means oil is suppressed by hydraulic pressing from an oil-bearing seed.
- Continuous screw: This is a process through which oil bearing material is squeezed through tapering oil let. Compared to hydraulic press, this method requires twice as much energy and wears out more quickly.

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• Solvent extraction: This is a means of extraction by the use of suitable solvent.

## 2.7.2.1 SOLVENT EXTRACTION METHOD

This method is based on counter current flow of solvent and oilbearing material in the extraction vessel. Solid s running in one direction is washed or leached by solvent passing in the other direction. Solvent is used to the best advantage and oil is removed more effectively as the material comes in contact with it.

## 2.7.2.2 ADVANTAGES OF SOLVENT EXTRACTION

- It is the most effective means of oil recovery from oil-bearing seed.
- It reduces residual oil in oil-bearing seeds to a relatively very low percentage.
- It yields oil at high quality.

## 2.7.2.3 DISADVANTAGES OF SOLVENT EXTRACTION

- The solvents used for the extraction process are usually very expensive.
- They are made of glass material and usually very fragile.
- There is also the problem of selecting suitable solvent to be used for the extraction.
- Except when non-flammable solvents are used there is always a posing danger of fire explosion.
- Solvent extraction equipments are relatively very expensive.

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#### 2.7.2.4 FACTORS AFFECTING RATE OF EXTRACTION

The selection of equipment for an extraction process will be influenced by the factors, which limit the extraction rate. These factors are discussed bellow.

• SOLVENT: solvent affect the rate of extraction since of its properties like viscosity, density, vapors pressure and so on vary from solvent to solvent. In this research work the solvent used in the extraction of oil from shea nut was HEXANE and it has the following properties.

#### **CHEMICAL PROPERTIES:**

- 1) Highly flammable
- 2) Can be explosive at high temperatures
- 3) Reacts vigorously with oxidizing materials.
- 4) Attacks rubber, plastics, skin, eye, and so on.
- 5) Slightly toxic.
- 6) No self-reactivity.

## **PHYSICAL PROPERTIES:**

1) Color:	Colorless
2) Solubility:	Slightly soluble
3) S. Gravity:	0.659
4) V. Density:	2.97
5) B. Point:	60
6) M. Weight	86.17

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7) Easily evaporates.

SOLVENT SELECTION: In other to be able to make a good juice in the solvent to be used, some of its characteristics are supposed to be known to aid in the selection of suitable solvent. These characteristics are:

1) Purity: the solvent to be used must be pure as impurity to some level will greatly affect oil production yield.

2) Solvency: This is the ability of the solvent to dissolve a wide variety of the substance. There a solvent with a high solvency power should chosen.

3) Relative volatility: The solvent to be used must have the ability to convert to gas or vapor quickly. Hence recovery of the solvent is by distillation.

4) Color: The color should be distinct from that of the solute so that the solvent can easily be distinguished from the oil.

5) Toxicity: The amount of toxic material contained in the solvent should be as low as possible.

6) Availability: The solvent to be used must be readily available.

7) Inflammability: This is the ability at which a solvent can be set on fire and burns quickly. All liquids with flash point below 32.2% are flammable. In other to avoid explosion, solvents that are inflammable should be chosen.

- PARTICLE SIZE: particle size also affects the rate of extraction. The smaller the particle size the greater the interfacial area between the solid and the solvent and hence the higher the rate of transfer of material. Smaller size also means the solvent have smaller distance to travel through the solid and hence an increase in the rate of extraction.
- TIME: it was discovered that the rate of extraction increases initially as the time increases and later decreases as the extraction proceeds.
- **TEMPERATURE:** increase in temperature lowers viscosity of oil and solvents, which in turn increases diffusion coefficient. Care must be taken in choosing extraction temperature so as to prevent explosion especially when dealing with flammable solvent.

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- FLAKE THICKNESS: like the particle size flake thickness is directly proportional to the rate of extraction. This is because as the flake thickness decreases cell wall thickness also decreases resulting into higher diffusion.
- AGITATION OF FLUID: this is important because it increases diffusion if solvent and increases the transfer of material from surface of the particle to the bulk of the solution. Agitation also prevents sedimentation and more effective use is made of the interfacial surface.
- MOISTURE CONTENT: This affect the rate of extraction as a decrease in moisture content means a high yield of oil to be produced.

