

indicator was added and the mixture titrated against 0.1M Potassium hydroxide solutions. End point was marked by the appearance of pink colour.

A.V is calculated from the formula

$$A.V = \frac{56.1 * M * V}{Mg}$$

### **3.5 Hair Cream Production Using Cottonseed Oil.**

Hair cream is an oily cosmetic for cleaning and protecting the hair. It is made up of fat, acid, petroleum products etc.

#### **3.5.1 Formation**

- i. Cottonseed oil (this is the binding agent.)
- ii. Petroleum jelly (the base of the product)
- iii. Paraffin wax (the builder agent)
- iv. Stearic acid (improves the cream nature of the cream and then the hair).
- v. Lanolin (fatty substance that keep the cream and hair mild).
- vi. Glycerine (shining agent)
- vii. Perfume (provides the fragrance)
- viii. Colour (for visual attraction)

#### **3.5.2 Procedure:**

The heating system was switched on at temperature between 50°C-60°C and the boiler (250ml cylinder) was placed on it. 25ml of cottonseed oil was measured into the boiler; colour (to taste) was added to the oil which was allowed to stir very well for a while. 1.5g of Stearic acid was added followed by 18g of jelly and 2.0g lanolin. 2teaspones of glycerine was then added. Followed by 3.0g of wax. To test the mixture, some drops of the mixture were dropped on the surface of water (warm) in a small container and tested with finger tip to see if it is alright. When tested okay, the heating system was put off and the mixture was

allowed to cool for a while and finally, the perfume (to taste) was added last to avoid it from evaporating. The product was then poured into a container.

### **3.6 Characterization of the Hair Cream.**

**3.6.1 pH determination using pH meter:** the pH meter was switched on for about 30 min to warm up. The pH of the soil was determined by dipping the reference electrode into the hair cream and a corresponding deflection read off on the meter. The result obtained as shown in chapter 4.

#### **3.6.2 Specific gravity**

Specific gravity is the ratio of weight of a substance to the weight of the same volume of water both at specified temperature. A 50ml measuring cylinder was weighed ( $W_0$ ). The measuring cylinder was filled with 20ml of water and re-weighed ( $W_1$ ). The measuring cylinder plus hair cream was weighed ( $W_2$ ), specific gravity (Sg) was computed using the formula:

$$S_g = \frac{W_2 - W_0}{W_1 - W_0}$$

#### **3.6.3 Hair shine**

Hair shine is evaluated by looking at the reflection of light on the hair under Standard conditions (natural daylight or a daylight lamp). From a distance of 0.5m and with slight head movement by the model, the light reflection or shine is evaluated as more or less.

#### **3.6.1 Creaminess**

The hair cream is lightly rubbed between the fingers and assessment is made if

it feel creamy or watery.

### **3.7 Lists of Equipment, Materials and Reagent Used.**

Soxhlet extractor apparatus.

Mortar and pestle

Heating mantle.

Stopwatch

Thermometer

Beakers

Weighing balance

Oven

PH meter

Muffle furnace

Measuring cylinder

Drying cabinet

Round bottom flask

Flat bottom flask

Electromagnetic stirrer

**Chemical and Materials Used:**

Cottonseed Oil

N-hexane

Diethylether

Phenolphthalein indicator

Potassium hydroxide

Petroleum jelly

Paraffin wax

Lanolin

Stearic Acid

Colour

Perfume.

## CHAPTER FOUR

### 4.0 RESULTS AND DISCUSSION OF RESULT

#### 4.1 CALCULATION

From the result shown in table 4.1, the percentage extraction was calculated using the relationship below:

$$\text{Percentage extraction (\%)} = \frac{\text{Weight of oil}}{\text{Initial Weight of sample}} \times \frac{100}{1}$$

For instance, in the first run extraction, the amount of oil extracted was 4.13 using 13g of sample

$$\begin{aligned} \% \text{ Extraction} &= \frac{4.13}{13} \times \frac{100}{1} \\ &= 31.77\% \end{aligned}$$

The same procedure was used for the other runs, the result is presented in table 4.1

#### 4.2 EXPERIMENTAL RESULT

The table 4.1 below shows the result of solvent extraction of cottonseed oil from cottonseeds for 8 runs or stages

