CHARACTERISATION OF COTTONSEED (*Gossypium hirsutum*) OIL

BY

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FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

NIGER STATE.

FEBRUARY, 2012.

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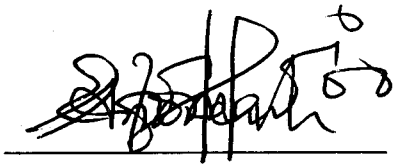
BEING A FINAL YEAR PROJECT REPORT SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF BACHELOR OF ENGINEERING (B. ENG.) DEGREE IN AGRICULTURAL AND BIORESOURCES ENGINEERING, FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA, NIGER STATE.

FEBRUARY, 2012.

DECLARATION

I hereby declare that this project work is a record of a research work that was undertaken and written by me. It has not been presented before for any degree or diploma or certificate at any University or Institution. Information derived from personal communications, published and unpublished work were duly referenced in the text.



28/02/2012

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Date

2006/24101EA

CERTIFICATION

This is to certify that the project entitled "Characterisation of Cottonseed (*Gossypium hirsutum*) Oil" by Efomah, Andrew Ndudi meets the regulations governing the award of the degree of Bachelor of Engineering (B.ENG) of the Federal University of Technology, Minna, and it is approved for its contribution to scientific knowledge and literary presentation.

Engr. Mrs B. A. Orhevba — **Date**

Supervisor

Engr. Dr. P. A. Idah — **Date**

Head of Department

External Examiner — **Date**

DEDICATION

This project is dedicated to Almighty God, the creator of the Heavens and the Earth.

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ABSTRACT

This work contains an experimental study on the extraction and characterization of cottonseed oil using solvent extraction method. During the extraction process, Normal hexane was used as solvent in extracting the oil from the seed in the Soxhlet apparatus. The extracted oil was then subjected to chemical analysis using AOAC (2004) method. The chemical properties of the oil determined were: saponification value, free fatty acid, iodine value, peroxide value and acid value. Also, the physical properties of the oil determined were: viscosity, specific gravity, refractive index, colour, odour, taste and pH. The values obtained for each physiochemical property are as follows; Saponification value (189mgKOH/g), free fatty acid (5.75mgKOH/g), iodine value (94.7gI_2/100g), peroxide value (9.25mEq/kg) and acid value (11.50mgKOH/g). The proximate compositions obtained in percentage are as follows: Carbohydrate (57.06), fat/oil (13.30), crude fibre (0.5), ash (1.5), moisture content (12.21) and crude protein (15.40). The results obtained indicate that cottonseed oil can be used as cooking oil with long shelf life. It can also be used in the soap making industry due to its high saponification value and it can also be used as a lubricant and in the production of biodiesel.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Cottonseed (*Gossypium hirsutum*) is an annual leguminous plant, which belongs to the family *malvaceae*. The Crop is found mostly in the northern part of Nigeria in areas like Zaria, Sokoto and Katsina States. It succeeds nearly in all types of soil but grows best on deep fertile solis with a light Calcium content. Cotton plant is essentially a short day plant. The crop is raised from seeds. It is a bushy upright plant much branched depending on the variety of the plant and growing conditions (Oje, 1993).

Cotton plant is somewhat salt and drought tolerant. this makes it an attractive crop for arid and semi-arid regions. According to the foods and nutrition encyclopaedia, the earliest cultivation of cotton discovered thus far in the Americas occurred in Mexico some 8,000 years ago. The indigenous species was *Gossypium hirsutum* which is today the most widely planted species of cotton in the world constituting about 89.9% of all production worldwide (Wikipedia, 2010).

The plant is a shrub native to tropical and sub-tropical regions around the world including the Americas, Africa, India and Pakistan. The fibre obtained from cotton plant is an important raw material in the clothing and textile industry; while the seed is classified as an oil seed known for its high oil and protein content. Also the meal/cake obtained after

the oil has been extracted is a very important livestock feed as such making this plant an all important crop because no waste is incurred in its uses.

The cottonseed has a similar structure to other oil seeds such as sunflower seed, having an oil bearing kernel surrounded by a hard outer hull (Wikipedia, 2010). The oil obtained from cottonseed undergoes intensive treatment after extraction to reduce the level of gossypol found in untreated cottonseed oil, the consumption of which may produce undesirable side effect. The fatty acid profile of the extracted oil consists of 70% unsaturated fatty acids including 18% monounsaturated (oleic), 52% poly unsaturated (linoleic) and 26% saturated (primarily palmitic and stearic) (Wikipedia, 2010).

Cottonseed oil is a cooking oil extracted from the seeds of cotton plant of various species, mainly *Gossypium hirsutum* and *Gossypium herbaceum*. Cotton grown for oil extraction is one of the big four genetically modified crops grown – around the world, next to soy, corn, and rapeseed (canola), mostly mosanto products.

This oil is manufactured from the delinted, decorticated cottonseed. The cleaned seed meats are first passed through a series of pressure rolls to produce thin flakes, after which the flakes are cooked under steam pressure, which ruptures the oil cells. Subsequently, the flakes are either pressed in hydraulic presses or processed in continuous screw – type expellers which remove the oil under high pressure. The solvent extraction method is gaining acceptance, although not to as great an extent as in soybean oil production. Average yield of oil is about 16 to 17 percent of the cottonseed (Encyclopaedia Americana, 2001). Treatment of the crude oil to produce refined grades is similar to that used for soybean; it involves alkali refining to remove colour bodies and other non-

glyceride impurities, bleaching with activated clays and finally, steaming under vacuum conditions to remove traces of odour (Encyclopaedia Americana, 2001).

The principal end uses of cottonseed oil are similar to those of soybean which are as follows; the production of shortenings and margarine. For both of these products, the oil is hardened by hydrogenation, which increases the melting point, improves stability of the oil against oxidation and also improves the plasticity to produce a desirable solid product. Miscellaneous uses of cottonseed oil includes the manufacture of biscuits, crackers, doughnuts, confections and potato chips and the preparation of ice cream substitutes (mellorines), in which process the oil replaces butter fat. Industrial uses of cottonseed oil includes; alkyd resins for interior paints, special lubricants and soft soaps (Encyclopaedia Americana, 2001).

Cottonseed oil is described by scientist as being "naturally hydrogenated" because the saturated fatty acid it contains are the natural oleic, palmitic and stearic acids. There are many applications of high-oleic acids oils: besides being excellent deep frying oils, they may be used in food systems that must have a high degree of oxidative stability, as vegetable-based lubricants and as feedstock for the oleochemical industry (Corbet, 2003: Mohammed *et al.*, 2003).

There are various methods of extracting oil from oil producing seeds and this to a large extent determines the quality of the oil. The various methods includes; mechanical extraction, traditional extraction, steam and high pressure method, solvent extraction, etc.

- **Mechanical extraction method:** This involves obtaining the oil through crushing the cottonseed using a screw press. Extracted oil is filtered and collected in a settling

tank. Materials removed from the oil called foot, is fed back into the stream of fresh material. Materials discharged from the press called cake, contains 8-10% oil.

- **Traditional extraction method:** This method is similar to the mechanical extraction method but the difference is that instead of a screw press, the oil is extracted by pounding the cottonseed and boiling them for some minutes in water. After which the material is strained through a cloth into a clean container and left overnight to allow the oil to separate from the water.
- **Steam and high pressure method:** This involves making use of high pressure to squeeze out oil from seeds. In this method seeds are subjected to high pressure to enable maximum extraction of oil. The disadvantage with this method is that most active ingredients are destroyed due to high temperature.
- **Solvent extraction method:** This involves using solvents mostly hexane to extract oil from oil bearing seeds. Solvent extraction method is generally seen as less desirable for high end culinary or cosmetic purposes given its chemical nature but for processed foods and bio-fuels, it can be a cost-saving high-yield operation (Fernandez, 2010).

1.2 Statement of the Problem

Cottonseed has a wide range of application; ranging from cotton production for the textile industry to oil production for the confectionary and soap industry. Also the cake meal got after oil extraction is a very important livestock feed to the livestock farmers. Lack of information on the effect of various extraction methods on the oil is an identified problem. As such, relevant data regarding the physiochemical properties of the extracted oil is not readily available which is the major aim of this project.

1.3 Objectives of the Study

The objectives of this project are:

i. To extract oil from cottonseed using solvent extraction method.

ii. To determine some chemical properties of the extracted oil.

iii. To determine some physical properties of the extracted oil.

iv. To determine the proximate composition of the extracted oil.

1.4 Justification of Study

Cottonseed oil is of utmost importance in the soap making, pesticide, insecticide and confectionery industries. This project is aimed at extracting oil from cottonseed using solvent extraction method. The oil obtained will now be subjected to chemical analysis and the results obtained would serve as an important engineering data for the chemical industries and even to the nutritionist.

1.5 Scope of Study

This study is limited to the extraction of oil from cottonseed using solvent extraction method. The oil obtained will be subjected to chemical analysis using AOAC (2004) method. The chemical properties of the oil to be determined would be limited to the following: saponification value, free fatty acid, iodine value, peroxide value and acid value. Also, the physical properties to be determined would be limited to the following: viscosity, specific gravity, refractive index, colour, odour, taste and pH.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Historical Background of Cotton Seed

Cotton is a warm-weather shrub or tree of the *Malvaceae* family, the tribe *Gossypieae,* and the genus *Gossypium* that grows naturally as a perennial but for commercial purposes is grown as an annual crop (Brien and Wakelyn, 2005). Botanically, cotton bolls are fruits (Esau, 1997). The principal domesticated species of cotton of commercial importance are *hirsutum, barbadense, arboreum, and herbaceum* (Percival, 1999). Many different varieties of these species have been developed through conventional breeding to produce cotton plants with improved agronomic properties and with improved cotton fiber and cottonseed properties (Calhoun *et al.,* 1999).