The extraction of oil from shea nut is basically a physical change. But chemical conversion may be required in refining and other processing of the fat. The oil contained in shea nut is maintained in small tough cell walls. During extraction the solvent migrate to the pore in other to extract the oil. In designing a large scale solvent extraction apparatus, particle size distribution should be considered to allow for optimum oil extraction.

## 2.8 CHARACTERISTICS OF PRODUCED FAT

The following are the basic characteristic for most fats and oil:

- l Iodine value
- 2 Saponification value
- 3 Refractive index
- 4 Specific gravity
- 5 Acid value
- 6 Free fatty acid
- 7 Boiling point
- 8 Melting point

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2.8.1 Iodine value: The iodine value of oil is related to its unsaturation. It is a measure of unsaturated acid present. The test measures the amount of iodine that can be absorbed by the acid. One major property of unsaturated compounds is the presence of double bonds and the ability to undergo addition reaction especially with halogens. The higher the iodine value, the greater the unsaturation and the greater the liquidity.

2.8.2 Saponification value: In this process ester is saponified and then back titrated to determine the mean molecular weight of the glycerol molecules. It is a measure of the mean molecular weight of fatty acid present in the fat or oil. This is also the hydrolysis of triglycerides into glycerol and potassium salts of fatty acids, using a solution of potassium hydroxide in alcohol. Saponification value gives the actual amount of alkali required by a fat or oil. The lower the molecular weight the greater the Saponification value.

2.8.3 Refractive index: This is measured by the angle through which light is bent when passing through a thin film of melted fat. The index of each fat falls within a narrow range and can be used in checking the purity the oil. It is temperature dependent and usually measured at 40.A temperature at which most fats are liquid.

**2.8.4** Specific gravity: This is defined as the density of substance relative to that of water. It compares the sample relative to water.

2.8.5 Acid value: this is defined as the number of potassium hydroxide or sodium hydroxide that is required to neutralize the free fatty acid in one gun of the sample. This result is usually expressed in percentage of free fatty acid.

**2.8.6** Free fatty acid: this is the amount of sodium hydroxide required to neutralize the free acid in one gram of the sample. It can also be expressed as percentage.

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**2.8.7 Boiling point**: this is defined as the temperature at which the fat is observed to start boiling.

## 2.9 REFINING OF THE PRODUCED FAT

When the fat has been produced it is necessary to refine it to make it suitable for consumption. Some of the sequences of operation involved in refining are:

- 1 De-acidification
- 2 Bleaching
- 3 De-gumming

4 De-odorisation

5 Cleaning

2.9.1 De-acidification: The newly produced fat is acidic; therefore deacidification is carried by the use of bases. This is the treatment of fat with dilute caustic soda. This effects the removal of fatty acid present. Soaps are formed by the reaction with the free acids as well as by Saponification of a portion of the oil. The resulting soaps are then removed by settling followed by washing with warm water.

2.9.2 Bleaching: This is mostly referred to as de-colorization of the fat. It is a means whereby colored bodies present in the fat are removed. Fats and oil mostly contains coloring matter as natural constituents. Such pigments are carotenes or chlorophyll, which gives the fat its color. Some pigments can be made colorless simply by oxidation. But this affects glyceroids and destroys natural antioxidants. it is therefore not a good method to be used.

Thermal bleaching: This is another means of removing color from fats. In this process the fat is heated to a very high temperature and some carotene pigments becomes colorless. However the high temperatures chars some of the pigment elements in the oil and are very difficult to remove. This method is also not advisable to be used in highly colored fats.

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Adsorptive bleaching: This process is widely accepted for most edible oils. It is done by the use of bleaching earth which has large surface that has affinity for pigment types. Color is removed without damage to the oils. Bleaching is usually followed by filtration, with the filter being an interwoven metal structure.

**2.9.3 De-gumming**: This is a process whereby gum that is formed during fat production is eliminated. The gum was formed as a result of the presence thick white latex present in the nut.

**2.9.4 De-odorisation**: This process removes any unpleasing smells that may be attached to the oil. It makes the oil widely accepted for consumption purposes.

2.9.4 Cleaning: This is the final process of making the oil ready for sale.

# CHAPTER THREE (EXPERIMENT**#**

## 3.1 Materials used:

The materials used in carrying out the experiment involve the use of apparatus and reagents.

