

**EVALUATION OF THE CHEMICAL AND PHYSICAL  
PROPERTIES OF SHEA-BUTTER (Butyrospermum Paradoxum)**

**BY**

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**A PROJECT REPORT SUBMITTED TO THE DEPARTMENT OF  
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## CERTIFICATION

This is to certify that this project has been read and approved as meeting the requirement of the Department of Agricultural Engineering, Federal University of Technology, Minna, for the award of Post Graduate Diploma (PGD) in Agric. Engineering.

  
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DATE

## **DEDICATION**

This Research work is specially dedicated to the Almighty God. He gave me success in this programme, for of my self I can do nothing.

## ACKNOWLEDGEMENT

I am most grateful to God for giving me the grace and strength to complete this work.

I also wish to thank my project supervisor, Engr. Chukwu, for his guidance during this research work.

I am most grateful to my dear husband, and my head of department, Dr. D. Adgidzi, for encouraging me to do this work, pushing me when I needed to be pushed and for pulling me when I needed to be pulled, into the path way of success. I also appreciate the entire staff of Agricultural Engineering Department for their co-operation throughout the period of this work. I wish to appreciate the entire management and staff of NAFDAC Kaduna, for allowing me and helping me to carry out the experiments in their laboratory. My gratitude also goes to Mrs. Grace Omojiba for her efforts in arranging for the practicals.

My warmest regards go to my mum, Mrs. Kate Ezeilo, my sisters; Chinwe Ezeilo, Uju Ezeilo and Chinelo Ezeilo for being good sisters indeed. I love you all and pray that you will get the best out of life, in Jesus Name, Amen!

Adgidzi Princess Pat

June, 2002.

## **ABSTRACT**

In this project work, the chemical and physical properties of shea butter were determined and evaluated with the view of ascertaining its edibility and suitability in various processes and applications.

Under approved standard laboratory conditions using standard methods and instruments, experiments were conducted and results obtained. The results obtained showed that shea-butter has the following properties: Saponification value, 196.90, Iodine number. 43.27; Acid value, 3.825; Unsaponifiable matter, 6.23%; Peroxide value, 12.85; Relative density, 0.906; Moisture content, 1.37%; Melting point, 27<sup>0</sup>c; Ash content, 1.26%; Fat content, 75.03% and Carbohydrate content, 22.34%; It also contains the following mineral nutrients: phosphorus, 261 ppm; copper, 0.034ppm; and magnesium, 0.006g/100ml. On comparison with a commonly acceptable oil (groundnut oil), chemical and physical properties of shea-butter apart from slight variations have been proved to be normal as an edible oil, and can fit into various processes and applications.

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# CHAPTER ONE

## 1.0 INTRODUCTION

### 1.1 BACKGROUND TO THE STUDY

The increasing population of Nigeria and consequent rise in standard of living gives rise to increase in the demand for fat and oil in the society, both for consumption in the human diet and for industrial purposes. Oil seeds like groundnut, castor, sunflower, palm fruit and melon have been the major supply of oil to the nation for several purposes. But to meet up with the increasing demand of the society, there is the need to tap into other oil seeds, like sheanut, bambara nut, sesame e.t.c. which have received little or no attention.

The increasing importance of such food materials as sheanut, bambara nut sesame or benniseed, e.t.c. with the complexity of modern technology for their processing, quality evaluation, storage and preservation demands a better knowledge of their chemical and physical characteristics.

Sheabutter extracted from sheanut is not widely used, except locally as medicinal ointment, hair cream, cooking fat e.t.c. Its utilization is not wide especially in the industrial circle, because of insufficient information about its nutritional values which will enhance its usage. It could serve as a good raw material for various industrial products and also for domestic purposes.

While efforts are being made to improve the extraction of shea-butter, this project work goes further to evaluate the chemical and physical properties of shea-butter.

### 1.2 PROJECT JUSTIFICATION

Shea-butter tree is well distributed in Nigeria and in the savanna zone of West Africa. The nut from shea-butter tree is mainly an export produce, apart from some rural consumptions. The maximum utilization has not been achieved, despite its many benefits. In view of this it is important to evaluate the chemical and physical properties of shea-butter, which will ascertain its edibility and determine its suitability in various processes and application. This will enhance shea-butter production in the nation and encourage its usage.

### 1.3 **OBJECTIVES OF THE PROJECT**

The objectives of this project are as follows

- (i) To evaluate the chemical and physical properties of shea-butter.
- (ii) To provide information for the common usage and suitability of shea-butter in various processes

## CHAPTER TWO

### 1.0 LITERATURE REVIEW

#### 2.1 SHEABUTTER TREE

The shea-butter tree (Butyrospermum Paradoxum Subspecies Parkii) is indigenous in the West African Savannah zone occurring abundantly all across West Africa up to the frontiers of sudano – Sahelian zone (Opeke, 1987).

In Nigeria, the spread of the tree is most predominant in the northern part of the country: Bauchi, Niger, Kwara, Adamawa, Zaria and Kaduna. In Western Nigeria, it is found in Oyo, Ibadan and Abeokuta. In Eastern Nigeria, it is mainly located in Ogoja province (Adgidzi, 1999). The local names are “Ka’danya,” “Kareje,” “Chammal,” “emi-emi” and “Osisi-okwuma” in Hausa, Fulani, Tiv, Yuroba and Igbo respectively.

Sheabutter tree belongs to the sapotaceae family. It is a small deciduous tree (it loses its leaves in dry season) up to 15m in height and has a spreading canopy with leaves clustered at the end of the branches. The bark is thick, deeply fissured and fire resistant. The leaves are terminal, oblong, glabrous when mature, and have a wavy margin. The tree produces flowers during dry season. The flowers are creamy white, sweet scented and are placed in terminal clusters (Opeke, 1987). Fruiting begins in the middle of rainy season with a seasonal yield of about 15 – 20kg fruit per tree (FAO, 1982).

The fruit is spherical, ellipsoid berry, 5cm long, borne on a peduncle. It consists of a thin brown cell, enclosing a single dark-brown and shiny egg – shaped seed embedded in a yellowish – green sweet pulp. The kernel is enclosed in a glazed fragile husk. The fruit contains one or two seeds and the sweet pulp is eaten raw at maturity in West Africa.

Fruit is allowed to fall naturally from the tree and is collected from the ground. The maturation period is fairly uniform, but for the entire territory it may vary from June to September with a maximum in July and August (Salunkhe and Desai, 1986).

Oil can be extracted from the seed, which is referred to as shea butter. The butter is prepared by roasting, pounding or grinding and boiling the kernel with water.

The growing world population and rising standard of living are the major pressure on food supply. Approximately 460 million people already have a diet below minimum recommended value for protein and energy supply (2500 – 2700 cal/day). Oil seeds apart from supplying oil either as a by-product or main product has been discovered as a potential new source of protein and energy for human consumption in the developing countries (FAO, 1980).

For example sesame (benniseed) is a valuable supplement for food and feeds because of the methionine content of its protein. It is used for treating scorpion stings, piles, dysentery, gonorrhoea, burns and scales. Like melon seed it is used for soup and can also be taken as a delicacy when roasted or fried and mixed with groundnut. Thus, it is used to enrich other foods.

Bambara nut with a protein content ranging from 18 – 24% can also be used to enrich other foods like maize to achieve a higher protein content of up to 62% (Lartey, 1974). Seeds like soyabean has been incorporated into wearing diet and other foods for enrichment. It is used in the fresh, fermented or dried form as flour, milk and oil condiment. Soyabean is an excellent source of protein (about 35%). It is also rich in calcium, phosphorus, iron and vitamins. It is the only plant source that contains all the Essential Amino Acids (EAA) (Ihekoronye and Ngoddy, (1985).

Therefore , the cultivation and processing of oil seed crops are becoming increasingly important in many tropical and sub-tropical areas. Many of them have industrial uses and are easily incorporated into locally manufactured products.

### 2.3 CHEMICAL AND PHYSICAL PROPERTIES OF SHEA BUTTER

The chemical compositions of an oil-the glycerides and non-glyceridic materials in trace quantities make up the chemical and physical properties of an oil, which in turn determine the suitability of the oil in various processes and applications. The triglycerides form the major component of the glyceridic materials. The fatty acids present in the triglyceride vary in the number of carbon present in the chain (chain length) and in the structure (presence of double bonds i.e. unsaturation). This variation largely defines the chemical and physical properties of oil (Hui, 1996).

### 2.3.1 **CARBOHYDRATES**

These are a group of compounds that contain the elements carbon, hydrogen and oxygen with hydrogen and oxygen being present in the same proportions as in water. Carbohydrates are found in food either as sugar or as starches and glycogen. These latter materials are long straight or branched chains of the many sugar molecules joined together. The chemical nature of the sugars determines their properties, their function in living tissues and how starches are formed and broken down.