Cotton is a cash crop for more than 20 million farmers in developing countries of Asia and Africa. It is mainly cultivated to meet the basic requirement for cotton fabrics. Cottonseed is a valuable by-product of the cotton plant and for every kg of cotton fiber, 1.65 kg of cottonseed is produced (Rathore, 2007). Global cotton cultivation in 2009-2010 yielded 23.3 million metric tons (MMT) of cotton and around 40 million metric tons (MMT) of cottonseed (Cotton Incorporated, 2010). According to Shekhar (2006), China tops in production of cotton in the world, whereas India stands second. Cottonseed contains approximately 18-25% of oil and 20-25 % high quality protein. The global annual production of cotton seed could potentially meet the total protein requirement of nearly half a billion people for a year at 50 grams per day (Rathore, 2007) but presently, cottonseed is not used in food preparations. It is used in animal feed in regulated manner due to the presence of gossypol. Cottonseed oil is rich in tocopherols which

inhibits rancidity development and thus contribute to its stability resulting in a longer shelf life for the product. Cottonseed oil is a naturally hydrogenated oil and is suitable for the heart due to the presence of palmitic, stearic, mysteric, oleic, linoleic and linolenic fatty acids in sufficient quantities. Cottonseed oil has also gained importance in food preparations due to its higher smoke point (about 232°C) compared to other cooking oils and is good for frying food articles (Brien and Wakelyn, 2005). Refined Cottonseed oil has a mild taste and light golden color. It also finds a number of other non food uses in biodiesel production, in paint industry and as an environmentally accepted lubricant additive to improve the lubricating abilities of the base oil SAE 20 W50 (Ertugrul and Filiz, 2004).

Cotton was first cultivated in the Old World 7,000 years ago (5th–4th millennia BC), by the inhabitants of the Indus Valley Civilization, which covered a huge swath of the northwestern part of the South Asia, comprising today parts of eastern Pakistan and northwestern India. The Indus cotton industry was well developed and some methods used in cotton spinning and fabrication continued to be used until the modern industrialization of India. Well before the Common Era, the use of cotton textiles had spread from India to the Mediterranean and beyond (Wikipedia, 2010).

In Iran, the history of cotton dates back to the Achaemenid era (5th century BC); however, there are few sources about the planting of cotton in pre-Islamic Iran. The planting of cotton was common in Merv, Ray and Pars of Iran. In the poems of Persian poets, especially Ferdowsi's Shahname, there are references to cotton ("panbe" in Persian). Marco Polo (13th century) refers to the major products of Persia, including cotton. John Chardin, a French traveler of 17th century, who had visited the Safavid Persia, has approved the vast cotton farms of Persia.

Although cotton has been grown for its fiber for several thousand years, the use of cottonseed on a commercial scale is of relatively recent origin. In ancient times, it is reported that the Hindus and the Chinese developed crude methods for obtaining oil from cottonseed, using the principle of the mortar and pestle. They used the oil in lamps and fed the remainder of the pressed seed to cattle. For many centuries, however, the use of cottonseed did not develop much beyond that crude stage and was confined to local areas (Wikipedia, 2010).

During the first part of the 19th century, plants in Europe began to crush small quantities of Egyptian cottonseed. These seed had little or none of the linters (short fibers) that remain on American upland varieties of seed after the longer cotton lint is removed at ginning. These fibers, plus the tough hull, made the development of practical methods for processing American cottonseed a difficult undertaking (Wikipedia, 2010).

2.1.1 Cotton Yields in Africa

Although cotton production in Africa is not significant on a global scale, a large number of African countries remain heavily dependent on cotton. For example, cotton accounts for 60% of foreign exchange earnings in Benin (Wikipedia, 2011). Between 1990 and 2007, West African countries reported cotton yield per hectare at approximately 1.1 tons. Beside West African countries, the case of Egypt deserves special consideration. Indeed, production and productivity levels were remarkably higher in Egypt than in any other African cotton producing country. Egypt produces nearly 740.000 tons of cotton over the 1990-2007 periods (about a fifth of the continental production). In terms of productivity, between 1990 and 2007 its yield per hectare was at 2.4 tons, that is to say, Egypt produced per hectare more than double the cotton of the

average West African countries. This performance originates in the fact that cotton is grown under irrigation in Egypt, a way of cultivation that is generally not used in West Africa (National Cottonseed Products Association, 2001).

2.1.2 Cottonseed Oil Industry Development

For many centuries the use of cottonseed oil did not develop much beyond the crude stage and was confined to local areas. During the first part of the nineteenth century, plants in Europe began to crush small quantities of Egyptian cottonseed. The resultant demand for less expensive fats and oils products led businessmen to extract oil from a variety of oilseeds and nuts; included in these endeavors was cottonseed from Egypt and India (Wrenn, 1995).

The sparsely populated United States had adequate supplies of animal fats while Western Europe was experiencing shortages. Nevertheless, the extraction of oil from cottonseed became an attractive solution for another problem. Cotton cultivation had increased rapidly in the United States after Eli Whitney invented the cotton gin in 1793 creating a surplus supply of cottonseed, after that required for planting, fertilizer, and animal feed. The excess cottonseed stocks became huge, worthless, rotting piles that dotted the countryside in the southern states. A few entrepreneurs began crushing cottonseed for oil between 1820 and 1830, but none of these ventures survived more than a few years. Even with all of the early setbacks, cottonseed crushing and refining became a profitable venture in the United States after 1870. Large quantities of cottonseed oil were exported for use in soap manufacture and some found its way into cooking, salad oils, and the oleomargarine product developed by a French chemist. Adulteration of olive oil with cottonseed oil resulted in import tariffs in the olive growing countries and complete

expulsion from Italy in 1883. These restrictions curtailed cottonseed oil exports to create an oversupply of cottonseed oil, which again decreased the value (Jones and King, 1996).

2.2 Cultivation of Cotton

2.2.1 Climatic Requirement

The seeds adapt to a wide range of climatic conditions for germination and growth. Cottonseed is highly photoperiodic, and this is a major constraint on selection of warm season (tropical) crops. Cotton can be profitably grown in regions with rainfall of 850-1100mm. However, economic yields cannot be realized with rainfall less than 500mm. Rains during boll bursting periods spoil the cotton quality. Cotton requires at least 450-500mm of water for ET in order to give higher yields. During flowering and fruiting, temperature of 26°C-32°C are desirable during the daytime, but the night should be cool. In general, temperature above 35°C are not desirable, however, when the moisture supply is favourable, the cotton plant is capable of enduring without permanent injury, very high temperature (up to 45°C) for short periods. If this high temperature persists for several days, however, the yields will be adversely affected. All the wild species of *Gossypium* are short day plants and do not flower so long as the day length exceeds twelve hours. Stanbury and co-workers (Stanbury *et al.*, 1954) found that high temperatures during seed development influenced saturated fatty acid formation. They determined that the average iodine decrease (reduction in unsaturated fatty acids) per °F temperature was 0.76 during boll development and 1.172 during seed development.

2.2.2 Soil Requirement

Cotton has a wide range of soil adaptation and is grown on a great variety of soils. Highest yields of cotton are usually obtained on alluvial soils. High yield of cotton is dependent on favourable air and moisture regime in the soil-hence the importance of soil structure and texture.

For a deep-rooted crop such as cotton, soil depth is also an important factor and shallow soils are not suitable. Cotton is not unduly sensitive to soil reactions. It can be grown on a variety of soils with pH ranging from 5 to 8 and above. Cotton is generally considered as fairly tolerant to salinity. Under moderate rainfall conditions cotton is grown in retentive clayey loams.

2.2.3 Weed Control

Young cotton seedlings are unable to compete with many fast growing tropical weeds and their control at this period is most important. Since it is difficult to cultivate cotton seed immediately, every effort should be made to reduce the weed population before planting. Cotton Plant depends more on their vertical than lateral root, and over deep weeding in operations are not as damaging in terms of yield production as might be expected (Willcut *et al.*, 1987). This factor should be considered when weighing the advantages of mechanical operations in weeding mature crops against possible yield reduction caused by weed competition.

Rotary hoes or finger weeders give excellent results in row crops with little damage to plant and two or three cultivation are usually sufficient. Flame weeder can be effective but requires skilled operators and a steady supply of fuel. The chemical control of weeds using compound available for pre emergence use includes bentazone, butralin, chloramben, fluchoralin, glyphosphate, linuron, metribuzin, nitralde, prometryne, and trifluralin.

2.2.4 Cottonseed Handling and Storage

Once a cotton boll opens, the cottonseeds within are susceptible to deterioration. The living seeds respire, producing carbon dioxide and heat. Other biological processes occur in the seeds as well. The rate of hydrolysis is dependent on temperature and moisture, but the hydrolytic enzymes may be inactivated by heat. Oxidation of the fatty acids in the oil can result from heating (Johnson, 1981).

Mauney and Stewart (1986) have considered cottonseed development. Another factor in cottonseed storage is the control of mould development on the seeds. Cottonseed is particularly susceptible to the fungus *Aspergillus flavus*, which produces aflatoxin. Aflatoxin formation in cottonseed is generally, but not exclusively, initiated in the field, rather than during storage (Wikipedia, 2011).

Ideally, cottonseed should be stored at a moisture content of less than 10% (Norris and Swern, 2001). Dehulled seeds should contain no more than 9% moisture and 1% FFA (Johnson, 1981). Prior to storage, cottonseeds must be sampled for moisture analysis. Seeds with 10–11% moisture may be stored immediately, but seeds with higher moisture require additional drying. Drying may take place at either ambient temperatures or up to 104.5^{0}C (220^{0}F), to 12% or less. There is no benefit to drying the seeds below 9% moisture. Although dryers may not be needed every season. The most common type of cottonseed storage facility is the seed house. However, an air-cooling system is vital to the successful storage of cottonseed. The temperature of the seeds is dependent on the ambient temperature and degree of ventilation in the storage area (Rusca and Gerdes, 1942). As the seeds are respiring, heat can build up, particularly if the seeds have a high moisture or FFA level (Norris and Swern, 2001). Overheated seeds must be cooled

› below 60^0F (15.6^0C) to prevent further deterioration. Storage for over one year is possible ›rovided that the seeds are held under adequate conditions.

Plate 2.1: **Cotton bolls ready for harvest**

2.2.5 Cotton Pests

Cotton insects are the principal cause of yield losses. Estimates indicate that the yield losses due to insect infections would amount to almost 15% of world annual production (Michael *et al.*, 2004)

More than 1300 different species of insect pests attack the crop. Among the most common and endogenous species found in cotton fields are:

• The pink bollworm (*Pectinophora gossypiella*) was first described in 1843 by W.W. Saunders as *Depressaria gossypiella*, from specimens found to be damaging cotton in India in 1842. The pink worm withdraws nutrients from the inside of the cottonseed and may cause serious yield losses. Although the most severe infestations have occurred in Africa and India, the pink bollworm has been recorded in nearly all cotton-producing countries and is a key pest in many of these areas. Infestations may be reduced by some management tactics, including plantation treatment and destruction of the infested crop (Michael *et al.*, 2004).

• The boll weevil (*Anthonomus grandis*), also known as bollworm, is most common in American cotton plantations.