**TABLE 4.1 RESULT OF THE EXPERIMENT**

	Weights of Thimble (g)	Weight of sample (g)	Weight of Thimble + sample before extraction (g)	Weight of Thimble + sample after extraction (g)	Weight of oil	% Extraction (g)
1 <sup>st</sup> Run	1.96	13.00	14.96	10.83	4.13	31.77
2 <sup>nd</sup> Run	2.03	13.00	15.03	10.96	4.07	31.31
3 <sup>rd</sup> Run	3.10	15.00	18.01	12.79	5.22	34.80
4 <sup>th</sup> Run	3.16	15.00	18.16	12.81	5.35	35.67
5 <sup>th</sup> Run	2.01	13.00	15.01	10.87	4.14	31.85
6 <sup>th</sup> Run	2.16	13.00	15.16	10.04	5.12	39.38
7 <sup>th</sup> Run	2.29	13.00	15.29	11.36	3.93	30.23
8 <sup>th</sup> Run	2.09	15.40	17.49	12.44	5.05	32.79

Table 4.2 shows the result of chemical and physical test

TEST	Oil	Hair cream
PH	5.42	8.5
Specific Gravity	0.918	0.96
Reflective Index	1.35	
Color	Dark-yellow	Green
Odor or Smell	Odorless	Not offensive
Acid Value	3.12	
Hair Shine		More
Creaminess		Creamy

### 4.3 DISCUSSION OF RESULTS

From the result of the extraction for the eight runs or stages presented in table 4.1, it was observed that the amount of oil extracted increases as the size decreases. This is because as the size decreases, solute available for solvent attack is increased and hence more oil extracted. Reducing the size also connotes increasing the inter-facial area between the solute and solvent and thereby increasing the rate of transfer of the oil from the solute. This is also in accordance with chemical engineering processes whereby increasing the surface area of the reactants increases the rate of reaction.

Reducing the size enhances proper mixing of the solute and solvent and this implies that the solvent will move shorter distance within the solid thereby increasing the reaction rate based on the extraction principle.

One important fact to note is that doubling the time does not necessarily double the oil quantity since after some time of extraction, the oil inside the cake can no longer be extracted. At this point, whether the time is increased or not the oil extracted is constant. This is because solvent extraction has a maximum percentage to extract and once this percentage has been attained, further increase in time is useless.

## 5.0 CONCLUSION AND RECOMMENDATIONS

In this chapter, the conclusion drawn and the recommendations made from this research work shall be highlighted.

### 5.1 CONCLUSION

From the result obtained, it was found that the specific gravity of the oil extracted was 0.918, refractive index 1.357, acid value 3.12, pH of hair cream 8.5, specific gravity 0.96, and each of the experimental values conforms to the general specifications. The various chemical and physical tests of the crude cottonseeds oil had shown that it can be used for the production of hair cream.

### 5.2 RECOMMENDATIONS

The following could be recommended as regards to this work.

- a. Investigations should be carried out to determine the optimum particle size for operating a packed bed extractor.
- b. If an extractor is available, the optimum solvent flow rate should be deduced so that flooding will not occur, which will render the packed bed extractor useless.
- c. Determination of the precise composition of cottonseed oil should be made using infrared spectrometer.
- d. Equilibrium solubility data between cottonseed oil and hexane or other organic solvent should be determined.
- e. Melting and freezing point of cottonseed should be determined.



f. Mechanical method of extraction should be employed and comparison of percentage extraction with that of solvent method should be made in order to know the method suitable for optimum yield of extracted oil.