The sugar includes the monosaccharides, disaccharides and the polysaccharides. Glucose and fructose are the monosaccharides nutritionally important. Sucrose, lactose and maltose are the disaccharides of the nutrient importance. The only polysaccharides of major nutrient importance are starches and glycogen because they can be digested in the human gut.

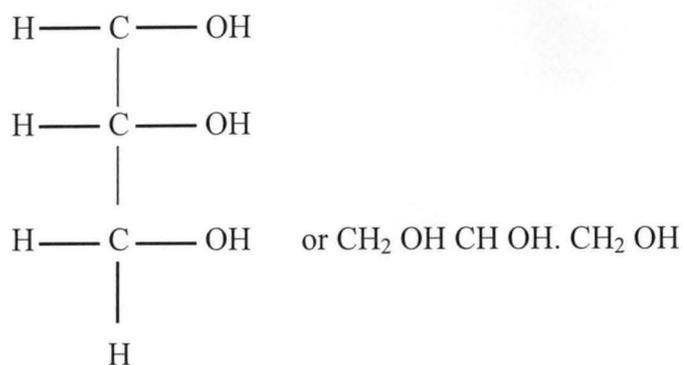
#### **Occurrence of carbohydrates**

Glucose is the keystone of metabolism in both plants and animals. Thus it is the main product of photosynthesis in green plants and occurs in starches as their main storage material in many seeds, roots and tubers. It is scarcely found free in plant materials, with grapes as the most significant exception. It is present in human blood, at about 80 – 120mg /100ml blood (5 – 6 mmol/litre), and is the only sugar that plays a major role in human metabolism. Glycogen is found in the liver and muscles of animals. Fructose is found free in some fruits, but galactose occurs only in combination as lactose in milk.

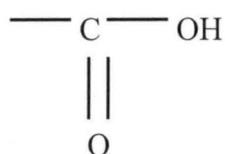
Among disaccharides, sucrose is widely distributed in plant fruit and other tissues e.g. sugar cane. Maltose is found in the starch of grain seeds when they begin to germinate (Mottram, 1979).

### 2.3.2 **FAT**

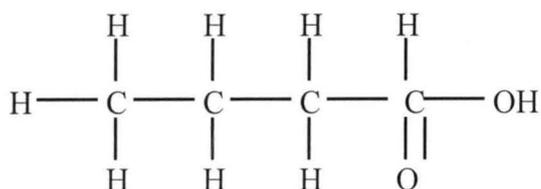
Fats are compounds of glycerol with fatty acids Glycerol known commercially as glycerine is commonly depicted as follows:



The fatty acids consist of chains of carbon and hydrocarbon atoms terminating at one end in the following acidic group:



For example, butyric acid is represented thus:



Many fatty acids exist with 16, 18 or 20 carbon atoms in the chains. Thus stearic acid contains 18 carbons;  $\text{C}_{17}\text{H}_{35} \text{COOH}$ , and no double bonds, oleic acid,  $\text{C}_{17}\text{H}_{35} \text{COOH}$  also contains 18 carbon atoms with one double bond, palmitic acid has 16 carbons, and linoleic acid has 18 carbon atoms with two double bonds. Oleic and linoleic acids are known as unsaturated fatty acids because they can accept additional hydrogen at the sites of double bonds. This is known as hydrogenation.

Usually three different fatty acids are found attached to the glycerol molecules. Thus in butter the main fat is glyceryl butyro – oleostearate, one molecule of each of the three acids being combined with glycerol molecules. The final nature of a fatty or oily substance is determined by the fatty acids that are present in the fat. Spontaneous chemical changes can take place in fat. In the presence of water and some micro – organisms, the links between glycerol and the fatty acids can be split. The fatty acids

may give an unpleasant taste as when butter becomes rancid. Another change results from oxidation reactions at the site of the double bonds in the unsaturated fatty acids. Such reactions may if uncontrolled, produce peroxide groups, which can have far-reaching and harmful effect upon living tissues.

### **Occurrence of fats**

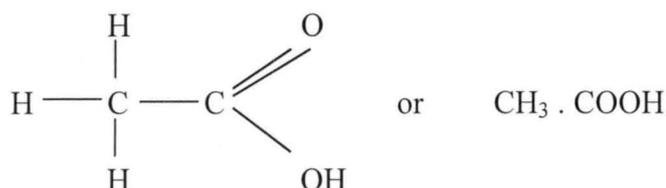
All living cells contain traces of fat in their structure. This is because fatty acids are among the components of cell wall and other intracellular membrane structures. In mammals and birds, fat deposits and stores are found throughout the body: between muscles, around internal organs and under the skin. Many fishes have fat stored exclusively in their liver, but in the herring, it is present throughout the flesh. In the vegetable kingdom, fats are found in the fruiting bodies of various plants, such as olives, maize, palm nut e.t.c.

Oil belongs to the same class as fats, except that oil at room temperature is liquid while fat at room temperature is solid. Thus, it is usually to mean liquid when we speak of oils and solids when we speak of fats. However, we could heat a solid fat until it melted, but we will not necessarily refer to the liquid obtained in this way as oil. For example, olive oil solidifies when cooled (when subjected to a temperature lower than room temperature) but we still refer to it as olive oil. This process is referred to as crystallization and the way in which the cooling is carried out, will determine the form which the fat crystals will adopt, and hence the consistency of the solid. Fat crystallization is important in many foods, as in chocolate, if not set to the correct consistency it will not have the snap which appeals to the consumers (Mottram, 1979).

### **2.3.3 PROTEINS**

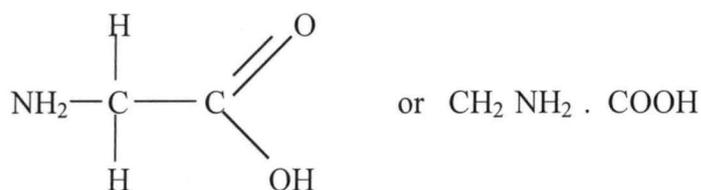
The word protein is derived from Greek, and means 'holding the first place'. Proteins literally hold the first place in the architecture and machinery of all living things. Without them no life can exist. No plant can grow or trap sunlight, no body can be born or reared unless proteins have been made. There is an enormous range of proteins: plant proteins, animal proteins, human proteins all different – but all built up

from the same 20 building blocks (the essential amino acids) in long chains. These can be arranged in any order and there may be several hundred amino acids in a single protein molecule. The amino acids are relatively simple substances. Acetic acid, the acid of vinegar has its structural formula as:



It is the simplest of the fatty acid series. To make an amino acid we replace a hydrogen atom, on the carbon next to the one with = O on it, with an amino group, - NH<sub>2</sub> (which is very closely related to ammonia, NH<sub>3</sub>).

Amino acetic acid (glycine)



All amino acids in proteins exist in one of these arrangements only, the mirror images never being found naturally, though chemists can make them artificially.

When amino acids combine to form protein they do this through the NH<sub>2</sub> group of one amino acid reacting with the OH of another amino acid; splitting of water into H and OH in the process. The nine essential amino acids are: histidine, isoleucine, leucine, lysine, methionine, phenylalanine, threonine, tryptophan and valine. Histidine is needed in children only (Mottram, 1979).

#### 2.3.4 MINERALS

These are inorganic compounds, which appear in food analysis as ash, that is, they are substances left behind, when the carbon, hydrogen and nitrogen (organic compounds) have all been burnt away by excess oxygen. An adult may have over 1kg of calcium in his body, whereas of chromium he has only 5-10mg and of copper 150mg.

The elements which are found in small amounts less than 5mg are called trace elements and are probably working as catalyst or in a small capacity. Though their presence in the body is small, but they are still required. Examples of some important trace elements are cobalt, copper, fluorine, iodine, manganese, molybdenum and zinc. The elements which occur in the body in more than trace amounts or in larger amounts are called macro elements. They include: calcium, sodium, iron, and magnesium e.t.c

All the elements enter into the fluids, cells and other structures of the body and may be needed in definite amounts for the proper functioning of these fluids, cells and structures (Mottram, 1979).

### 2.3.5 VITAMINS

Vitamins are organic substances or compounds needed in very small amount in human body that perform a specific metabolic function and must be provided in the diet of man and animals. Plants can manufacture vitamins from the elements available to them from the soil. The minimum need for a vitamin varies from few micro-organisms to 30mg. The water soluble vitamins are those soluble in water and are heat-labile. They include, vitamin C, vitamin B<sub>1</sub>, vitamin B<sub>2</sub>, B<sub>3</sub>, B<sub>6</sub> and B<sub>12</sub>, (Ascorbic acid, Thiamin, Riboflavin, Niacin, Pyridoxine and Cobalamin respectively). The fat soluble vitamins are present in fats and are not heat labile. They include vitamin A (Retinol), vitamin D (Cholecalciferol), Vitamin E (tocopherols) and vitamin K (naphthoquinones).