• The Egyptian (spiny) bollworm (*Earias insulana*) and the red bollworm (*Diparopsis castanea*) feed on the developing cotton bolls.

• Cotton stainers (*Dysdercus superstitious*) attack maturing cotton bolls and seeds. They may cause the staining of the lint. In addition, feeding wounds may allow the entry to the boll of saprophytic fungi (organisms which draw nutrients from the host, but do not harm it, contrary to parasites).

• Other insect pests of cotton, such as the white flies (*Bemisia gossypiella*), may adversely affect lint quality and yield potential. They suck sap from leaves and pose the most serious threat in India and Africa.

• The cotton aphid (*Aphid gossypii*), also known as the melon aphid, infests the cotton seedlings. Cotton aphids are among the most injuring insects found in cotton. They suck sap from leaves

and secrete honeydew on the undersides of leaves. Honeydew secretions may burn the leaves and interfere with photosynthesis. In addition, aphid is a vector of viruses and a carrier of other insects. In Africa, aphid infestations are among the most injuring insect pests in terms of economic yield lost (Michael *et al.*, 2004).

• Nematodes: There are approximately 128 species of nematodes associated with cotton. Five parasitic forms pose the most serious threat to the crop, including the *Meloidogyne incognita* (or root knot nematode) and the *Rotylenchulus reniformis* (or reniform nematode). These two species can become serious pests (in the United States, particularly in the State of Virginia, they accounted for 99% of the damage caused by cotton parasitic nematodes). These parasites live in the soil (the root knot nematode favors rough soils) and withdraw nutrients from the plant roots. Symptom patterns associated with nematodes include stunting, potassic deficiency or early maturity. Nematodes can reduce yields (in Alabama, United States, yield losses are estimated to average 10% or 20%, but can peak to 50% in arenaceous dry soil). Also, depending upon the stage of development of the infested crop, they can hamper the quality of cotton. Root knot nematodes do produce plant damage symptoms that are rather easy to recognize, such as the yellowing or whitening of normally green plant tissue because of a decreased amount of chlorophyll. Damage symptoms caused by other kinds of nematodes (for example, the reniform nematode) are more difficult to detect, since they are generally small and sparse. Besides the direct damage, nematodes are also an important factor in the incidence of Fusarium and other wilts of cotton. Nematodes may be controlled by cultural practices, such as crop rotations, soil tilling, and use of resistant varieties, or by chemical treatment through nematicides. The two types of nematodes seldom coexist in the same fields.

2.3 Cottonseed products.

2.3.1 Cottonseed Meal

Cottonseed meal is the second most valuable product of cottonseed, usually accounting for over one-third of total product value. It may be sold in the form of meal, cake, flakes, or pellets. Cottonseed meal is used principally as feed for livestock and is usually sold at a 41% protein level (Calhoun *et al.*, 1995). Its major value is as a protein concentrate. One of the essentials of a ration is good quality protein which is necessary to build muscles, nerves, blood, internal organs, hair, and skin. Meat, milk, and wool--the major products of the livestock industry--are all rich in protein.

Cottonseed meal may be used to some extent in the rations of all classes of livestock. It is sufficient as a sole source of protein for mature ruminants such as beef cattle and sheep and can provide much of the protein for dairy cows. Since it is a natural protein source its nitrogen is effectively utilized and there is little danger of excess ammonia being produced in the rumen or stomach of these cud-chewing animals as sometimes occurs when feeding synthetic protein materials. High quality cottonseed meal, used correctly as an ingredient of properly formulated swine and poultry rations, improves economy and efficiency (Jones *et al.*, 2005).

2.3.2 Cottonseed Hulls

Cottonseed hulls are removed from whole seed. The hull is mainly hemicelluloses and lignin compounds with a nearly pure cellulose linter fiber attached (Tharp and Bailey, 1948). No

pigment glands have been reported on the hull fiber or linter fiber fractions. The residual oil and protein that may be present from the decortication or removal of the hull from the cottonseed meats may contain some free gossypol. Advances in mechanical and air separation techniques over the last 20 years have minimized the amount of residual oil and protein found in cottonseed hulls. This results in hulls typically reported as having less than 0.049 % free gossypol content (Forster and Calhoun, 1995). Pelleting hulls for transportation and convenient handling purposes can reduce this small free-gossypol level even further. Pelleted hulls have been shown to have the same feeding characteristics as loose hulls (Brown, 1991). Due to the low levels of gossypol found in hulls, gossypol poisoning from feeding hulls alone is not biologically possible.

2.3.3 Cottonseed Linters

Cottonseed linters, the short fibers removed from seed as the first step in processing, are sometimes referred to as "the fabulous fuzz." Through mechanical or chemical conversion, they enter a wider variety of end use products than any of the other products of cottonseed (Wikipedia, 2011).

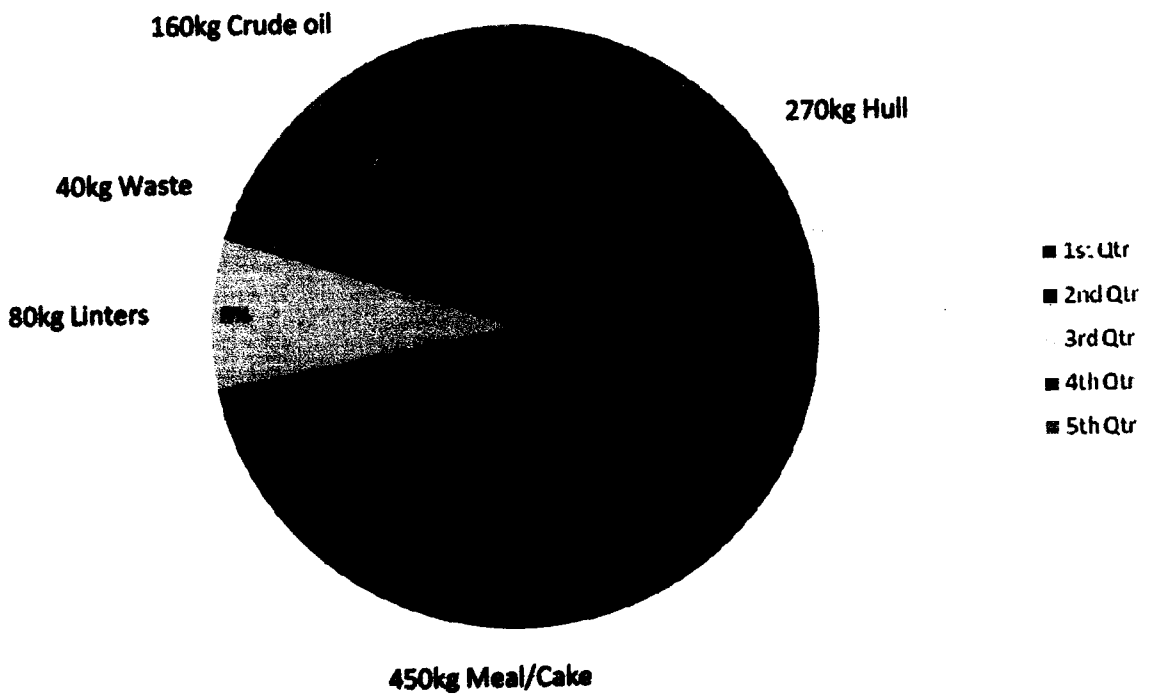

Fig.2.1: Cottonseed Products Yield Per ton of Seed Crushed.

Source: National Cottonseed Products Association, (2001).

2.3.4 Cottonseed Oil

Crude cottonseed oil from the mill requires further processing before it is used in food. The first step in this process is refining, which is carried out by warming the oil and adding sodium hydroxide. This alkali combines with a portion of the oil to form what is known as soap stock or foots. The soap stock, together with impurities that may be present, is then separated from the oil

by means of a high-speed centrifuge (Wikipedia, 2011). The refining process also removes darker coloring materials present, leaving clear yellow oil.

In addition to flavor stability, cottonseed oil also has superior nutritive qualities. Cottonseed oil has no cholesterol (National Cottonseed Products Association, 2001). All vegetable oils contain fatty acids that can be either harmful or helpful to our health and this is presented in Table 2.1.

Table 2.1: Principal Fatty Acids Composition (%) found in Cottonseed Oil

Fatty Acids	Cottonseed Oil %
Oleic Acid	18
Linoleic Acid	52
Palmitic Acid	16
Stearic Acid	10
Other Lower Fatty Acids	4

Source:www.wikipedia.org/wiki/cottonseed-oil

The oxidative stability of vegetable oils depends principally on the following;

- The source of the vegetable oil.
- The calculated iodine value of the oil.
- The total double bonds present in the oil.

The oxidative stability of various vegetable oils is presented in Table 2.2

Table 2.2: Vegetable Oils Oxidative Stability

Rating	Inherent Oxidative Stability	Vegetable Oil Source	Calculated Iodine Value	Total Double Bonds
Best	0.3	Coconut	9.6	11
	0.4	Palm Kernel	17.2	20
	1.5	Palm	52.2	61
	1.6	Olive	82.4	96
	3.7	Peanut	97.6	113
	5.3	Canola	116.5	135
	5.8	Cottonseed	112.4	130
	6.5	Corn	128.4	148
	7.2	Sunflower	136.2	157
	7.5	Soybean	132.5	153
Worst	8.0	Safflower	146.1	169

Source: Firestone, 1999.

Oils high in saturated fatty acids have been shown to raise blood serum cholesterol levels, which may lead to heart attacks. Polyunsaturated oils have been shown to reduce serum cholesterol levels. All the domestically grown vegetable oils, including cottonseed oil, have one thing in common-they are high in polyunsaturates, moderate in monounsaturates, and low in saturated fat. Cottonseed oil has a 3:1 ratio of unsaturated to saturated fatty acids. This meets the recommendations of many health professionals and allows cottonseed oil to be used without further processing (Wikipedia, 2011).

Cottonseed oil is under scrutiny by some nutritionists, who deem it too high in saturated fat and too low in monounsaturated fat (Andrew, 2010). Detractors say that cottonseed oil may contain natural toxins called gossypol and unacceptable high levels of pesticide residue. Gossypol is a biologically-active yellow polyphenolic compound produced by cotton and other members of the order *malvaceae*, such as okra (Jones and King, 1996). The natural toxin, gossypol is eliminated in the refining process of commercially edible cottonseed oil. The Food and Agricultural Organization of the United Nations has documented the 'lack' of appreciable residues in cottonseed and cottonseed oil.