## 3.1.1 Apparatus used: these include,

- Soxhlet extractor
- Measuring cylinder
- Conical flask
- Round bottom flask
- Burette
- Pipette
- Beaker
- Heating plate
- Density bottle
- Electric oven
- Weighing balance
- Filter paper

## 3.1.2 Reagents used: these include,

- Hexane
- Hydrochloric acid
- Potassium iodide
- Ethanol
- Starch solution
- Water
- Indicator
- Potassium hydroxide
- Sulphuric acid
- Shea butter cake

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#### 3.2 Material sample used:

The material (shea butter nuts) used for this research work was collected at kaiama L.G.A kwara state. While the solvent used was hexane.

## 3.3 METHODOLOGY

In this research work, two methods were used in the extraction of shea nut from the seed. These methods are the water separation method (local method), and the continuous extraction method using the soxhlet apparatus.

The two methods employed involve the preparatory and the extraction stages. The preparatory stages are:

Seed picking: The shea nut seeds usually scattered all over in the bushes are picked up and gathered. The gathered seeds are sun dried.

**Pretreatment:** The dried shea nut is then decorticated. This is the process whereby the shell of the nut is removed is separated from the kernel. The kernel is now washed to remove impurities.

**Drying:** The kernels are then largely dried in the sun to reduce the moisture content of the seeds. In this research work the drying of the kernels took place in the electric oven. The weight of kernels before and after drying was noted.

**Crushing:** The kernels were then crushed using a crusher or the local mortar and pestle to obtain fairly fine grains with less coarse particles.

Grinding: The fines grains are now ground using a grinder until a fine oily paste is formed.

### 3.3.1 Water separation method:

The fine oily paste is collected into a large bowl and water is added to it.

Treading: This is the process whereby mixing of the paste is done either with hand or a stick. When fully treaded the fat is usually seen as a scum on the top of the paste.

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Washing: The fat that is that is already formed on the top is then washed with warm water and treading continuous. The scum also continuous to increase.

Fat separation: when the fat has fully risen it is then separated into another container, leaving the impurities at the bottom.

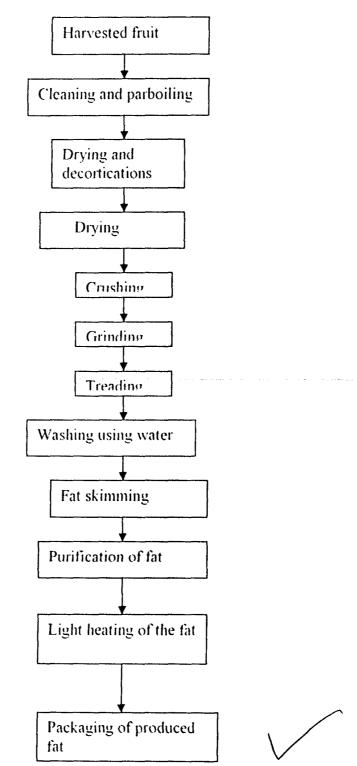
Purification: The collected scum is now further heated for about 2-3 hours to evaporate the water content in the scum. Other impurities that might have escaped with the scum are also eliminated.

Drying: the purified oil is now heated lightly for about 10-30 minutes. This process improves the taste and reduce the odor of the fat. The oil is now left to cool and solidify ready for use.

Packaging: The oil is packaged into different containers based on the purpose of which it is intended. At temperatures between 20-30°C the oil begins to solidify. The fat also assumes the shape of the container into which it is put.

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#### 3.3.2 Continuous extraction method:

The second method employs the use of the following,

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- Soxhlet extractor
- Condenser
- Heating mantle
- Thimble

- Round bottom flask
- Wool

#### **Procedure:**

The soxhlet extractor with the reflux condenser cooling the solvent vapor, and converting it to liquid. A known weight of the sample was put into a thimble and covered with wool. The thimble was then placed into the extraction column of the extractor. A known volume of solvent was poured into around bottom flask and placed on a heating mantle. The heater was put on. The solvent boiled gently and recycled continuously. The set up was left for about 2-3 hours.

The solvent was then evaporated to recover the oil produced. The experiment was carried out keeping time constant and varying weight, also keeping weight constant and varying time. The oil content of the seed was calculated using the formula:

Percentage yield = <u>Weight of oil extracted</u> × 100 Weight of sample

$$= \frac{W2-W3}{W1} \times 100$$

Where, W2 = Weight before extraction W3 = Weight after extraction W1 = Weight of sample.