#### Retinol (Vitamin A)

It was originally called vitamin A, and is largely composed of carbon and hydrogen as are fatty acids and is soluble in fat. The one OH group at the end of chain of carbon atoms put retinol in the class of alcohols and enables it to combine with fatty acids and other substances. It is a pale yellow oil, very easily oxidized when exposed to air and heat or ultra-violent light. Such oxidation destroys its biological activity. Ordinary cooking processes, however, do not harm retinol or the carotenes from which

it can be formed. Cooking enhances their digestibility and so makes more of the carotenes available for absorption (e.g. carrots).

Vitamin A has the ability to protect epithelia from infection and this has endowed it with the name anti-infective vitamin. In food it is present either as the pre-formed vitamin or as a provitamin many of which are found in vegetable foods. These provitamins are highly coloured and belong to the carotenes group of vegetable dyes; which is the orange coloured material in these vegetables.

It also occurs with chlorophyll in all green vegetables, though it masks the colour of carotene. Yellow-coloured fruits, such as peaches, apricots and oranges and vegetables such as sweet potatoes and pumpkins also owe their colour to carotene. The pre-vitamins are changed to retinol during absorption through the intestinal wall.

Retinol is present in fish and mammalian livers, the flesh of fatty fish, in whole milk, egg yolk and liver (Mottram, 1979).

### **Ergosterol (vitamin D)**

Vitamin D has been known previously as the sunshine vitamin because sunshine is one of its sources to the body, and ricket preventive factor, because of its effectiveness in curing rickets. Since the precursor of vitamin D can be produced in the body, it is considered by some to be technically a hormone. However, when it is supplied by the diet it is technically a vitamin. If it comes from animal sources it is designated as cholecalciferol (vitamin D<sub>3</sub>) and Ergosterol (VitaminD<sub>2</sub>) if it comes from vegetable sources. Vitamin D promotes normal development of bones and teeth, since it facilitates the absorption and utilization of calcium and phosphorus for bone formation. No amount of vitamin D will be of any use if the dietary calcium is deficient or combined as oxalate. It also has a direct action on the kidneys, promoting the reabsorption of phosphate from the urine.

Other sources of vitamin D apart from sunshine include egg, milk, butter and fish – liver oil (Guthrie, 1979).

### **Tocopherol (Vitamin E)**

These were discovered in 1923, they are fat – soluble materials principally in seeds bearing oils, wheat germ oils having the most (Mottram, 1979). It was found to be necessary for normal reproduction in animals. Since a deficiency was shown to produce permanent sterility in male animals and a decrease in the ability of the female animals to conceive or to carry a foetus to term, vitamin E became known as the anti-sterility factor. Its role in human nutrition, however, is still poorly understood. Moreso, other nutrients are capable of performing some, but not all of the functions of these nutrients. Other reasonable sources include diary products, eggs, and green vegetables. As research continues it is possible that an important clinical use of the vitamin will emerge (Guthrie, 1979).

### **Naphthoquinone (vitamin K)**

Vitamin K was discovered in 1934 by a Danish scientist who identified it as the fat soluble factor necessary for the coagulation of the blood. It is used to designate a group of substances belonging to a chemical group known as quinones. These include the naturally occurring fat-soluble vitamins phytyl-menaquinone occurring primarily in green leafy plants and multiprenylmenaquinine which is produced by bacterial synthesis in the gastro intestinal tract and has also been isolated from putrified fish meal, and menaquinone – the synthetic related substance formerly known as vitamin K<sub>3</sub>. The active compounds are present in some of the clovers but are readily formed in the human intestine by bacteria normally living there.

Naphthoquinone deficiency may occur early in infancy before the normal intestinal bacteria become established. Milk contains but little pre – formed material. In adults, deficiency may be due to poor absorption from the intestine or the liver (Guthrie 1979).

#### **2.3.6 IODINE VALUE.**

The iodine value of an oil is defined as the number of grams of iodine absorbed in one gram of oil. It is a measure of the proportion of unsaturated acid present in the oil/fat. ( Hui,1996).

### 2.3.7 **MELTING POINT.**

This is the temperature at which transition from solid to liquid state is observed. Thus, at this temperature a fat melts on the application of heat according to Brown (1987). The lipid rich in saturated fatty acid (e.g. vegetable oil) has low melting point which can be related to difference in the three dimensional shapes between the hydrocarbon chain. The saturated fatty acid (animal fat) has melting point above room temperature because of its compact nature and interaction by dispersion forces.

### 2.3.8 **SAPONIFICATION VALUE**

This is the number of milligrams of potassium hydroxide required to neutralize the free fatty acids resulting from complete hydrolysis of 1g of the oil sample and saponify the esters in one gram of fat (Hui,1996).

### 2.3.9 **FREE-FATTY ACID (FFA)**

These are odorous substances producing irritation on the tongue and in the throat. They make some oil unsuitable for consumption depending on their concentration. If it occurs in minute quantity it may become unnoticeable. Careful harvesting and storage of seeds can keep FFA low in crude oil. To make oil more refined and suitable they have to be removed, (Kirk and Sawyer, 1991).

### 2.3.10 **ACID VALUE**

The acid value of oil is defined as the number of milligrams of potassium hydroxide required to neutralize 1g of the oil sample. The result is expressed as the percentage of free fatty acids (FFA). The value of a measure of the extent to which glyceride in the oil has been decomposed by lipase or other actions. Heat and light accelerate the decomposition. The determination is often used as a general indication of the condition and edibility of the oil (Kirk and Sawyer, 1991).

### 2.3.11 UNSAAPONIFIABLE MATTER.

These are the remaining substances (materials) present in oil and fat after saponification of the oil/fat. These include hydrocarbons, higher alcohols, and sterols e.g. cholesterol. Most oils and fats of normal purity contain less than 2% of unsaponifiable matter (Kirk and Sawyer, 1991).

### 2.3.12 REFRACTIVE INDEX

Light is refracted when it travels through different media because it has different velocities in different media. Light is therefore refracted when it travels through a thin film of melted fat or oil. The refractive index of oil is the ratio of the incident angle to the refracted angle when light travels through the oil at a given wavelength. Various fats or oils have specific refractive indices which are used as a characteristic for identifying fats/oils and checking purity (Kirk and Sawyer, 1991; Nelkon and Parker, 1975).

### 2.3.13 PEROXIDE VALUE

The peroxide value is a measure of the peroxide contained in the oil, which is the first product of oxidation of unsaturated fats/oils. During storage, peroxide formation is slow at first during an induction period, which may vary from few weeks to several months according to the particular oil, the temperature e.t.c. (Kirk and Sawyer, 1991). When the concentration of peroxide reaches a certain level, complex changes occur with the formation of ketones, aldehydes and hydroxyl groups which are volatile and mainly responsible for the off flavours and odours. It is expressed as peroxide per kilogram of fat or in million equivalent (Abdulrahim *et al.*, 2000).

### 2.3.14 RELATIVE DENSITY

Density measurement can be used for pure substances. It is an indication of total solids. It helps to determine the purity of a liquid. Relative density is dimensionless and changes with temperature. Densities have an inverse relationship with molecular weight and a direct one with the degree of unsaturation of the oil (Hui, 1996).

### 2.3.15 VISCOSITY

Viscosity is as a result of frictional force existing in liquids. If the frictional force is low, the viscosity is low and if it is high the viscosity is high. The viscosity of an oil determines whether it is suitable for one product or the other.

## 2.4 EXTRACTION OF SHEABUTTR

### 2.4.1 EXTRACTION OF SHEABUTTER BYTRADITIONAL METHOD

The oil in sheanut is contained in the seed of ripened fruits. In the crude method of extraction, the fruits collected are buried in the ground and allowed to ferment. The pulp disappears rapidly and increased temperature prevents seed germination. Some times, the fruits are heaped in the field for a few days to facilitate the decomposition of the pulp and then trampled underfoot. The extracted seeds are dried in the sun for about 2days. Nuts containing 49% - 50% moisture must be dried to 6 – 7% moisture content for safe storage (Salunkhe and Desal, 1986).

Adequate drying is very important as it minimizes the danger of deterioration in the nut as a result of the action of enzymes.

The nuts are then decorticated. This is the process of cracking the nut and separation of broken shell from the kernel. This process enhances the effectiveness of handling the actual oil bearing parts of the nuts and increases the capacity of the extraction equipment. The decorticated kernels are placed in an oven prior to extraction or fried. This pre-heat treatment is to facilitate bursting of the cells and to improve the milling process in order to allow production of better quality oil.