2.3.4.1 Effect of Seed Storage on Cottonseed Oil

Cottonseed oil from improperly stored cottonseeds will develop a dark color that requires additional processing. Boatner (Boatner and Bailey, 1948) noted that the following conditions favour the development of colored or "reverted" oil: harvest of immature (bollie) seeds, extremes of moisture or temperature, or other damage to the cotton plant during cultivation or harvest. Oxidation of gossypol and other pigments has been proposed as the chemical cause of

the colour (Norris and Swern, 2001). To avoid this color change, processors avoid intermingling seed known to be damaged or immature with good quality seeds. Seeds with higher moisture or higher than normal free fatty acid (FFA) content are usually processed first. Only seeds with good quality are stored to be processed throughout the crop year before the next crop season begins. The moisture, temperature, and FFA level of seeds in storage are monitored periodically and serve as the basis for further processing and handling decisions.

2.4 Feeding Value of Whole Cottonseed

Over the past 20 years, the percentage of whole cottonseed (WCS) fed directly to cattle has increased dramatically. Although its bulky physical form makes it a rather inconvenient feedstuff to handle. Dairy producers have increasingly embraced the use of whole cottonseed (WCS) as a source of energy, protein, and fiber in lactating dairy cow diets. Levels, as high as 25% of the ration, are fed with mostly positive results (Coppock and Wilks, 1991). Recent research has shown the benefits of feeding limited amounts of oilseeds (About 4% of total dry matter intake) to beef cows in marginal body condition before the breeding season. For example, feeding about 3.5 pounds of WCS daily to a mature, 1,100 pound cow will help many cows begin cycling as early as possible. Levels containing up to 50% whole cottonseed in the concentrate portion have been evaluated for beef cattle growing rations. After 68 days of receiving the diet, yearling cattle fed 50% WCS began scouring when they consumed 12 pounds of WCS per head daily. The concentration of whole cottonseed in the diet was reduced to 25%, then gradually increased to 40% by the end of the 112- day trial, and no further digestive disturbances were reported. Arizona research (Hale *et al.*, 1983; Swingle *et al.*, 1983) determined that increasing levels of WCS in beef cattle finishing diets resulted in a concomitant decrease in the energy utilization from WCS, and that a level of 20% showed a small advantage in cattle performance.

2.5 Chemical Composition and Nutritional Value of Cottonseed

The oil and protein together account for about 60% of cottonseed by weight; protein 40% and oil 20%. The reminder consist of 30% carbohydrates and 5% ash, phosphotide, sterols and other constituent are also present as minor constituent also present as minor constituents. The principal soluble carbohydrate, saccharine, of mature cotton are the disaccharide, sucrose, (range 2.5 – 8.2%), the disaccharide raffinose (0.1- 1.0%) compose of one sucrose molecule connected to one molecule of galactose, and the tetrasaccharide stachyose (1.4- 4.1%) connected of one sucrose molecule connected to two molecules of galactose. A variation ranging from 13.9 – 23.2% in oil and 32.4 to 50.2% 0n protein has also been recorded. The variation in protein and oil content in cottonseed is due to the locality where cottonseed is grown and also the cultivar of cottonseed cultivated. The chemical composition of refined cottonseed oil is shown in Table 2.3

Table 2.3: Chemical Composition of Refined Cotton oil

Parameter	**Value**
Specific gravity at 25°C	0.915-0.921
Iodine Value	109-120
Saponification Value	190-198
Peroxide Value	5.0
Colour Gardner	AOCS Td la-64
Appearance	Golden Yellow, Bright and Clear Oily Liquid
Odour	Bland

Source:www.welch-holme-clark.com/cottonseed_oil_refined_html.

The proximate composition of cottonseed part is shown in Table 2.4

Table 2.4: Proximate Composition of Cottonseed Part

Part	%seed crude protein	%crude fat	%N.free extract	% Ash
Seed	40.40	22.30	31.90	4.90
Cotyledons	43.40	24.30	21.40	5.00
Hypocotyl	40.80	12.00	42.40	4.50
Hull	9.00	0-.90	86.20	4.00

Source: National Cottonseed Products Association. (2001)

2.5.1 Carbohydrate

Cottonseed contains about 30% carbohydrate. The soluble carbohydrate in cottonseed is about 5% sucrose, 1% raffinose and insoluble fiber, 20 % seeds of cotton do however contain starch. The insoluble carbohydrate includes the pectin, cellulose and hemicelluloses that are associated with cell walls in cottonseed. This is also the dietary fiber component which is increasingly recognized as an important part of the human diet. The fiber is more prevalent in cottonseed coats (hull) than in cotyledons (Wikipedia, 2011).

2.5.2 Protein

Cottonseed is an excellent source of dietary protein. Cottonseed protein constitutes about 41% of the total solids and is known to play a vital role in global food processing. It is however important to know that there are significant variations in the protein content of different cultivars of cottonseed: protein content may range as low as 35% to as high as 44% with some cultivar even having a protein and oil content in cottonseed. According to Olajide and Igbeka (2003) cultivar possessing higher protein, is mainly composed of globules, which have been characterized by their solubility in salt solution. pH of the water has been reported to strongly affect the solubility of cottonseed protein in water. Close to about 80% of the protein in raw seeds or unheated meal can be extracted at neutral or alkaline pH. As the acidity is increased, solubility drops rapidly and a minimum is observed at pH 4.2 – 4.6, this is the bioelectric region of cottonseed protein taken as a whole.

2.5.3 Lipids

The lipids of cottonseed (crude cottonseed oil) consist typically of 96% triglyceride, 2% phospholipids, 1.6% unsaponifiable, 0.5% free fatty acids and minute amount of carotene pigments. Phospholipids generally are substances that are found on the surface of the oil bodies. The relatively high content of phospholipids in cottonseed is due to the small size of the oil bodies, resulting in a layer surface per unit weight of lipids. Although the phospholipids fraction of cottonseed contains a number of distinct substances, the technical term "lecithin" is used to name the entire fraction (Wikipedia, 2011).

Lecithin is known as an excellent emulsifier and has many foods, medical and industrial uses. Due to this emulsifying property, the bulk of phospholipids are removed from the crude oil before refining can take place. The process of removing the bulk or phospholipids is referred to as "Degumming", simply because the phospholipids are separated as hydrate gums.Tocopherols and sterol make up the composition of the unsaponifiable fat content. This unsaponifiable fat content, through the process of deodorization are partially removed.

The process of refining and bleaching helps to remove the free fatty acids and pigment, thus bringing about a reduction in the concentration of triglyceride usually less than 1% in the refined oil. The variety of cottonseed and growing conditions affects the fatty acid composition of cottonseed oil. The unsaturated portion account for over 80% of the cottonseed oil and is generally referred to as semi-drying oil due to its high content of linolenic acid. The presence of linoleic as main characteristic acids is the oxidative rancidity property on cottonseed oil. (Willcut *et al.*, 1987).

2.5.4 Mineral

Cottonseed is a good source of fiber, calcium, iron , zinc, phosphorus, magnesium, thiamine, riboflavin, nicotinic acid, and folacin. Fermented cottonseed food has been reported to contain vitamin B12, either naturally or by contamination, but this may be mostly in the form of analogous and cannot be considered reliable sources of vitamins. Foods using the whole cottonseed, such as temph (fermented cottonseed cake), ratio (fermented cottonseed) and miso (fermented cottonseed paste),retain much of the fibre in the whole cottonseed. Fibre (7 – 10g) has been shown to have a modest beneficiary effect in regulating blood glucose level in diabetes,

whereas larger amount (25g) have been shown to have lower total cholesterol and low density lipo protein (LDL) cholesterol. Cotton-fibre contains an appreciable amount of soluble fibre. Calcium content of cottonseed product is somewhat unclear. Values reported by the US department of Agriculture (USDA) are higher than one would expect.

2.5.5 Vitamins

Cottonseed constitute a good source of water-soluble vitamins as shown in Table 2.5:

Table 2.5: Composition of Vitamins in Cottonseed

Vitamin	Content (mg)
B-carotene	0.30- 2.40
Thiamin	11.0 – 17.50
Riboflavin	2.3
Niacin	20.0 – 25.90
Panthothenic acid	12.00
Pyridoxine	6.4
Biotin	0.6
Folic acid	2.30
Cholin	3.40
Ascorbic acid	0.20

Source: National Cottonseed Products Association (2001).

2.6 Twenty facts about cottonseed oil

1. Cottonseed oil is extracted from cottonseed. Cotton has long been known as nature's unique food and fiber plant. It produces both food for man and feed for animals in addition to a highly versatile fiber for clothing, home furnishings and industrial uses.

2. Cottonseed oil has been a part of the Americas diet for well over a century. Until the 1940's, it was the major vegetable oil produced in the United States. Now, with annual production averaging more than 1 billion pounds. Cottonseed oil ranks 3rd in volume behind soybean and corn oil representing about 5 – 6% of the total domestic fat and oil supply.

3. Cottonseed oil has many applications; as a Salad oil, it is used in mayonnaise salad dressings, sauces and marinades. As a cooking oil, it is used for frying in both commercial and home cooking. As a shortening or margarine it is ideal for baked goods and cakes .

4. Cottonseed oil is primarily used in the US as a salad or cooking oil. About 56% is consumed in that category while about 36% goes into baking and frying fats, and a small amount into margarine and other uses.

5. Cottonseed oil has a mild, nut like taste. It is generally clear with a light golden color but like most oils, the degree of color depends on the amount of refining. Clear, colorless oils are not necessarily better oil, but may have been refined more severely.

6. Cottonseed oil is often used as the yardstick for measuring flavor and odor qualities in other oils.

7. Cottonseed is one of the few oils considered acceptable for reducing saturated fat intake.

8. Cottonseed oil is among the most unsaturated oils. Others include safflower, corn, soybean, canola, and sunflower seed oil.
9. Cottonseed oil has a 2:1 oil ratio of polyunsaturated to saturated fatty acids. Its fatty acid profile generally consist of 70% unsaturated fatty acids including 18% monounsaturated (oleic) and 52% polyunsaturated (linoleic) and 26% saturated.(primarily palmitic and stearic)
10. Cottonseed oil is rich in tocopherols these natural anti oxidants, which have varying degree of Vitamin E activity, also contribute to its stability, giving products that contain it, a long shelf life.
11. Cottonseed oil is described by scientists as being "naturally hydrogenated" because of the level of oleic, palmitic, and stearic acids which it contains. These make it a stable frying oil without the need for additional processing or the formation of trans fatty acids.
12. Cottonseed oil does not have to be as fully hydrogenated for many purposes as some of the more polyunsaturated oil. When it is partially hydrogenated, however, its monounsaturated fatty acids actually increases, when hydrogenated to a typical iodine value about 80, for example its fatty acid profile shifts to 50% monosaturated, 21% polysaturated, and 295 saturates all well within current diet/heath guidelines.
13. Like all major food crops, cottonseed production is regulated by food protection agencies of the federal government's strict standards for purity.
14. Refined and deodorized cottonseed oil is one of the purest food products available, few food can be as highly cleaned and refined and still maintain their nutritional quality.
15. Cottonseed oil's light, non-oily consistence and high smoke point make it most desirable for cooking, "stir fry" and other oriental dishes as well as for frying fish.