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In the experiment, the effect of time, particle size and concentration on the rate Of oil produced was determined.

### **3.3.3.1 Determination of saponification value.**

3g of the oil was placed in a conical flask. Then 250 ml of sodium hydroxide was added to the flask. The content was placed on a heater for about 30 mins, with occasional shaking of the flask.

The solution was then titrated against a standard 0.5m hydrochloric acid using phenolphthalein as indicator. Blank titration was also carried out in the same way without using the oil. The saponification value was calculated using the formula,

$$SV = \underline{b-a (28.05)}{M}$$

Where b = the volume of acid for blank test.

a = the volume of acid used with oil.M= mass of the oil used.

### **3.3.3.2 Determination of iodine value:**

2 dried reagent bottles of 250ml capacity were obtained. 0.5g of oil was inserted into one of them, about 5ml tetrachloromethane was added and the solution mixed with iodine chloride. The bottle and content were allowed to stand in the dark for about an hour.

10ml of potassium iodite was added to the sample with distilled water. The mixture was swirled lightly and then titrated against an aqueous solution of sodium thiosulphate in the presence of starch as indicator. The titration continuous until the black color just disappears. The iodine value was calculated using the formula

Where b = blank titration a = titration with oil

#### 3.3.3.3 Refractive index:

This is the rate of sine of angle of incidence to that of sine of angle of refraction. the oil was rendered optically clear and with the aid of abbe's refractometer the refractive index was read.

## **3.3.3.4 Determination of specific gravity:**

Density bottle was used in calculating the refractive index of the oil. The bottle was weight as W1 then filled with oil; a stopper inserted then weight again as W2. The bottle was emptied, washed, properly rinsed in water then dried. The bottle was now filled with water, excess water cleaned off from the bottle and weight as W3.

The specific gravity was calculated from the formula below.

$$SG = \frac{W2-W1}{W3-W1}$$

Where W2-W1 = density of the oil W3-W1 = density of water

### **3.3.3.5 Determination of acid value:**

This is a means of determining the amount of sodium hydroxide required to neutralize the free fatty acid in the sample. 25ml of ethanol and 25ml of dietyl ether were mixed with an indicator. The mixture was then neutralized with 0.1m sodium hydroxide. 3g of the oil was added to the solution and titrated against 0.1m aqueous sodium hydroxide until a pink color was obtained. The acid value was calculated from the formula,

$$AV =$$
titre value (5.61)  
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## **3.3.3.6** Determination of free fatty acid:

This determines the amount of sodium hydroxide needed to neutralize the free acid in 1g of the sample. It is expressed as a percentage.

> Acid value = 2 (FFA).  $\rightarrow$  FFA = <u>Acid value</u> 2

Some free fatty acid contents of shea nut are shown below.

Oleic acid	70-78%
Palmitic acid	6%
Linoleic acid	6-9%
Stearic acid	8-9%

### **3.3.3.7** Boiling point:

This is the temperature at which the oil is noticed to start boiling. The oil was placed in a beaker and thermometer inserted. The sample was carefully heated using a Bunsen burner the temperature at which boiling started was noted and recorded.

#### **3.4 REFINING PROCESS**

**3.4.1 Degumming:** This the process of removing gum formed alongside fat produced.

**Procedure:** A certain weight of the oil was put into a separating funnel. Distilled water was boiled and added to it. It was shaken for about two mins. The gum was collected along with water at the bottom of the funnel.

**3.4.2** Neutralisation: This is an alkali refining process. The objective is to remove the free fatty acid present in the fat. The type and strength of the alkali influences efficiency of neutralization and the ability of the process to remove other undesirables, i.e. residual phosphate and so on.

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**Procedure:** 0.5m of sodium hydroxide was added to the degummed oil stirred and filtered off to obtain neutralized oil. The minimum amount of alkali required to neutralize the FFA in the oil is gotten from the formula,

 $NAOH = % FFA \times 0.142$ 

Neutralization is very important, as acidic fats are usually very difficult to bleach. And they can also affect the yield of finished product.