Cleaning is done using a blower to increase the quantity of the butter obtained. While still warm the kernels are pounded in a mortar and reduced to coarse brown paste, which is further ground on a large mill to produce a greasy homogenous mass or milled using attrition mill. The correct milling is essential for efficient extraction of butter.

The milled mass known as shea-butter meal is subjected to mixing process with water, which involves the simple shear deformation and redistribution of fluid and then cooked with water (Olaoye, 1994). The cooking stimulates the separation of submerged

clear shea-butter from water and other impurities. The fat floating on water surface is collected. The skimmed product is then subjected to further heating or boiling to evaporate water while other impurities are filtered off.

#### 2.4.2 **MODERN METHOD OF EXTRACTION OF SHEABUTTER.**

Oil may be removed from heated milled shea nut by mechanical pressing, centrifuge or solvent extraction (Asiedu, 1979). In mechanical extraction, oil is extracted by mechanical devices such as screw or hydraulic presses, lever and wedge. The extraction is carried out by hot presses in continuous screw presses, followed by filtering and hydraulic pressing.

The Royal Tropical Institute in Netherlands developed the new hydraulic press for obtaining shea-butter which is provided by the German Appropriate Technology Exchange (GATE/GT2) Scheme. The press is based on the dry method in which the butter is squeezed out of heated milled sheanut (sheapowder) under high pressure.

The procedure is as follow:

- 1 Milling the kernel to a very fine powder
- 2 Heating of the powder to about 100% in a large pot or vessel.
- 3 Keeping the powder hot for about an hour in hot air oven.
- 4 Pressing the hot powder in the hydraulic hand press and the fat is collected.

To obtain maximum output, the boiling process is repeated. The resulting cake is an excellent fuel for ovens and considerably reduces the fuel demand during heating process.

The major difficulty in extraction of shea-butter is that the latex agglutinates and clogs the machine delaying movement of fat from the press. Solvent extraction is alsodifficult as the latex prevents good penetration of solvent into the mass. However, industrial extraction removes about 80% of the fat (Salunkhe and Desal, 1986).

#### 2.4.3 **BENEFITS OF SHEABUTTER TREE**

Shea-butter tree is of great benefit from the root to the seed since most part of it is used by man for different purposes. The tree can generally be used as wood, habitat for restoration of soil fertility as animal fodder and watershed.

In northern Nigeria, the wood is also used to make mortars and pestles, wooden bowls, stakes and houses post, because it is hard and termite proof Okafor (1973); Momodu (1987); and Olajide (1992).

The fat extracted from sheanut is locally used as cooking fat, illuminant, medicinal ointment, hair dressing cream, soap making ingredient (Purseglove, 1974). Shea-butter is used in the manufacture of soap, candles and butter substitutes (Opeke, 1987). The consistency of the fat makes it ideal for the preparation of the creams which go into chocolate (Vickery and Vickery, 1979).

Okafor (1973), and Momodu (1987) reported that shea – butter is used for curing leprosy and headache and to aid childbirth. It is also used for cooking and serves as lubricant to rub the body against rheumatic pains and sprains.

In Europe, shea-butter (fat) is used for soap and candle making, in cosmetics and as a constituent for filling for chocolate cream (Purseglove, 1974).

According to Annon (1956) and Purseglove (1974), in west Africa, shea nut production has been estimated at half a million (0.5 million) tonnes per year. For Nigeria, Ghana, Burkina-Faso, Senegal, Mali and Republic of Benin, Sheanut has been a major export produce (Purseglove, 1974). Holland and Belgium used to be the main importers, but in recent years, most of the produce goes to the United Kingdom, Japan and Denmark.

Nigeria, a nation situated in the western coast of the African continent is endowed with abundant natural resources in which agricultural produce plays a major economic role. Agricultural produce of oil seeds like groundnut, palm fruits, cotton seeds, coconut, sunflower, melons and sheanut constitute the major oil bearing agricultural materials. Of these materials, groundnut, cotton-seeds and sheanut are high oil yielding materials predominant in northern Nigeria and have replaced the oil palm as a source of edible oil (Purseglove, 1984).

The north in Nigeria has been identified with high production activities of shea butter both by the Federal Government and World Health Organisation (WHO). This is evidenced by the establishment of shea-butter processing centres at Egbanasara in Katcha Local Government Area of Niger State and at Kure Oja in Ilorin East Local Government Area of Kwara State (Adgidzi, 1999).

## CHAPTER THREE

### 3.0 MATERIALS AND METHODS

The sample used to determine the physical and chemical properties of shea-butter was obtained from shea nut processing centre, Egbanasara, Katcha Local Government, Niger State (FGN/NGSG/WHO assisted). The shea-butter sample was used without further preparations. The test and analyses were carried out at (NAFDAC Food Laboratory Kaduna, between 29<sup>th</sup> May to 7<sup>th</sup> June 2002.

### 3.1 REAGENTS AND INSTRUMENTS.

In the course of the practical work carried out in the project, the reagents and instruments used are listed below.

#### 3.1.1 REAGENTS

Tetraoxosulphate (VI) acid,  $H_2SO_4$

Hydrogen Chloride acid, HCl

Blue litmus papers

Methyl red indicator

Sodium hydroxide, NaOH

Wijs solution

Potassium iodide solution, KI

Tetrachloromethane (carbon tetrachloride)  $CCl_4$

Trioxoborate (III) acid (Boric acid)  $H_3BO_3$

Ammonium chloride solution,  $NH_4Cl$

Copper Tetraoxosulphate (VI) (copper sulphate)  $CuSO_4$

Potassium Tetraoxosulphate (VI) (Potassium sulphate)  $K_2SO_4$ .

Phenolphthalein in ethanol

Ethanoic KOH solution.

Ethanol ( $C_2H_5OH$ ).

Glacial ethanoic acid (Glacial acetic acid).

Starch solution

Sodium trioxothiosulphate (VI) solution

(sodiumthiosulphate solution)  $Na_2S_2O_3$ .

Ethoxyethanol (Diethyl ether)  $C_2H_5O C_2H_5$ .

Iodine trichloride.  $ICl_3$

Trichloromethane (chloroform)  $CHCl_3$

Propanone (acetone).  $CH_3COCH_3$ .

Solo chrome black indicator

Dilute trioxonitrate (V) acid (Dilute nitric acid)  $HNO_3$ .

Vanado – molybdate reagent.

### 3.1.2 INSTRUMENTS

Air oven

Petri dishes

Dessicator

Weighing balance

Forceps

Filter papers

Water bath

Reflux condenser

Kjeldahl flask

Distillation apparatus

Heating devices

Density bottle

Glass flask

Micro burner

Round bottom flask

Refractometer with prism

Spectrophotometer

Pipette

Burette

Spatula

Retort stands

Beakers

## 3.2 EXPERIMENTAL PROCEDURES

### 3.2.1 DETERMINATION OF ACID VALUE

Reagents

Diethyl ether

Ethanol

1% phenolphthalein

0.1 M sodium hydroxide

#### Procedure

25ml diethyl ether was mixed with 25ml ethanol. 1ml of 1% phenolphthalein solution was added and the mixture was neutralized with 0.1M NaOH. 1.10g of the fat was dissolved in the neutralized solvent mixture and titrated with 0.1M NaOH solution with constant shaking until a pink colour appears, indicating complete neutralization.

Acid value =  $\frac{\text{Titration (ml)} \times 5.61}{\text{Weight of sample used}}$  (Usoro *et al.*, 1982).

### 3.2.2 DETERMINATION OF IODINE VALUE.

Principle:

The fat is treated with an excess of carbon tetrachloride. The excess of carbon tetrachloride is estimated by treating it with potassium iodide and titrating the liberated iodine with standard sodium thiosulphate solution.

Reagents:

1. Wijs solution: 8g iodine trichloride was dissolved in 200ml glacial acetic acid. 9g iodine was dissolved in 300ml carbon tetrachloride. The two solutions were mixed together and diluted to 1 litre with glacial acetic acid.
2. Carbon tetrachloride.
3. 10% potassium iodide solution.
4. 0.1M sodium thiosulphate solution
5. starch indicator (1% aqueous solution of gelatinized starch).