16. Cottonseed oil is favorite for salad oil, mayonnaise, salad dressing, and similar product because of its flavor stability.
17. Unlike some oils, cottonseed oil does not deteriorate or "revert" rapidly in flavor when used at high temperatures.
18. In addition to oil many products from cotton seed are part of our daily life, cellulose and cellulose derivatives from cottonseed linter fiber are used as food ingredients.
19. Cottonseed can be found as an ingredient in many food products and is available on the grocery shelf only in limited areas.
20. Because cottonseed oil is America's original vegetable oil, it has been the standard to which other oils are compared

Source: (National Cottonseed Products Association, 2001).

2.7 Relationship between Cottonseed oil and other seed oils

Cottonseed serves as a good oil producing seed and is also used in the eastern part of Nigeria for making local soup delicacies. Some seeds also has the ability to produce oil with similar extraction methods like cottonseed. Such seeds includes; Soybean and sesame seed.

2.7.1 Soybean Oil (*Glycine max*)

Soybean oil is a vegetable oil extracted from the seeds of the soybean (*Glycine max*). It is one of the most widely consumed cooking oil. Being one of the drying oil it is also used as a base for printing inks and oil paints (Wikipedia, 2010).

To produce soybean oil, the soybeans are cracked, adjusted for moisture content, heated to between 140°F and 190°F, rolled into flakes, and solvent-extracted with hexane. The oil is then refined, blended for different applications, and sometimes hydrogenated. Soybean oils, both liquid and partially hydrogenated, are sold as "vegetable oil," or end up in a wide variety of processed foods. Most of the remaining residue (soybean meal) is used as animal feed. In the 2002–2003 growing season, 30.6 million tons of soybean oil were produced worldwide, constituting about half of worldwide edible vegetable oil production, and thirty percent of all fats and oils produced, including animal fats and oils derived from tropical plants(Wikipedia, 2010).

100g of soybean oil has 16g of saturated fat, 23 g of mono unsaturated fat, and 58g of poly unsaturated fat. The major unsaturated fatty acids in soybean oil triglyceride are 7–10% alpha Linolenic-acid (C-18:3); 51% linoleic acid (C-18:2); and 23% oleic acid (C-18:1). It also contains the saturated fatty acids 4% stearic acid and 10% palmitic acid which are long chain saturated fatty acids (Wikipedia, 2010). Soybean oil is mostly used for frying and baking. It is also used as a condiment for salads. Soybean oil will slowly harden on exposure to air, forming a flexible, transparent, and waterproof solid. For this reason, it is used in some printing ink and oil paint formulations. While soybean oil has no direct insect repellent activity, it is used as a fixative to extend the short duration of action of essential oils such as geranium oil in several commercial products (Barnard and Xue, 2004).

2.7.2 Sesame Seed Oil (*Sesamum indicum*)

The sesame fruit serves as a symbol for wealth. When the fruit capsule opens, it releases a real treasure: the sesame seeds. However, a great deal of manual work is necessary before this point is reached. That is why sesame is hardly ever cultivated in western industrialised agricultural areas. The sesame seeds are protected by a capsule, which does not burst until the seeds are completely ripe. The ripening time tends to vary for this reason, the farmers cut plants by hand and place them together in upright position to carry on ripening for a few days. The seeds are only shaken on a cloth after all the capsules have opened.

Sesame oil is an edible vegetable oil derived from sesame seeds. Sesame oil is composed of palmitic acid 12.0%, palmitoleic acid 0.5%, stearic acid 6.0%, oleic acid 50.0%, linoleic acid 50.0%, linolenic acid 1.0%. Sesame oil is a source of vitamin E. The extraction of sesame seed is not a completely automated process. There are many variations in the colour of sesame oil: Cold pressed sesame oil is pale yellow, white Indian sesame oil (Gingerly or till oil) is golden, Chinese and Korean sesame oils are commonly a dark brown colour. This dark colour and flavour is derived from roasted sesame seeds. Cold pressed sesame oil has a different flavour than the toasted oil, since it is produced directly from raw, rather than toasted seeds. In East Asian countries, different kinds of hot- pressed sesame oil are preferred (Wikipedia, 2010).

Despite sesame oil's high proportion (41%) of polyunsaturated (omega-6) fatty acids, it is least prone, among cooking oils with high smoke points, to turn rancid when kept in the open. This is due to the natural anti-oxidants present in the oil. Light sesame oil has high smoke point and is suitable for deep-frying, while heavy (dark) sesame oil (from roasted sesame seeds) has a slightly lower smoke point and is unsuitable for deep-frying. Instead it can be used for the stir-

frying of meats or vegetables, or for making an omelette. The sesame oil can also be used for the following: Body massage, hair treatment, food manufacture, drug manufacture and industrial uses (Microsoft Encarta, 2009).

2.8 Methods of Extraction

Cottonseed oil can be obtained through pressing (crushing) of the seed kernel by cold pressing or through the process incorporating temperature controls. The oil is also extracted traditionally by kneading and alternate wetting with hot water until the oil in the dough-like materials begins to ooze out (Olaifa and Adenuga, 1998).

Cottonseed oil can also be obtained by solvent extraction method using hexane as the solvent with the aid of the soxhlet apparatus. The mechanical method can also be used to extract the oil from the seed. The quality of the oil is affected by the method of extraction (Wikipedia, 2011).

2.8.1 Solvent Extraction Method

Solvent extraction method entails obtaining oil from oil bearing seeds with the use of solvents like hexane and then recovering the oil from the oil/hexane solution through distillation. This method is accomplished by the use of soxhlet apparatus. This apparatus was designed by Franzon Soxhlet in 1939, first with automated extraction apparatus and later in the intervening years several modifications of the apparatus have been developed. In the soxhlet apparatus extraction, the solvent used must meet several requirements:

- They must dissolve maximum amount of desired components and minimum amount of undesired materials.

- The solution must be easy to separate from the undissolved substance. In the extraction of solids, this separation is usually not difficult, but in liquid extraction a solvent must be chosen that will separate quickly and completely from the liquid being extracted.
- The solvent must be easy to separate from the extracted material, usually by distillation without affecting the quality of the product(Microsoft Encarta, 2009)

Solvent extraction is generally seen as less desirable for high end culinary or cosmetic purposes, given its chemical nature, but for processed foods and bio-fuels it can be a cost saving high-yield operation (Fernandez, 2010).

2.8.2 Mechanical Extraction Method

This method entails extracting oil from oil bearing seeds with the use of mechanical screw-presses. The NIFOR mechanical screw-press is the latest used by the small-scale oil processing industry in Nigeria. This consists of a perforated tube inside which a transport screw rotates. The pitch of the screw flights gradually decreases towards the discharge end, to increase the pressure on the pulp as it is carried through the barrel (Wikipedia, 2011). The press outlet is more or less closed by a cone that regulates the pressing pressure. The worm transports and gradually compresses the macerated seeds. Released oil drains through the perforations in the tube. The press is mounted directly below a feed conveyor, which is fed by gravity by the horizontal digester. The body of the feed conveyor is perforated to allow oil release in the digester to drain away.

2.8.3 Traditional Extraction Method

Pounding and oil extraction are the most tedious and essential operations in traditional method of oil extraction. Traditional method of oil extraction consists of:

- Seed maceration by pounding cooked/ soaked seeds in large wooden or concrete mortars.
- Steeping the pounded seed marsh in hot or cold water.
- Removing fiber and other debris in small baskets and hand squeezing.
- Filtering out residual fiber from the oil/water emulsion in perforated metal colanders.
- Boiling and skimming the oil from the oil/water mixture.
- Drying the recovered oil.

Tribesmen in Oman used this technique to extract oil from *Moringa peregrina* seed with some success (Folkard and Sutherland, 2005).

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

3.1.1 Sample Collection and Preparation of Samples

The cottonseeds used as sample for the analysis were obtained from the seed unit, Institute for Agricultural Research, Ahmadu Bello University, Samaru, Zaria, Kaduna State, Nigeria. The variety of cottonseed used is Samcot 11(bar 36). This variety is a long staple cotton having a yield of 2.0 – 3.0 tonnes/ hectare and a maturity of 130 – 140days. The collected seeds were properly cleaned so as to remove farm residues and other impurities like stones. A picture of the collected seeds is shown in Plate 3.1

Plate 3.1: **Cottonseeds**

3.1.2 Apparatus Used in the Extraction and Characterisation of Cottonseed Oil

Soxhlet apparatus (Pyrex, England).

Thomas Willey milling machine (Model ED-5).

Muslin Bag

Hydraulic Screw press (Apex 438510 model 515145 by Dorkent)

Condenser (Pyrex, England).

Electronic Hotplate (Gallen kamp)

Filter paper

N – Hexane

Viscometer.

Refractometer.

Silica dishes.

Petri dishes.

Weighing balance (Analytical balance machine by Salter, model 250).

Spatula.

Pipette (Pyrex, England).

Measuring Cylinder (Pyrex, England).

Kjeldahl digestion block (Model No. 451699, England.)

Burette (Pyrex England).

Flat bottom flask (Pyrex, England).

Retort stand.

Flame photometer

Beaker (Pyrex, England).

Electric air oven (Gallen kamp furnace, model No. 2346AA).

Test tube (Pyrex England).