3.4.3 Bleaching: The clay sample used was gotten from bosso town and tested to be a kaolinite clay. The clay was dried for 2 hours to eliminate moisture. The clay was then calcinated by further heating. A sieve shaker was used in obtaining different sizes of the clay. For this work,0.2mm and 0.5mm sieve sizes were used.

The sieve samples were then wetted with 0.2m hel and heated to a temperature of 110°C for about two hours. The clay became activated clay.

Procedure: the bleaching was done in a round bottom flask containing the oil. It was placed on a heater and the temperature kept constant. A known mass of the clay was poured into the flask with a contact time of 20 mins. The same result was obtained when activated coal was used with no difference in the color observed.

**3.4.4 Deodorization:** this method was used in removing any odor contained in the fat to make it suitable for use the odor might be as a result of the decomposition of the natural pigment present in the oil. In this work two methods were used in the deodorization process.

- Use of onions
- Use of garlic.

**Procedure:** 50ml each of extracted oil sample were placed in two separate beakers. They were heated. When fully heated 5g of sliced onions were added to one sample and to the other, 5g of sliced garlic. It was further heated and then filtered. The odor of the two contents was observed.

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# CHAPTER FOUR RESULTS OBTAINED.

The results of various experiments conducted on the extracted nut oil are shown in the tables below.

## Table 4.1

## Effect of time on the rate of oil produced.

Initial	weight of	Extraction time	Weight after	Percentage
sampl	e. (g)	(hrs)	extraction (g)	of oil
				extracted
				(%)
2	g	0.5	1.749	13
2	ζζ	1	1.5	25
2	, en la companya de	1.5	1.0	50
2	٤.	2	0.84	58
2	٤٤	2.5	0.836	58.2
2	در	3	0.835	58.22

## Table 4.1B

Initial weight of	Extraction time	Weight after	Percentage of oil
sample. (g)	(hrs)	extraction (g)	extracted (%)
10	1	9.1	10
10	2	8.2	18
10	3	7	30
10	4	5.9	41
10	5	4.7	53
10	6	4.2	58

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## Table 4.2

# Effect of particle size on oil produced.

Initial weight of	Extraction time	Weight after	Percentage of
sample. (g)	(hrs)	extraction (g)	oil extracted (%)
10	2	8.2	18
8	2	6	25
6	2	4.2	30
4	2	2.1	47.5
2	2	0.83	58.9

## Table 4.3

# Effect of concentration

Mass of sample	Sample	Solvent	Percentage
(g).	concentration.	concentration.	of solvent
			used. (%)
11	0.042	0.962	96.2
9	0.035	0.968	96.8
7	0.027	0.975	97.5
5	0.020	0.982	98.2
3	0.012	0.990	99
2	0.008	0.993	99.3
]	0.004	0.996	99.6

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## Table 4.4

## **Bleaching process**

Sieve	Mass of clay	Contact time	Color when	Color when
size(mm)	used (g)		hot	solidified
0.25	2	20 mins	Colorless	Milkish
0.50	2	20 mins	Colorless	Milkish

## Table 4.5

## Deodorization test result.

Property	Garlic	Onions
Color	Changes to brown color	Changes to dark brown
Odor	Pleasant. Groundnut	Odorless
	like odor.	

## Table 4.6

# Results of characteristics of oil produced.

property	Experimental value	Standard value	
Iodine value	66.20	55-71	
Saponification vale	179	170-188.07	
Melting point	35°C	33-38°C	
FFA value	1.57 0.48%	0.5%	
Acid value	3.15	<u>≤6%</u>	
Specific gravity	1.15	1.5	
Refractive index	1.46		
Odor	Pleasant	Dependable	
Oil content	58%	≥45%	
Boiling point	138°C	120-140°C	
Moisture content.	6.6%	≤7%	

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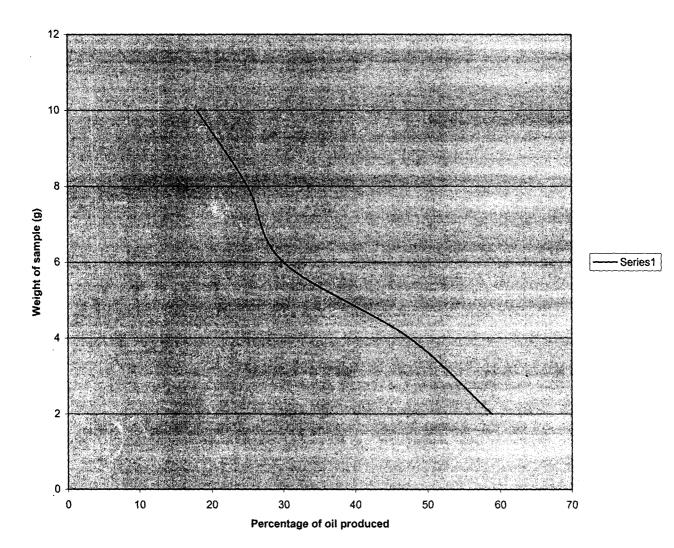
## Table 4.7