### **Procedure:**

0.5g of the fat was weighed into a glass stoppered bottle of 250ml capacity. 10ml carbon tetrachloride was used to dissolve the sample. Exactly 20ml of Wij's solution was added and the stopper moistened with potassium iodide solution was inserted into the mouth of the bottle. The contents were mixed and the mixture allowed to stand in the dark for 30 minutes. 15ml potassium iodide solution was added and 10ml of water. After mixing, titration was done with standard thiosulphate solution with starch used as indicator before end point (titration = a ml). A blank was carried out simultaneously omitting the fat (titration = b ml).

$$\text{Iodine value} = \frac{(b - a) 1.269}{\text{Wt in g of sample}} \quad (\text{Uoro } et \text{ al.}, 1982)$$

### **3.2.3 DETERMINATION OF SAPONIFICATION VALUE**

#### **Principle:**

The fat is saponified with excess of alcoholic KOH. The amount of KOH remaining after saponification is determined by titration with standard acid solution.

#### **Reagent:**

1. Alcoholic solution of KOH.
2. 0.5M hydrochloric acid solution.
3. Phenolphthalein 1% solution in ethanol.

#### **Procedure**

2g of the sample was weighed into a flask. 25ml of alcoholic KOH solution was pipetted into the flask. A reflux condenser was attached to the flask before heating on a boiling water bath for 1 hour with occasional shaking. 1ml of phenolphthalein solution was added and standard HCl (a ml) was used to titrate while hot. A blank determination was carried out (b ml).

$$\text{Saponification value} = \frac{(b - a) 28.05}{\text{Wt of sample}} \quad (\text{Uoro } et \text{ al.}, 1982)$$

### 3.2.4 DETERMINATION OF UNSAPONIFIABLE MATTER

1ml of aqueous 3M KOH solution was added to the neutralized liquid left after the titration for the saponification value. The liquid was transferred to a separator, using a quantity of water 50ml less the volume of 0.5M HCl used in the titration for saponification value. While still warm, extraction was done with 3x50ml diethyl ether. The ether layer was washed thrice by shaking vigorously with 20ml portions of aqueous 0.5M KOH solution and then with 20ml portions of water until the wash water no longer turn red litmus paper to blue.

The ether extract was poured into a flask, and the ether was evaporated off by heating on a water bath. 2ml acetone was added while the heating was going on to complete the removal of the solvent. The fat residue in the flask was dried to constant weight at 100<sup>0</sup>c, and dissolved in 2ml of ether. 10 ml of neutralized ethanol was added and the mixture was titrated with 0.1M alcoholic KOH (v) and no colour change was observed.

$$\text{Unsaponifiable matter} = \frac{M_2 - 0.0282V \times 100g}{M_1}$$

M<sub>1</sub> = mass of fat taken for saponification value

M<sub>2</sub> = mass of unsaponifiable matter

(Usono et al., 1982).

### 3.2.5 DETERMINATION OF PEROXIDE VALUE

#### **Principle;**

The fat is treated with potassium iodide in an organic solvent. The peroxides liberate the iodine from potassium iodide. The amount of iodine liberated by the sample is a measure of the active oxygen present.

#### **Reagent:**

1. Potassium iodide.
2. Solvent mixture consisting of 2 volumes of glacial acetic acid one volume of chloroform.
3. Sodium thiosulphate solution.
4. Starch indicator

**Procedure:**

1g of the fat was weighed into a conical flask and dissolved with 10ml of solvent mixture of glacial acetic acid and chloroform (3:1). Saturated solution of potassium iodide was added to the dissolved sample and the mixture was allowed to stand for 15 minutes with constant shaking. 30ml of water was added and a drop of starch indicator melted in hot water. The mixture was titrated with sodium thiosulphate until the brown colour clears. A blank determination was carried out.

$$\text{Peroxide value} = \frac{S \times M \times F}{\text{Weight in g of sample}}$$

Where: S = volume of thiosulphate solution

M = molarity of thiosulphate solution

F = factor (= 1000).

3.2.6 **DETERMINATION OF RELATIVE DENSITY (40<sup>0</sup>C).**

**Apparatus:**

Density bottle

Water bath

Thermometer

**Procedure:**

- i. The density bottle was cleaned, dried, cooled in a dessicator and weighed
- ii. It was filled with distilled water and maintained at 20<sup>0</sup>c until the water inside reaches 20<sup>0</sup>c.
- iii. The outside of the bottle was wiped and weighed.
- iv. The bottle was emptied of water, dried and filled with oil sample. (40<sup>0</sup>c).
- v. The outside was wiped and the bottle and oil weighed.

$$\text{Relative density} = \frac{W_3 - W_1 \text{ (weight of oil)}}{W_2 - W_1 \text{ (weight of water)}}$$

W<sub>1</sub> = weight (g) of density bottle.

W<sub>2</sub> = weight (g) of density bottle + water.

$W_3$  = weight (g) of density bottle + oil (Abdulrahim *et al.*, 2000).

### 3.2.7 DETERMINATION OF REFRACTIVE INDEX

#### Apparatus

Refractometer with prism.

#### Procedure:

The hinged prism of the refractometer was unclamped. Both prism surfaces were cleaned with cotton wool to remove dirt.

A drop of the sample at 40<sup>0</sup>c was applied to the lower prism surface and the prism was resealed. The oil was observed through the telescope while the control knob was adjusted.

The refractive index was read from the scale on the telescope.

The refractive index of water at 20<sup>0</sup>c was also measured to determine the accuracy of the instrument (Usoro *et al.*, 1982).

### 3.2.8 DETERMINATION OF MELTING POINT (CAPILLARY TUBE METHOD).

The fat sample was melted and taken up into the capillary tube (up to 1cm). The capillary tube was kept in a cold condition until it solidifies.

The capillary tube was attached to a thermometer and dipped inside water in a beaker and placed on a heating device. When heated, once the oil slips and appears clear in the capillary tube the reading on the thermometer was taken as the melting point (Hui,1996).

### 3.2.9 DETERMINATION OF MOISTURE CONTENT.

Moisture content is the amount of moisture (water) present per given weight of sample. It is checked in order to preserve the oil, thereby promoting shelf – life because oxidative rancidity, microbial growth and infestation are prevented or reduced by moisture removal.

## Air Oven method

### Principle:

This is based on weight loss as a result of drying the sample to constant weight in an air oven at specific temperature and time.

No reagent required.

### Apparatus

Oven (100 – 102<sup>0</sup>c).

Petri – dishes

Dessicator

Weighing balance

Forceps

### Procedures

1. Empty dish is dried in oven for 15 minutes, cooled in the dessicator and weighed
2. The sample was mixed thoroughly, and 5g was transferred into the dish. The dish and the sample were weighed as rapidly as possible.
3. The dish is placed in the oven avoiding contact of the dish with oven walls. The sample is dried for 6 hours starting when the temperature of the oven reached 100<sup>0</sup>c.
4. The dish is removed from the oven, cooled in a dessicator and reweighed when cold
5. Drying was done further for an hour to ensure that constant weight has been achieved.

$$\% \text{ Moisture} = W_2/W_1 \times 100$$

$$\% \text{ Total solids} = W_3/W_1 \times 100$$

$W_1$  = Weight in grammes of original sample.

$W_2$  = Loss in weight.

$W_3$  = weight in grammes of dried sample. (Osborne and Vooget, 1978).

### 3.2.10 DETERMINATION OF NITROGEN AND CRUDE PROTEIN.

#### Macro Kjeldhal method

##### **Digestion :**

1g of sample was weighed into a Kjeldhal flask. 25ml conc. Sulphuric acid was added to the flask using a measuring cylinder.

2 tablets of mercury were added. The flask in an inclined position was heated in a fume cupboard. Digestion was continued until the liquid became clear, free from black and brown colour. The flask was swirled from time to time to wash down charred particles from the sides of the flask.

##### **Steam Distillation:**

After digestion, the flask was cooled, and made up to 200ml. 100ml was taken and diluted with 10ml distilled water. 50ml of 50% NaOH and 25ml of 25% sodium thiosulphate were added. The distillation apparatus consisting of 500ml capacity was set up. 50ml of boric acid solution was measured into a 500ml conical flask and few drops of screened methyl red indicator were added and the flask was placed under the condenser of the distillation apparatus so that the delivery tube dips just below the level of the boric acid.

The contents of the Kjeldhal flask were transferred into the distillation flask and rinsed with about 50ml of water. 100ml of NaOH solution was poured carefully down the neck of the distillation flask from a measuring cylinder, with the flask attached to the splash head of the distillation apparatus.

The alkaline liquid is placed on a heating device until it started boiling and boiling continued for 20 minutes. The conical flask was lowered just before terminating the distillation, so that the outlet of the adapter is above the liquid level.

The outlet of the adapter above the liquid was rinsed internally and externally with a little water. The completion of the ammonia distillation was verified by testing the distillation from the condenser with red litmus paper. If it no longer turns blue then heating was stopped.

Titration:

The content of the conical flask was titrated with hydrochloric acid solution

The volume was recorded.