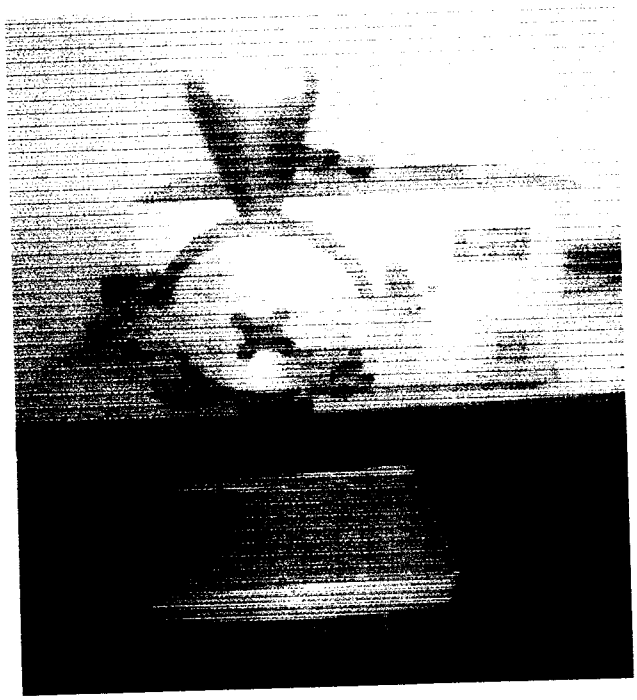

Plate 3.2: **Thomas Willey Milling Machine.**

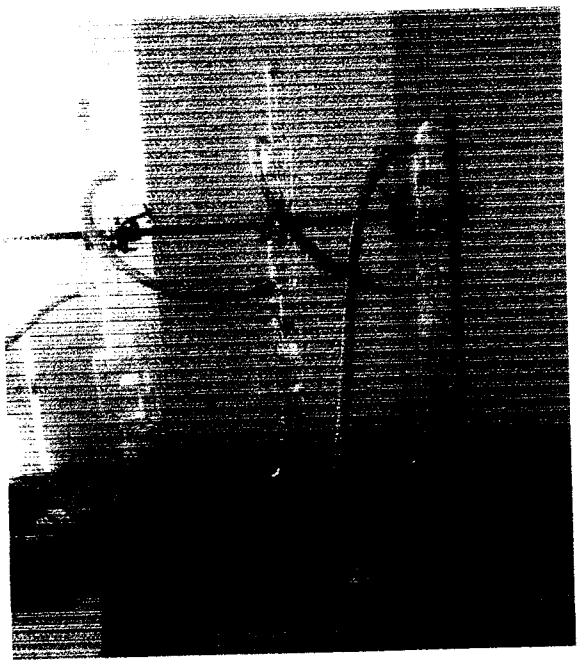

Plate 3.3: **Soxhlet Apparatus**

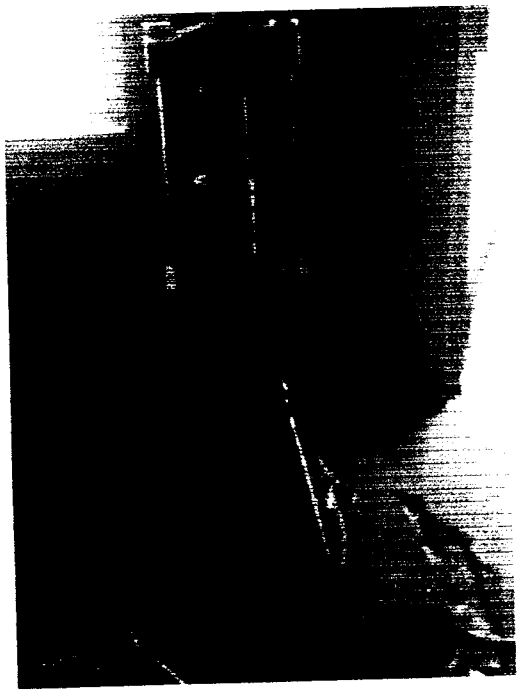

Plate 3.4: **Hydraulic Screw Press**

3.1.3 Reagents Used in the Analysis of Physiochemical Properties of Cottonseed Oil

The following reagents were used in the physiochemical analysis of the extracted cottonseed oil.

Distilled water.

Potassium hydroxide (KOH) solution.

Tetraoxosulphate (VI) acid.

Petroleum ether

Acetic acid.

Chloroform solution

Saturated potassium Iodide solution.

Sodium Hydroxide (NaOH) solution.

Alcoholic potassium hydroxide solution

Phenolphthalein indicator.

Hydrochloric acid (HCL)

Carbon tetrachloride

Aqueous potassium Iodide solution.

Sodium thiosulphate.

Starch indicator

3.2 Extraction Method

The samples collected were properly cleaned in order to remove any foreign materials. They were oven dried in the laboratory to a moisture content of 12%. This was done because the lesser the moisture content, the more the oil yield (Fernandez, 2010). The seeds were then crushed into powder using Thomas Willey milling machine. 12g of the crushed sample was weighed and mixed with 5ml of N – hexane. The mixed sample was placed on a filter paper and the filter paper was then properly folded and inserted into the assembled Soxhlet apparatus. The weight of the filter paper and sample was recorded. 150ml of the solvent (N- Hexane) was measured using a measuring cylinder and then poured into a 500ml round bottom flask which is the lower part of the soxhlet apparatus. This was now heated with a heating mantle at 60°C for 6 hours. As the solvent boiled, it evaporated into the reflux condenser and this hot solvent vapour was cooled by the surrounding water which flowed continuously through the soxhlet arrangement. The cooled solvent then condensed back into the portion of the Soxhlet containing the folded sample and this facilitated the extraction of the oil from the sample. As such, the oil that dropped into the round bottom flask was a combination of oil and solvent. The sample left after the oil had been removed was subjected to hot pressing using hydraulic press to remove the bulk of the oil remaining in the press cake. This sample was then weighed and the percentage oil yield was calculated as:

$$\frac{\text{Weight of sample before extraction} - \text{weight of sample after extraction}}{\text{Weight of sample before extraction}} \times 100$$

The oil was recovered by solvent evaporation. It was heated at a temperature higher than that of the solvent until the solvent finally evaporated leaving behind the oil extracted. The boiling point of N – Hexane is 69^0C (Wikipedia, 2011). While the boiling point of Cottonseed oil is 1017^0C (National Cottonseed Product Association, 2011).

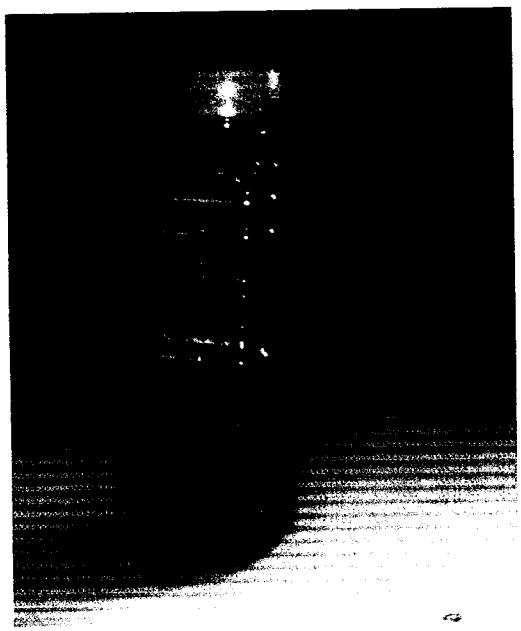

Plate 3.5: **Extracted Cottonseed Oil.**

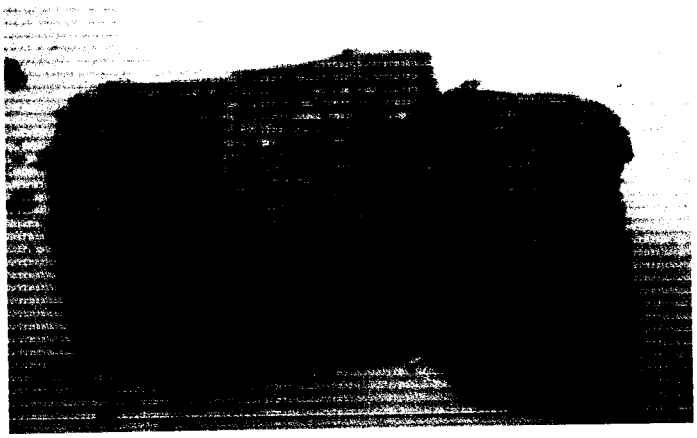

Plate 3.6: **Cottonseed Cake**

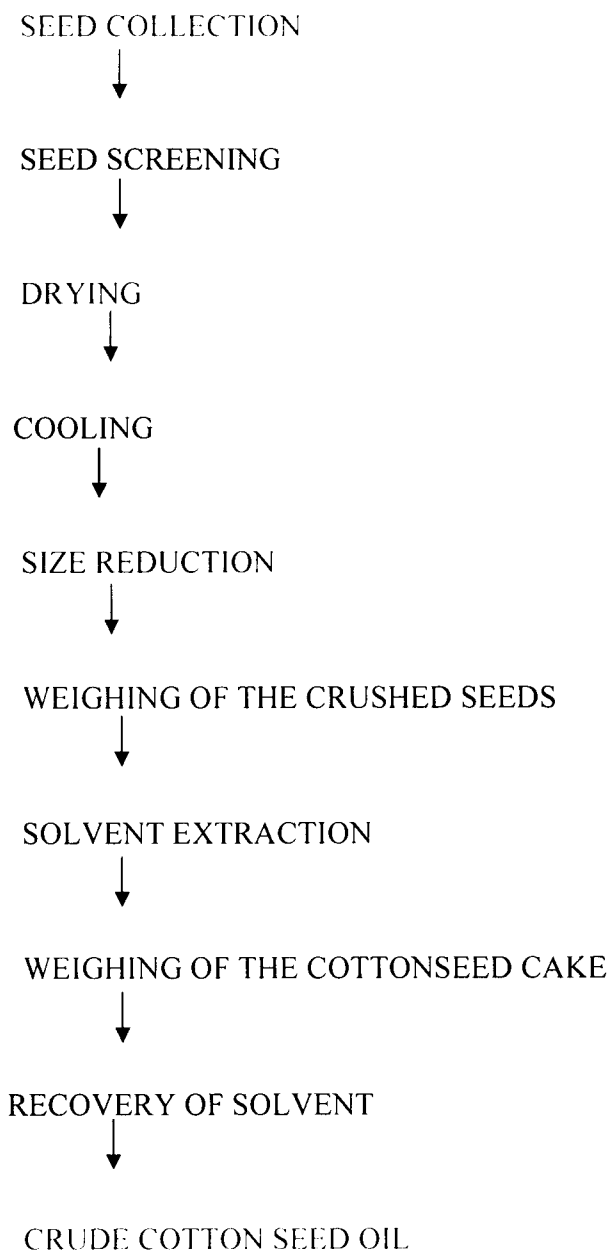

Fig 3.1 **Flowchart of the experimental process**

3.3 Determination of the Physiochemical Properties

The physiochemical analysis was carried out at the National Cereals Research Institute (NCRI), Badeggi, Bida, Niger State, Nigeria. The physiochemical properties; Saponification value, Peroxide value, acid value, free fatty acid, iodine value, refractive index, specific gravity, viscosity, colour, taste, and odor were determined using the methods of AOAC (2004).