# Liquifaction test result.

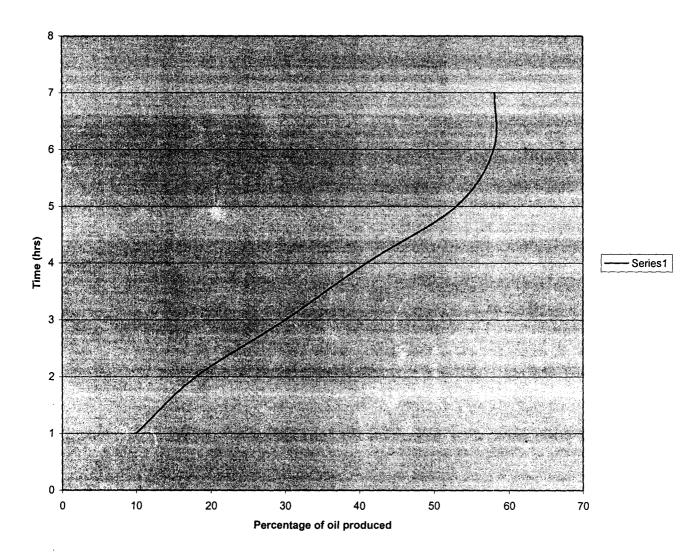
No of days	Pure shea butter	Garlic treated	Onions treated
	oil	sample	sample.
1	Completely solid	Completely	Completely
		liquid	liquid
2	"	"	"
3	"		"
4	"	"	"
5	"	Starts solidifying	"
6	"	"	"
7	"	"	Starts solidifying
8	"	solidified	
9	"	"	solidified

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#### Effect of weight on oil produced



#### Effect of time on oil produced



#### **CHAPTER FIVE**

## **DISCUSSION OF RESULT**

The solvent extraction method of shea nut production is proven to be the most effective with only a disadvantage of cost. The result obtained shows the effect of temperature and particle size on the rate of oil produced. At optimum condition the seed is a good source of oil. A characteristic of the produced shea nut oil was done in other to determine some of its chemical and physical properties. The iodine value obtained was 66.20, saponification value was 179, the acid value was 3.15, and the free fatty acid value was 1.57. the low iodine value shows the seed has a long shell life. The saponification value shows that the oil is a good raw material for the production of soap. The boiling point that was found to be 138°c is slightly higher than that of water. Odor of the produced fat was found to be pleasant.

Refining process was also carried out to purify the fat and make it ready for use. The deodorization process was observed to be a liquifaction process. This is the only means by which the fat can remain a liquid for a number of days. An advantage of the spicies used in carrying out the process is that they do not have any effect on the chemical or physical properties of the fat. As the time increases for a particular weight the amount of oil produced increases until a time when the percentage of oil produced remains constant. The oil content of 58% obtained shows that the shea nut seed has a good oil yield. A prototype reactor can be designed on the basis of these results.

### **CHAPTER SIX**

## CONCLUSION AND RECOMMENDATION

### 6.0 CONCLUSION.

The extraction of shea nut oil was successfully carried out using the soxhlet extractor apparatus. It was seen that the continuous extraction method was more effective but with a disadvantage of cost. In the local method of production, the hot water method was found to be more effective. The saponification value of 179 obtained shows that the fat has high content of fatty acid and therefore a good raw material for the manufacture of soap. The shell life of the seed is long due to its low iodine value of 662. The iodine value obtained also shows that the fat is very reactive. The fat produced is a solid at room temperature with an acid value of 3.15. The refining process was to make the fat edible. The research work done on shea nut oil was generally success. The values obtained in this research work were seen to be in agreement with the set standard. Hence can be used in designing a prototype extractor for shea nut oil.