Blank test:

A blank test was conducted following the same procedure but no sample was added.

$$\% \text{ Total Nitrogen} = \frac{(V_2 - V_1) \times N}{w} \times 1.4$$

$$\% \text{ Crude Protein} = \frac{V_2 - V_1 \times N}{w} \times 6.25$$

Where 6.25 is the general factor.

W = Weight of the test sample

V<sub>1</sub> = Volume (ml) of hydrochloric acid solution required for the blank test.

V<sub>2</sub> = Volume (ml) of hydrochloric acid solution required for the test portion

N = Normality of hydrochloric acid (Osborne and Vooget, 1979).

3.2.11 **DETERMINATION OF TOTAL FAT**

**Principle:**

The protein is precipitated by alcohol and dissolved by ammonia. The freed fat is then extracted with ether and petroleum ether.

**Procedure:**

3g of the fat was weighed into an extractor (w). The sample was dispersed with 10ml water; and 2ml of 0.88 ammonia solution was added and mixed. 10ml of alcohol (95%) was added and mixed well.

25ml of diethyl ether was added, the tube was corked and shaken vigorously for 1minute. 25ml of light petroleum was added and the mixture shaken for 30 secs. The extraction was repeated twice using 25ml portion of a mixture (1:1) of diethyl ether and petroleum ether and the ether of fat was collected in a weighed flask (W<sub>1</sub>). Sodium sulphate was added into it.

The ether was evaporated by placing on a water bath and the residue dried in an oven at 100<sup>0</sup>c, cooled and then weighed (W<sub>2</sub>).

NB: This method can be used in the presence of carbohydrate.

$$\% \text{ Fat} = \frac{W_2 - W_1}{W} \times 100$$

W = weight (g) of the sample.

W<sub>1</sub> = weight (g) of flask

W<sub>2</sub> = weight (g) of flask and sample after evaporating or residue. (Usono *et al.*, 1982).

### 3.2.12 ASH CONTENT DETERMINATION

#### Principle:

The organic component of food is burnt off in air. The residue is ash which consists of the inorganic components in the form of their oxides.

Apparatus: Platinum dish.

Procedure: Platinum dish was cleaned, dried, cooled in a dessicator and weighed. 5g of the sample was weighed into the platinum dish. The sample was charred on a hot plate in a fume cupboard until no more soot is given off. The dish is cooled in dessicator and the weight of dish and ash taken.

$$\% \text{ Ash} = \frac{W_3 - W_1}{W_2 - W_1} \times 100 \quad (\text{Usono } et \text{ al.}, 1982).$$

### 3.2.13 DETERMINATION OF MINERALS

The minerals are determined using the ash recovered.

#### Phosphorus

The ash was dissolved with drops of water and concentrated HCl, and made up to 100ml. 10ml was taken in 100ml volumetric flask, and was neutralized with dilute ammonia, and again made acidic with dilute nitric acid. 25ml of vanado – molybdate reagent was added and then made up to 100ml mark with distilled water. Serial concentration of 4, 8, 16ppm phosphate standards were made.

The absorbances of the yellow colour developed were read with a spectrophotometer at 470nm after 10minutes.

The absorbances were plotted against the concentrations in a linear graph and the concentration for the sample was traced from the graph.

## Magnesium

### Reagents

Ammonium chloride

Solo chrome Black indicator

0.05M EDTA

### Procedure:

40ml of sample solution was taken into a conical flask. This was diluted to 100ml with distilled water. 5ml of Ammonium chloride buffer and 0.03g solo-chrome black indicator was added. Titrate with 0.05M EDTA to blue end point.

$$\text{Magnesium} = \frac{S \times M \times W}{V}$$

S = Titre value of test titration

M = molarity of EDTA

V = volume of sample solution

W = molecular mass of magnesium

## COPPER DETERMINATION

10g of sample was ashed

10ml of 6M HNO<sub>3</sub> was added and heated to dissolve the ash and then cooled.

10ml 6M H<sub>2</sub>SO<sub>4</sub> was next added and the solution heated to white fumes and then cooled.

20ml of water was added and solution heated for two minutes. Cooled 6M Ammonia was added until there was a blue colour.

3M H<sub>2</sub>SO<sub>4</sub> was added to discharge the colour.

The solution was transferred to 200ml volumetric flask and was made up to the mark with distilled water. 50ml of solution was taken in a 250ml volumetric flask.

2ml of phosphoric acid was added followed by 2g Potassium iodide dissolved in 10ml of water.

The resulting yellow solution was titrated with 0.01M sodium thiosulphate with 2ml 1% starch as the indicator.

Titre value was recorded for further calculation.

## CHAPTER FOUR

### 4.0 EXPERIMENTAL RESULTS AND DISCUSSIONS

#### 4.1 Presentation of Data and Analysis of Data.

##### 4.1.1 ACID VALUE DETERMINATION

The results of the experiment carried out to determine acid value are in Table 2

Table 2: Titre values from titrating with 0.1M NaOH

	1 <sup>st</sup> Titration	2 <sup>nd</sup> Titration
Final burette reading (ml)	0.80	0.70
Initial burette reading (ml)	0.00	0.00
Volume of 0.1M NaOH (ml) used	0.80	0.70

Calculations:

$$\text{Average titre} = \frac{0.80 + 0.70}{2} = 0.75\text{ml}$$

$$\begin{aligned} \text{Acid value} &= \frac{\text{Titre value} \times 5.61}{\text{Weight of sample}} \\ &= \frac{0.75 \times 5.61}{1.1} = 3.825 \text{ mg NaOH/g oil} \end{aligned}$$

##### 4.1.2 IODINE VALUE DETERMINATION

The results obtained from the experiment carried out to determine iodine value are in Table 3.

Table 3: Titre values from titrating with standard thiosulphate

	1 <sup>st</sup> titration		2 <sup>nd</sup> titration	
	Test	Blank	Test	Blank
Final burette reading (ml)	17.10	34.00	16.30	33.50
Initial burette reading (ml)	0.00	0.00	0.00	0.00
Volume of 0.1M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> (ml) used	17.10	34.10	16.30	33.50

Calculations:

$$\text{Iodine number} = \frac{(b - a) 1.269}{\text{Weight of sample}}$$

b = Titre value for blank titration

a = Titre value for test titration

$$\text{: - For 1}^{\text{st}} \text{ Titration Iodine number} = \frac{(34.00 - 17.10) 1.269}{0.5}$$

$$= 42.8922$$

$$\text{For 2}^{\text{nd}} \text{ Titration Iodine number} = \frac{(33.50 - 16.30) 1.269}{0.5}$$

$$= 43.65$$

$$\text{Average Iodine number} = \frac{42.89 + 43.65}{2} = 43.27$$

#### 4.1.3 SAPONIFICATION VALUE DETERMINATION.

The results from the experiment carried out to determine saponification value are presented in Table 4.

Table 4: Titre values from titrating with standard HCl

	1 <sup>st</sup> titration		2 <sup>nd</sup> titration	
	Test	Blank	Test	Blank
Final burette reading (ml)	13.90	28.50	14.30	29.00
Initial burette reading (ml)	0.00	0.00	0.00	0.00
Volume of 0.5M HCl (ml) used	13.90	28.50	14.30	29.00

Calculations:

$$\text{Saponification value} = \frac{(b - a) 28.05}{\text{Weight of sample}}$$

Where:

b = Titre value of blank titration.

a = Titre value of test titration

28.05 = Number of milligrams of KOH in 1ml 0.5M HCl solution.

$$\text{For 1}^{\text{st}} \text{ Titration, Saponification value} = \frac{(28.50 - 13.90) 28.05}{2.087}$$

$$= 196.229$$

$$\text{For 2}^{\text{nd}} \text{ Titration, Saponification value} = \frac{(29.00 - 14.30) 28.05}{2.087}$$

$$= 197.573$$

$$\text{Average saponification value} = \frac{196.229 + 197.573}{2}$$

$$= 196.90.$$

#### 4.1.4 UNSAAPONIFIABLE MATTER DETERMINATION

The results obtained during the course of determining unsaponifiable matter with solution left after the titration of saponification value are listed below.