3.3.1 Determination of Acid Value

The acid value of oil is the number of milligram of potassium hydroxide required to neutralize 1gram of the sample (Ewing, 1971). The acid value measures the extent to which the glycerides in the oil have been decomposed by lipase action. 25ml of diethyl ether and 25ml of alcohol was mixed with 1ml of phenolphthalein and this was carefully neutralized with 0.1M sodium hydroxide. 2g of the oil was dissolved in the mixed neutral solvent and this was titrated with aqueous 0.1m sodium hydroxide shaking constantly until a pink colour which persisted for 15seconds was obtained.

$$\text{Acid value} = \frac{\text{titre (ml)} \times 5.61}{\text{Weight of sample used}}$$

Acid value= FFA X 2

3.3.2 Determination of Saponification Value

The saponification value is the number of milligram of potassium hydroxide required to neutralize the fatty acids resulting from complete hydrolysis of 1g of the oil. 2g of the oil was weighed into a conical flask and 25ml of alcoholic potassium hydroxide solution was now added. A reflux condenser was attached to the flask containing the mixture and this was heated in

boiling water for one hour, shaking frequently. 1ml of phenolphthalein indicator was added to the hot solution and this was titrated with 0.5M hydrochloric acid to the end point until the pink colour of the indicator disappears. (Titer value = a ml). A blank was also carried out at the same time (titer value = b ml).

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

3.3.3 Determination of Iodine Value

Iodine value measures the degree of unsaturation in vegetable oil. This value for oil is defined as the weight of iodine absorbed by 100 parts by weight of the sample. 1g of the oil was weighed into a conical flask and 20ml of carbon tetrachloride was added to dissolve the oil. 25ml of Wijis' solution was added to the flask using a safety pipette in fume chamber. A stopper was inserted and the content of the flask was vigorously swirled. The flask was placed in the dark for 30 minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 100ml water was added using a measuring cylinder and titrated with 0.1M of sodium thiosulphate solution using starch as indicator until the yellow colour almost disappeared. The same procedure was carried out for blank at the same time commencing with 10ml of carbon tetrachloride.

$$\text{Iodine Value} = \frac{(a - b) \times 1.269}{\text{Weight of samples}}$$

3.3.4 Determination of Peroxide Value

The peroxide value (PV) of oil is the measure of its content of oxygen. This is used to monitor the development of rancidity through the evaluation of the quantity of peroxide in the product. The test is a volumetric one where I_2 formed from potassium iodide in the presence of peroxides, is titrated with thiosulphate. This means Meq. $_{peroxide}$ = Meq. $_{thiosulphate}$ at the equivalent point. 1g of the sample was weighted into a 250ml Erlenmeyer flask. 30ml acetic acid, chloroform solution (3:2) was added under a fume hood and swirled to dissolve the oil. 0.5ml saturated potassium iodine solution was added and swirled for 1 minute. 1ml of indicator was also added and titrated using starch. The same procedure was performed for blank at the same time.

$$\text{Peroxide Value} = \frac{S - B \times 0.1 \times 1000}{\text{Weight of sample}}$$

3.3.5 Determination of Specific Gravity

Specific gravity is the ratio of the density of a substance to the density of a saturated substance under specified conditions. For liquids and solids the standard is usually water at 4°C or some other specified temperature. For gases the standard is often air or hydrogen at the same temperature and pressure as the substance. A pycnometer bottle was used to determine the density of the oil. A clean and dry pycnometer bottle of 50ml capacity was weighed (w_0) and then filled with the oil, a stopper was inserted and reweighed to give (w_1). The oil was substituted with water after washing and drying the bottle and weighed to give (W_2). The expression for specific gravity (Sp.gr) is:

Specific Gravity = $(W_1 - W_2) / (W_0 - W_2)$

= Mass of the substance / Mass of equal volume of water.

3.3.6 Determination of Viscosity

The viscosity of oil is an indication of the degree of flow (fluidity) at different temperature. A clean, dried viscometer with a flow time above 200 seconds for the fluid to be tested was elected. The sample was filtered through a sintered glass (fine mesh screen) to eliminate dust and other solid material in the liquid sample. The viscosity meter was charged with the sample by inverting the tube's thinner arm into the liquid sample and suction force was drawn up to the upper timing mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into a holder and inserted to a constant temperature bath set at 29°C and allow approximately 10minutes for the sample to come to the bath temperature 29°C. the suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time by timing the flow of the sample as it flowed freely from the upper timing mark to the lower timing mark was recorded.

3.3.7 Determination of Refractive Index

Refractive index is the ratio of the speed of light at a definite wave length in a vacuum to its speed in the medium and this varies with the wave length of light and temperature. Refractometer was used in this determination. Few drops of the sample were transferred into the glass slide of the refractometer. Water at 30°C was circulated round the glass slide to keep its temperature uniform. Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with intersection of the cross. At no parallax error, the pointer on the scale

pointed to the refractive index. This was repeated and the mean value noted and recorded as the refractive index.

3.3.8 Determination of pH value

2g of the sample was poured into a clean dry 25ml beaker and 13ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was then cooled in a cold water bath to 25°C. The pH electrode was standardized with buffer solution and the electrode immersed into the sample and the pH value was read and recorded.

3.3.9 Determination of the Percentage of Cottonseed Oil Extracted

12g of the sample was placed in the thimble and about 150ml of normal hexane was poured into the round bottom flask. The apparatus was heated at 60°C and allowed for 3hrs continuous extraction using Soxhlet apparatus. The experiment was repeated for different weights of the sample. At the end, the solvent was distilled and the percentage of oil extracted was determined.

3.4 Determination of the Proximate Composition of Cottonseed Oil

The proximate composition of the oil was carried out at the laboratory of National Cereals Research Institute, Badeggi- Bida, Niger state, Nigeria. The proximate composition of the oil was determined as described below.

3.4.1 Determination of Percentage Crude Fat

In general lipids are characterized by their sparing solubility in water and their considerable solubility in organic solvents. The property that shows their hydrophobic, hydrocarbon nature. Determination of fat content of a food does not actually reflect the estimation of the true fat

content but of the lipid fraction of the food (Hamitton and Bhati, 1980). The weight of empty filter paper was taken and noted as W_1, 2.5g of the sample was weighed into a filter paper and 2.5g of water extract was also weighed and added to the sample on filter paper. The filter was then wrapped and put in the thimble; 250ml round bottom flask was filled with petroleum ether up to 75% of its volume. The soxhlet extractor was set up with a reflux condenser adjusted and allowed to boil gently for 6 hours. The petroleum ether containing fat was evaporated and fat was recovered. The weight of the fat was taken as W_3 (for both sample and water extract).

$$\text{Fat (\%)} = \frac{W_1 - W_3}{W_2 - W_1} \times 100$$

3.4.2 Determination of Percentage Moisture Content

Moisture content was determined by the direct air oven method of Association of Analytical Chemists (AOAC, 2004). A clean dry flat bottom silica dish was weighed and 2g of the sample pipetted into it. The sample was evaporated on a water bath and with the aid of forceps: it was then transferred into the air oven previously set at 105°C. After three hours, it was then removed from the oven, cooled in desiccators and reweighed again. The process was repeated until constant weight was obtained.

$$\text{Moisture content} = \frac{A - B \times 100}{A}$$

3.4.3 Determination of percentage Crude Protein

Amino acids are the building blocks of protein. Proteins are therefore polymers of amino acids, most of which are α-amino acids having the general formulae NH_2CHR_2COOH. It is the only macronutrient in food that contains nitrogen. Crude protein was determined by the kjeldahl method (AOAC, 2004). The process involved three stages; digestion, distillation and titration.

Digestion: About 2g of the sample formulated was measured into a 50ml kjeldahl flask, digestion tablets were then added to the flask followed by 20ml of concentrated sulphuric acid (H_2SO_4) which was poured slowly down the side of the flask while holding the flask in a slant position. The flask was then placed on a digestion block in a fume cupboard at about 300°C until a clear solution was obtained. The flask was then made up to 100ml with distilled water after cooling at room temperature.

Distillation: About 5ml of the diluted digest of each sample was transferred into a separate Kjeldahl flask and 20ml of 40% sodium Hydroxide (NaOH) was added and rinsed with few drops of distilled water, the flask was then placed at the heating end of the distillation unit. 5ml of 4% boric acid solution was placed in 100ml Erlenmeyer flask and 2 – 3 drops of mixed indicator was added (Bromo cresol green + methyl red indicator 5 : v/v ratio) before placing from the receiving end of the distilling unit. Ammonia was distilled into Boric acid solution until 75ml mark was reached. The boric acid distillate was then treated with 0.1N hydrochloric (HCL) to a pink end point, which persist for about 15minutes. Percentage protein was calculated by multiplying the percentage of nitrogen with a suitable factor (6.25)

%Nitrogen = A/B x 0.1 x 1/E

Where: A = Volume of acid used to neutralize the distillate

B = Volume of sample taken for distillation

C = Volume made after distillation (100ml)

D = Volume of sample

E = Acid factor

Crude Protein (%) = Nitrogen x factor (6.25)

3.4.4 Determination of Percentage Ash Content

Ash in food constitutes the residue remaining after all the moisture have been removed as well as the organic materials (fats, proteins, carbohydrates, vitamins, organic matter, etc.) have been burnt away by igniting at a temperature of around 500°C (Ihekoronye and Ngoddy, 1985). Ash was determined by the method of AOAC (2004). 2g of sample was weighed and placed in a water bath to be evaporated. It was then placed in a separate porcelain crucible (England) and its content placed in a Gallenkamp furnace and ashed to 105°C for about 10 hours. The sample was then removed from the furnace, cooled in desiccators and weighed. The percentage ash was calculated as:

$$\text{Ash (\%)} = \frac{\text{weight of crucible + ash} - \text{weight of crucible}}{\text{Initial sample}}$$