#### 6.1 **RECOMMENDATION**

I recommend that next student undertaking a research work on shea nut oil should first dry the paste, grind it to obtain different sieve sizes and see which of the sizes will give the highest amount of oil at constant time.

Equipment on the production of various edible oils should be put in place in the chemical engineering lab so that with time F. U. T. chemical engineering department will be producing large quantities of edible oil to serve as a means of revenue generation for the department.

I recommend that the government should impose the law of deforestation as most especially it affects the she nut trees that are constantly being used as fire wood. Or used for construction purposes.

The karite tree should be given mechanical production so as to bring about high quality products and high productivity.

The commercial value of the oil be revisited. That is the shea nut oil should be used once more as a source of foreign exchange since the fat is largely produced in this part of the world.

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## APPENDIX

# SAMPLE CALCULATIONS.

Moisture content:

 $\frac{W2-W1}{W1} \times 100$ Where W2 = Weight of sample W1 = Weight after drying.

Saponification value:

B = blank titration

A = titration with oil.

Blank titration values

Readings	Volume of solvent used (cm)
Initial	00.00
Final	24.10
Difference.	24.10

Titration with oil

Readings	Volume of solvent used (cm)	
Initial	00.00	
Final	05.00	
Difference	05.00	

 $S \cdot V = \underline{b-a (28.05)} \\ M \\ = \underline{24.10 - 05.00 (28.05)} \\ 3 \\ = 179$ 

Iodine value:

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# B = blank titration

# $\Lambda$ = titration with oil

# Blank titration

Reading	Volume of solvent used (cm)		
Initial	00.00		
Final	31.44		
Difference	31.44		
Reading	Volume of solvent used (cm)		
Initial	31.44		
Final	36.78		
Difference	5.34		

 $I . V = \underline{b-a (1.269)}{M}$ 

=<u>26.1 (1.269)</u> 0.5

=66.26

Acid value:

Acid value =  $titre value \times 5.61$ 

Wt

Volume of	solvent	used
(cm)		
00.00		
1.68	•	
1.68		
-	(cm) 00.00 1.68	00.00

Acid value = 1.68(5.61)

Free fatty acid value:

Acid value = 2 × FFA.  

$$\Rightarrow$$
 FFA = Acid value / 2  
 $\therefore$  FFA = 3.15/ 2  
= 1.575

Specific gravity:

 $\frac{\text{Density of substance}}{\text{Density of water}}$  $\rho = M/v$ 

density of substance = 11.3/10=1.13kg/m<sup>3</sup>

density of water =  $1 \text{ kg}/\text{m}^3$ 

$$\Rightarrow S.G = \frac{1.13}{1}$$
$$= 1.13$$

33

# APPENDIX B

# Percentage of oil obtained

Effect of particle size: Let initial weights be = a Let final weights be = b Time t is constant at 2 hrs.

If a = 10gAnd b = 8.2g

Percentage of oil obtained =  $a-b/a \times 100$ 

 $= 10-8.2 \times 100$ 10 = 18%

a = 8g b = 6g % obtained =  $8-6 \times 100$ 8 =25%

a = 6g b = 4.2g% obtained = <u>6-4.2</u> × 100 6 = 30%

a = 4gb = 2.1g

% obtained =  $\underline{4-2} \times 100$ 

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a =2g  
b =1.2g  
% Obtained = 
$$2-1.2 \times 100$$
  
2  
= 57.9%

# Effect of time on oil produced.

Let a = initial weight which is constant. b = final weight c = time.

If a = 10g b = 9g t = 1hr% of oil produced =  $10-9 \times 100$  10= 10%

If b 8.2g

t =2hrs

% of oil produced =  $10-8.2 \times 100$ 

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10 = 18%

If b = 7g

t = 3hrs

% of oil produced =  $10-7 \times 100$ 

35

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If b = 5.9g t = 4hrs% of oil produced = <u>10-5.9</u> × 100 10 = 41% If b = 4.7g t = 5hrs% of oil extracted = <u>10-4.7</u> × 100 10 = 53%

10

= 30%

If b = 4.2g t = 6hrs% of oil extracted =  $10-4.2 \times 100$ 10 = 58%

If 
$$b = 4.19$$
  
 $t = 7hrs$   
% of oil extracted = 10-4.19 × 100  
10  
= 58.1%

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