Weight (g) of flask used + residue after extraction = 65.92g

Weight (g) of flask = 65.79g

$M_2$  = mass of unsaponifiable matter = 0.13g

Mass of fat taken from saponifiable matter ( $M_1$ ) = 2.087g

$$\text{Unsaponifiable matter} = \frac{M_2 - 0.0282V \times 100}{M_1 \quad 1}$$

$M_1$  = mass of fat taken

V = ml of 0.1M KOH required to neutralize the residue in the determination of unsaponifiable matter

$M_2$  = mass of unsaponifiable matter

$$\begin{aligned} \text{Unsaponifiable matter} &= \frac{0.13 - 0 \times 100}{2.087 \quad 1} \\ &= 6.23\% \end{aligned}$$

#### 4.1.5 PEROXIDE VALUE DETERMINATION

The results from the experiment carried out to determine peroxide value are presented in Table 5

Table 5: Titre values from titrating with sodium thiosulphate

	1 <sup>st</sup> titration		2 <sup>nd</sup> titration	
	Test	Blank	Test	Blank
Final burette reading (ml)	0.18	0.05	0.20	0.05
Initial burette reading (ml)	0.00	0.00	0.00	0.00
Volume Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> (ml) used	0.18	0.05	0.20	0.05

$$\text{Peroxide value (milliequivalent/kilogramme)} = \frac{S \times M \times F}{\text{Weight of sample}}$$

Where S = volume of thiosulphate solution

M = molarity of thiosulphate solution

F = Factor (= 1000).

Calculations:

$$\text{For 1}^{\text{st}} \text{ Titration, peroxide value} = \frac{(0.18 - 0.05) \times 0.1 \times 1000}{1.09} = 11.93$$

$$\text{For 2}^{\text{nd}} \text{ Titration, peroxide value} = \frac{(0.20 - 0.05) \times 0.1 \times 1000}{1.09} = 13.76$$

$$\begin{aligned} \text{Average peroxide value} &= \frac{11.92 + 13.76}{2} \\ &= 12.845 \text{ meq./kg oil.} \end{aligned}$$

#### 4.1.6 RELATIVE DENSITY DETERMINATION.

In the course of the experiment the following results were obtained.

Weight of the empty relative density bottle ( $W_1$ ) = 35.21g

Weight of relative density bottle + water ( $W_2$ ) = 85.89g

Weight of relative density bottle + oil at 40<sup>0</sup>c ( $W_3$ ) = 81.13g

$$\begin{aligned} \text{Relative density} &= \frac{W_3 - W_1}{W_2 - W_1} \\ &= \frac{81.13 - 35.21}{85.89 - 35.21} = \frac{45.92}{50.68} \end{aligned}$$

∴ Relative density = 0.906

#### 4.1.7 REFRACTIVE INDEX DETERMINATION

Oil and water when viewed through the refractometer gave the following results.

<u>Sample</u>	<u>Temperature</u>	<u>Refractive index</u>
Water	20 <sup>0</sup> c	1.444
Oil	40 <sup>0</sup> c	1.452

#### 4.1.8 MELTING POINT DETERMINATION

The reading in the thermometer when the fat melted was 27<sup>0</sup>c

Therefore melting point for shea butter is 27<sup>0</sup>c.

#### 4.1.9 MOISTURE CONTENT DETERMINATION

After oven drying, the following results were obtained.

	Dish A	Dish B
$W_1$ = Weight of original sample	5.000	5.000
$W_3$ = Weight(g) of dried sample	4.934	4.929
$W_2$ = Loss in weight (g)	0.066	0.071

Calculations:

$$\begin{aligned}\% \text{ moisture content of dish A} &= \frac{W_2}{W_1} \times \frac{100}{1} \\ &= \frac{0.066}{5.000} \times \frac{100}{1} \\ &= 1.32\%\end{aligned}$$

$$\begin{aligned}\% \text{ moisture content for dish B} &= \frac{0.071}{5.000} \times \frac{100}{1} \\ &= 1.42\%\end{aligned}$$

$$\text{Average \% moisture content} = 1.37\%$$

$$\% \text{ Total solids} = \frac{W_3}{W_1} \times \frac{100}{1}$$

$$\text{For dish A} = \frac{4.934}{5.000} \times \frac{100}{1} = 98.68\%$$

$$\text{For dish B} = \frac{4.929}{5.000} \times \frac{100}{1} = 98.58\%$$

$$\begin{aligned}\text{Average \% total solids} &= \frac{98.68 + 98.58}{2} \\ &= 98.63\%\end{aligned}$$

#### 4.1.10 NITROGEN AND CRUDE PROTEIN DETERMINATION

During distillation, there was no colour change in the boric acid solution as a result of the distillation. This implies the absence of nitrogen in the sample.

The digestion and distillation was repeated and the same result was gotten.

$V_1$  = volume (ml) of HCl solution for blank test = 0

$$\% \text{ Total Nitrogen} = \frac{(V_2 - V_1) N}{W} \times 1.4$$

$$\% \text{ Crude Protein} = \frac{(V_2 - V_1) N}{W} \times 6.25$$

#### 4.1.11 TOTAL FAT DETERMINATION

$W$  = Weight (g) of the sample = 3.00g

$W_1$  = Weight (g) of flask = 56.715g

$W_2$  = Weight of flask + residue after extraction = 58.966g

$$\% \text{ fat} = \frac{W_2 - W_1}{W} \times \frac{100}{1}$$

$$= \frac{(58.966 - 56.715)}{3} \times \frac{100}{1}$$

$$= 75.03\%$$

#### 4.1.12 ASH CONTENT DETERMINATION

$W_1$  = Weight (g) of platinum dish = 36.550g

$W_2$  = Weight (g) of sample + dish = 41.550g

$W_3$  = Weight (g) of dish + ash = 36.613g

$$\% \text{ Ash} = \frac{W_3 - W_1}{W_2 - W_1} \times \frac{100}{1}$$

$$= \frac{0.063}{5} \times \frac{100}{1}$$

$$= 1.26\%$$

#### 4.1.13 CARBOHYDRATE DETERMINATION

After determination of percentage moisture, fat and ash in the absence of protein and fibre, the carbohydrate was determined by subtraction.

Thus, % carbohydrate =  $100 - (75.03 + 1.37 + 1.26) = 100 - 77.66$

$$= 22.34\%$$

#### 4.1.14 PHOSPHORUS DETERMINATION

Reading from spectrophotometer under 470nm. (see Appendix A)

<u>Concentrations [Blank]</u>	<u>Absorbances</u>
4ppm	0.040
8ppm	0.073
16ppm	0.142
Concentration (Test)	
(a) 5ppm	0.039
(b) 3ppm	0.039

The concentration for sample can also be gotten from the graph. When the absorbances were plotted against the concentration of the blanks

The reading from the graph is 3ppm (see Appendix B)

The average concentration from the spectrophotometer

$$\begin{aligned} &= \frac{3 + 5}{2} \\ &= 4\text{ppm} \end{aligned}$$

However, the value for the graph was taken for calculating the actual concentration of phosphate in the sample and phosphorus.

Dilution factor = 200

Actual concentration of phosphate =  $3 \times 200 = 600\text{ppm}$

$$600 \times 10^{-6} = 0.06\%$$

$$= \frac{\text{Molecular mass of phosphorus} \times \text{concentration of phosphate}}{\text{Molecular mass of phosphate} \times 1}$$

$$\frac{62}{142} \times \frac{600}{1} = 261\text{ppm}$$

$$261 \times 10^{-4} = 0.026\%$$

#### 4.1.15 MAGNESIUM DETERMINATION

$$\text{Magnesium} = \frac{S \times M \times W}{V}$$

S = Titre value of test titration = 2ml

M = Molarity of EDTA = 0.05

V = volume of sample solution = 40ml

W = molecular mass of magnesium = 24

D = Weight of sample taken for ash determination = 5g

$$\begin{aligned}\text{Magnesium} &= \frac{2 \times 0.05 \times 24}{40} \\ &= 0.06\text{g/litre} \\ &= 0.006\text{g}/100\text{ml}\end{aligned}$$

$$\begin{aligned}\% \text{ of magnesium in the 5g sample taken for ash} &= \frac{0.006}{5} \times \frac{100}{1} \\ &= 0.12\%\end{aligned}$$

#### 4.1.16 **COPPER DETERMINATION**

Actual concentration for copper in the solution

$$\begin{aligned}\text{Molarity of sample solution} &= \frac{\text{titre} \times \text{molarity of thiosulphate}}{\text{Volume of sample}} \\ &= \frac{2.7 \times 0.01}{50} \\ &= 0.00054\text{mol}\end{aligned}$$

Concentration = Molarity x molar mass of copper

$$\begin{aligned}&= 0.00054 \times 63.5 \text{ mg/ml} \\ &= 0.034\text{mg/ml} \\ &= 0.034\text{ppm} \times 10^{-6} \\ &= 0.0000034\%\end{aligned}$$

## 4.2 **DISCUSSION OF RESULTS**

### 4.2.1 **CHEMICAL AND PHYSICAL PROPERTIES**

In this discussion, shea butter is compared with groundnut oil, one of the highly consumed oils from seeds.