3.5.5 Determination of Percentage Crude Fibre

Fibre represents the organic residue left behind after the sample has been treated under standardized conditions with light petroleum, boiling dilute sulphuric acid, boiling dilute sodium hydroxide solution, dilute hydrochloric acid, alcohol and ether. 2g of the sample was defatted with petroleum ether. This was now boiled under reflux for 30minutes with 200ml of a solution containing 1.25g of H_2SO_4 per 100ml of solution. The solution was then filtered through several layers of cheese cloth on a flute funnel followed by washing with boiling water until the washing is no longer acid. The residue was transferred to a beaker and boiled for 30minutes with 200ml of a solution containing 1.25g of carbohydrate free NaOH per 100ml. The final residue was then filtered through a thin but closed pad washed and ignited asbestos in a grouch crucible. This is accompanied by drying in an electric oven and weighing. After weighing, it was then incinerated, cooked and re-weighed. The loss in weight after incineration x 100 is the percentage crude fiber.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Results

4.1.1 Physiochemical Characteristics of Cottonseed

The Physiochemical properties of the extracted cottonseed oil are presented in Table 4.1

Table 4.1 Physiochemical Properties of Cottonseed Oil

Parameters	Values
Free fatty acid	5.75mgKOH/g
Acid value	11.50mgKOH/g
Iodine value	94.7gI_2/100g
Saponification value	189mgKOH/g
Peroxide value	9.25mEq/kg
Refractive index	1.464
Specific gravity	0.92
Colour	Reddish brown colour
pH	4.82
Taste	Mild taste
Viscosity	74

4.1.2 Proximate Analysis of Cottonseed Oil

The Proximate Composition of the extracted Cottonseed Oil are presented in Table 4.2

Table 4.2 Proximate Composition of Cottonseed Oil

Nutrient	Composition (%)
Fat/oil	13.30
Moisture content	12.21
Crude protein	15.40
Ash	1.50
Crude fiber	0.50
Carbohydrate	57.06
% Oil Yield	15.00

4.2 Discussion of Results

Table 4.1 shows the physio-chemical properties of the extracted cottonseed oil. The free fatty acid was found to be 5.75mgKOH/g. It is reported that oil with low free fatty acid are edible and can stay for a very long time without getting rancid (Brien and Wakelyn, 2005). Kramer and Twigg, (1990) reports that oils with low value of free fatty acid contains tocopherol. Tocopherols are natural antioxidant which has varying degrees of vitamin E and this contributes to the stability of the oil giving products that contain it a long shelf life. Upon refining this oil, free fatty acid will be reduced thus increasing the iodine value (Brien,1998). The acid value was obtained by multiplying the free fatty acid by 2 giving a value of 11.50mgKOH/g.

It is reported that oils with low free fatty acid usually have high saponification value. Saponification value is a measure of the average molecular weight of the glycerides in the oil. The result obtained for the saponification value is (189mgKOH/g). This value is in accordance with the range of (190 – 198mgKOH/g) given by Jones *et al.*, (2005). As such cottonseed oil is good for soap production due to its high saponification value.

The iodine value which gives the degree of unsaturation in vegetable oils was found to be 94.7gI_2/100g for cottonseed oil. This value is close to pumpkin seed oil with an iodine value 100gI_2/100g (Markovic and Bastic, 1976). This means that the oil is highly unsaturated making it to have a stable physical state at varying room temperature. Brien, (1998) states that refined cottonseed oil has an iodine value in the range of (109 - 120) and refining increases iodine value. As such upon refining, the iodine value tends to increase. With this, the result obtained (94.7gI_2/100g) is correct because the oil is crude cottonseed oil. As such, upon refining, the iodine value will increase.

The percentage oil yield was found to be 15.0% which is close to the 17% as stated in the Encyclopedia Americana (2001). The pH of cottonseed oil was found to be 4.82, this may be due to the various chemicals used to control the cotton pests.

The peroxide value was found to be 9.25mEq/kg indicating that the oil is fresh oil. This is because fresh oils usually have peroxide values well below 10mEq/kg (Ewing, 1971). It is reported that when Cottonseed oil is refined, the peroxide value drops to about 5.00mEq/kg presenting a golden yellow, bright and clear oil.

The refractive index was obtained to be 1.464 at 20°C which is close to neem oil of 1.466 at 30°C (Soetaredjo *et al.*, 2007). Jones *et al.*, (2005) recorded that the refractive index for refined cottonseed oil is in the range of (1.468 – 1.472).

The specific gravity was obtained to be 0.92 which is close to pumpkin seed oil of 0.918 (Pearson, 1981). Jones *et al.*, (2005) reports that the specific gravity of refined cottonseed oil is in the range of (0.915 – 0.921). The value of viscosity at 20°C was determined to be 74.

The extracted cottonseed oil has a reddish brown colour with a mild taste and a bland odour. This reddish brown colouration is because the oil is unrefined and also due to the presence of gossypol. After proper refining through bleaching, decolorization, and deodourisation the colour changes to a light golden colour.

CHAPTER FIVE

5.0 Conclusion and Recommendations

5.1 Conclusion

The result of this study shows that most of the characteristics of cottonseed oil indicate that it can be used as cooking oil with long shelf life, it can also be used in the soap making industry due to its high saponification value and it can as well be used as a lubricant. The physiochemical properties determined are as follows; Free fatty acid value (5.75mgKOH/g), acid value (11.50mgKOH/g), iodine value (94.7gI_2/100g), saponification value (189mgKOH/g), peroxide value (9.25mEq/Kg), refractive index (1.464), specific gravity (0.92), pH (4.82) and viscosity (74). The values of the proximate composition were also determined as; Fat/oil (13.30%), moisture content (12.21%), crude protein (15.40%), Ash (1.50%), Crude fibre (0.50%) and carbohydrate (57.00%). The results obtained compared favourably with other conventional seed oils. These positive results indicates that it is an edible and non – drying oil which has good potential for use as domestic and industrial oil. Also, the result indicates that it can be used in the production of bio – diesel.

5.2 Recommendations

The following recommendations could be made from the study.

1. The planting of cotton plants should be encouraged throughout Nigeria. This is because during the project work, cottonseed was not readily available. As such this necessitated the travelling to very far distances in search of this seed.

2. The shutting down of many textile industries in Nigeria is due to the lack of 'cotton'. As such this is a wakeup call for the government to sensitize the public on the importance of growing this crop thereby creating employment for the unemployed and boosting the oil sector.

3. Other chemical and physical properties should be investigated.

4. Other extraction methods should be investigated and the results obtained from various extraction methods should be compared.

5. Pesticides and other agrochemicals should be readily available for the proper growth of this crop. This is because this crop is associated with varieties of pests and diseases.

6. More effort is needed in the production of vegetable oil in Nigeria using new and improved machineries. This is because more than 50% of the vegetable oils in Nigeria are imported mainly from Malaysia.

7. More research should be carried out to investigate/seek newer sources of cheaper and readily available oil seeds.

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APPENDICES

Determination of Saponification Value

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

Where a = Volume (ml) of 0.5 mol/l hydrochloric acid consumed in the blank test

b = Volume (ml) of 0.5 mol/l hydrochloric acid consumed in the test

The experiment goes thus:

Titre Value (a) = 14.30

Titre Value (b) Blank = 27.78

Weight in g of sample used = 2g

$$\text{Saponification Value} = \frac{(27.78 - 14.30) \times 28.05}{2}$$

$$= \frac{13.48 \times 28.05}{2}$$

$$= \frac{378.114}{2}$$

$$= 189$$

Determination of Acid Value and FFA

$$\text{Acid Value} = \frac{\text{Titre Value (ml)} \times 5.61}{\text{Weight of sample used}}$$

Titre Value (ml) = 4.1

Weight of Sample used = 2g

$$\text{Acid Value} = \frac{4.1 \times 5.61}{2}$$

$$= \frac{23.001}{2}$$

$$= 11.50$$

Acid Value = FFA x 2

$$\text{FFA} = \frac{\text{Acid Value}}{2}$$

$$= \frac{11.50}{2}$$

$$= 5.75$$

Determination of Peroxide Value

$$\text{Peroxide Value} = \frac{S - B \times 0.1 \times 1000}{\text{4g of Oil}}$$

Where S = sample titre value

B = Blank titre value

0.1N = Molarity of sodium thiosulphate

S = 6.65

B = 6.28

$$\text{Peroxide Value} = \frac{(6.65 - 6.28) \times 0.1 \times 1000}{4}$$

$$= \frac{0.37 \times 0.1 \times 1000}{4}$$

$$= \frac{0.0925 \times 0.1 \times 1000}{4}$$

$$= 9.25$$

Determination of Iodine Value

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

Where a = sample titre value

b = Blank titre value

Weight of sample used = 1g

a = 7.16

b = 81.82

Weight in g of sample = 1g

$$\text{Iodine Value} = \frac{(81.82 - 7.16) \times 1.269}{1}$$

$$= 74.66 \times 1.269$$

$$= 94.7$$

Determination of Specific Gravity

$$\text{Specific Gravity} = \frac{\text{Weight of Oil}}{\text{Weight of Water}}$$

Weight of empty pycnometer bottle = 25.10

Weight of Oil + empty pycnometer bottle = 71.10g

Weight of water + empty pycnometer bottle = 75.10g

$$\text{Specific Gravity} = \frac{71.10 - 25.10}{75.10 - 25.10}$$

$$= \frac{46}{50}$$

$$= 0.92$$

Determination of Percentage Yield of Cottonseed Oil

$$\% \text{ Yield of Oil} = \frac{\text{Weight of sample before extraction - Weight of sample after extraction}}{\text{Weight of sample before extraction}}$$

Weight of the timble + sample before extraction = 12.00g

Weight of the timble + after before extraction = 10.20g

$$\% \text{ Yield of Oil} = \frac{12.00 - 10.20}{12.00} \times 100$$

$$= \frac{1.80}{12.00} \times 100$$

$$= 15\%$$

Determination of Moisture Content of the Cottonseeds before Extraction

$$\% \text{ Moisture content} = \frac{\text{Weight of moisture in the cottonseeds}}{\text{Weight of cottonseeds before oven drying}} \times 100$$

Weight of empty petri dish = 41.81g

Weight of cottonseeds + petri dish before oven drying = 61.61g

Weight of cottonseeds before oven drying = 61.61 – 41.81

= 19.8g

Weight of cottonseeds + petri dish after oven drying = 59.21g

Weight of cottonseeds after oven drying = 59.21 – 41.81

= 17.4g

$$\% \text{ Moisture Content} = \frac{19.8 - 17.4}{19.8} \times 100$$

= 12%