From the test and analyses carried out, shea butter has low moisture content of about 1.37%, and this is an indication that rancidity of the butter cannot easily take place. As a result microbial growth is small compared to groundnut oil which is susceptible to oxidative rancidity with a moisture content of 3.9% - 13.2% (Freeman *et al.*, 1954).

There is virtually no protein in shea-sbutter, though for shea nut kernel meal there may be small percentage of protein, yet the protein is low compared to groundnut oil which has a

protein content ranging from 21.0% - 36.4% (Freeman *et al.*, 1954). The fat content of shea butter is high, 73.03%, which explains why it is solid at room temperature, and can be used as oil substitute for cooking and other processes.

The ash content obtained is 1.26%, which is of considerable amount and represents the minerals or organic materials present in shea-butter.

Shea butter is high in carbohydrate compared to groundnut oil with both the reducing sugar, disaccharides and starch as 3.05%. Thus it can be a good source of energy.

Table 6 shows the chemical composition of groundnut.

Table 6: Proximate Analysis of groundnut oil

Constituents	Range (%)
Moisture	3.9 – 13.2
Protein	21.0 – 36.4
Lipid	35.8 – 54.2
Ash	1.8 – 3.1
Reducing sugar	0.1 – 0.3
Disaccharide sugar	1.9 – 5.2
Starch	1.0 – 5.3

Source: Freeman *et al.*, (1954).

Table 7 (shows the characteristics of groundnut oil).

Table 7: Chemical and Physical characteristics of groundnut oil

Melting point	0 – 3 <sup>0</sup> c
Iodine number (wijs)	-26
Saponification number	188- 196
Free fatty acid as oleic	0.02 – 0.60%
Unsaponifiable matter	0.40%
Refractive index ( $n_D^{40^0c}$ )	1.46 – 1.465
Acid value	0.30mg KOH/g oil
Acid value (virgin oil max.)	4mg KOH/g oil
Peroxide value (max)	10 meq. O <sub>2</sub> / kg oil
Relative density (40 <sup>0</sup> c)	0.915

Source: Hui (1996).

Tables 8 and 9 show the properties of shea-butter from the experiment I conducted.

Table 8: Physical properties of shea-butter

Constituents	Quantities
Moisture	1.37%
Ash	1.26%
Total fat	75.03%
Carbohydrate	22.34%
Refractive index (40 <sup>0</sup> c)	1.452
Relative density (40 <sup>0</sup> c)	0.0906
Melting point	27 <sup>0</sup> c

Table 9: Chemical properties of Shea-butter

Acid value	3.825 mg KOH
Iodine number	43.27
Peroxide value	12.85 meq. /kg oil
Saponification value	196.90
Unsaponifiable matter	6.23%

The high fat content of shea-butter makes it solid at room temperature, thus it has a high melting point of 27<sup>0</sup>c when compared to groundnut oil which has a melting point of 0-3<sup>0</sup>c. The consistency of shea butter makes it ideal for the preparation of creams which go into chocolates.

There was considerable prejudice against shea-butter for edible purposes owing to its high proportion of unsaponifiable matter. The prejudice however, has not been justified and large quantities of edible oil made from shea-nut are being consumed. The consumption of shea-butter as an edible oil therefore explains that the unsaponifiable value of shea-butter, 6.23% is within the human consumption limit. However, the presence of this unsaponifiable matter reduces considerably the value of the oil for soap making purposes (Williams,1950).

Shea-butter has acid value of 3.825 which is fairly higher than 2. An oil is considered acidic if its acid value is greater than 2 (FAO, 1979). However, the acid value of shea-nut may not be harmful to the body, since groundnut which is being consumed extensively used has its acid value as 4 mg KOH/oil.

Apart from the highlighted properties, the chemical and physical properties of shea-butter are normally compared to other edible oils, and can fit into various products. The oil when refined, can be made practically tasteless and odourless.

## **CHAPTER FIVE**

### **5.0 CONCLUSION AND RECOMMENDATIONS**

#### **5.1 CONCLUSION**

In the course of this work, it has been established that the chemical and physical properties of shea-butter are comparable to that of other oil seeds. Thus, the properties should not be a limitation to its use as an edible oil and as raw material for several domestic and industrial purposes and applications. An exception is its limited use for soap making purposes because of the high unsaponifiable matter.

#### **5.2 RECOMMENDATIONS.**

The following recommendations are made after completing this work;

- (1) Due to the fact that shea-butter is a product of shea nut, there is need for evaluation of the whole shea nut kernel meal, to determine the chemical and physical compositions:
- (2) There is need for a study to be conducted on the refining of shea butter, with the view of making it odourless and tasteless.

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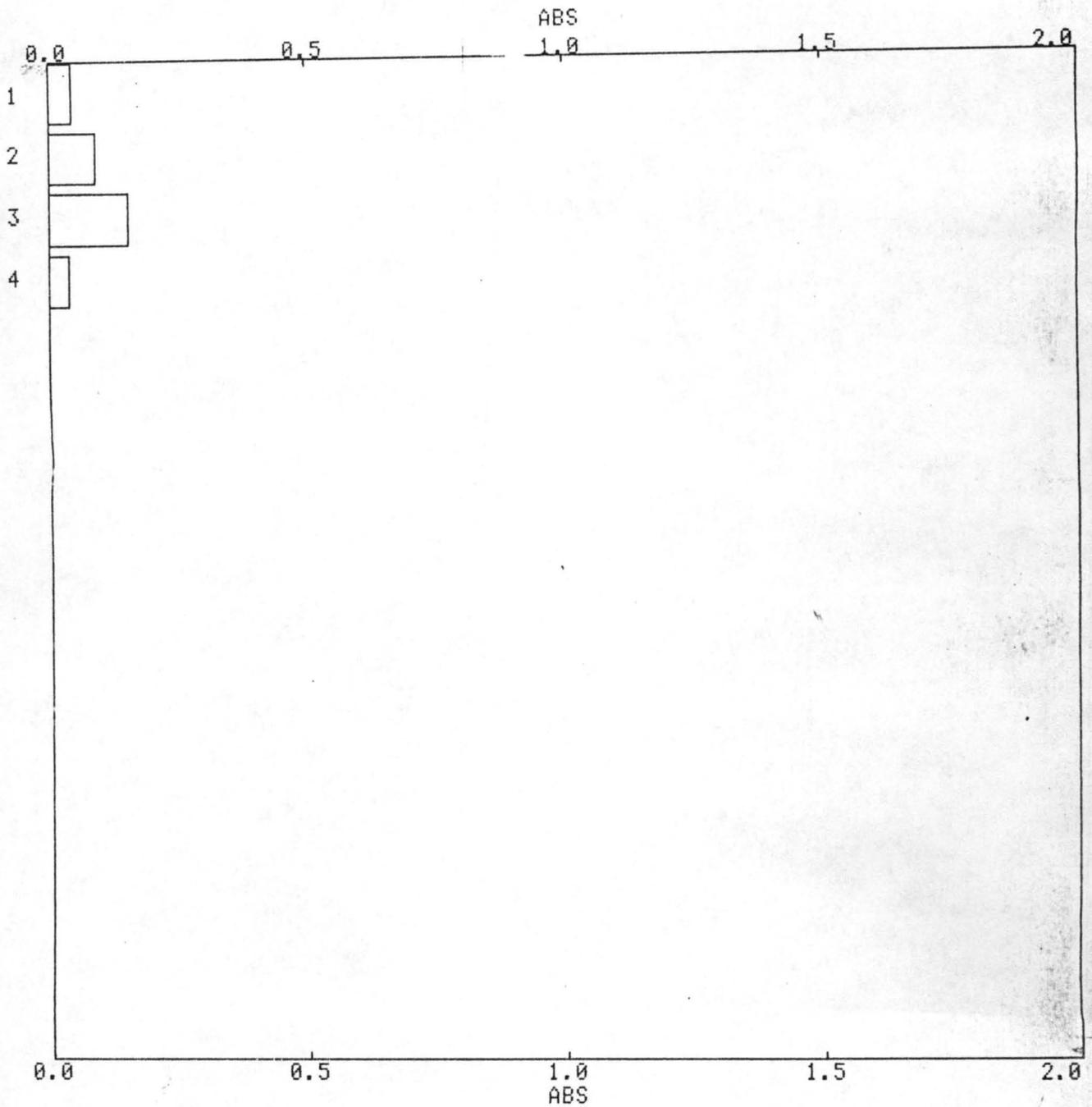
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# Appendix A

I.D. 8 1 \470.0 B2.0 ABS

~SHEABUTTER /Ph



SAMPLE ----- REFERENCE -----  
 CELL PATH ----- OPERATOR -----

Sample No.	Cycle No.	ABS
5	1	0.045
6	1	0.091
7	1	0.156
8	1	0.039

Appendix B