## APPENDICES

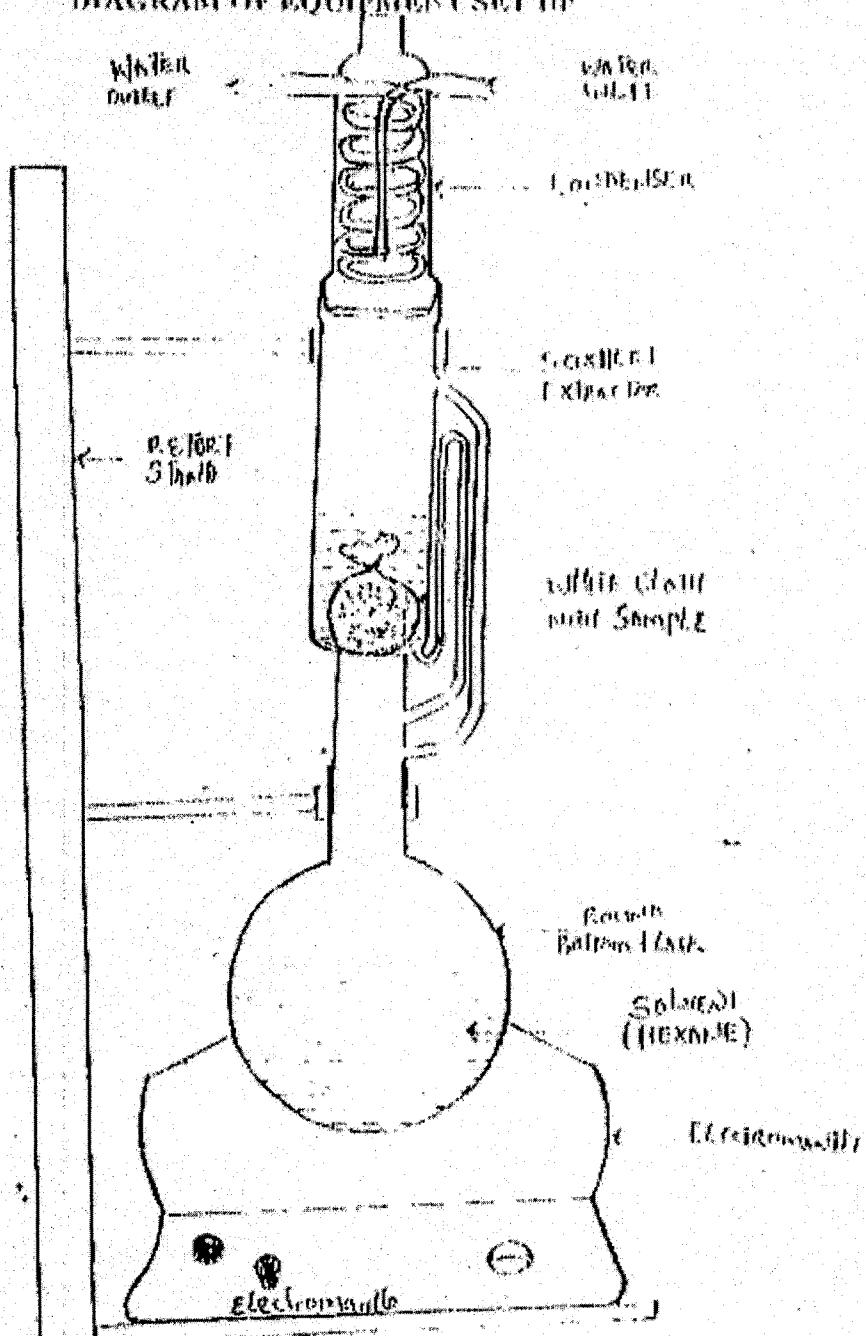
### A1.1 Moisture content calculation

$$\left( \frac{\text{weight before drying} - \text{weight after drying}}{\text{weight after drying}} \right) * 100$$
$$= \left( \frac{200 - 225}{225} \right) * 100$$
$$= 2.22\%$$

### A1.2 Calculation of oil yield.

% Yield	=	Weight of oil	X	$\frac{100}{1}$
		$\frac{\text{Initial weight of sample}}$		
1 <sup>st</sup> run % yield	=	$\frac{4.13}{13.00} X \frac{100}{1}$	=	31.77%
2 <sup>nd</sup> run % yield	=	$\frac{4.07}{13.00} X \frac{100}{1}$	=	31.31%
3 <sup>rd</sup> run % yield	=	$\frac{5.22}{15.00} X \frac{100}{1}$	=	34.8%
4 <sup>th</sup> run % yield	=	$\frac{5.35}{15.00} X \frac{100}{1}$	=	35.67%
5 <sup>th</sup> run % yield	=	$\frac{4.14}{13.00} X \frac{100}{1}$	=	31.85%
6 <sup>th</sup> run % yield	=	$\frac{5.12}{13.00} X \frac{100}{1}$	=	39.38%
7 <sup>th</sup> run % yield	=	$\frac{3.93}{13.00} X \frac{100}{1}$	=	30.23%
8 <sup>th</sup> run % yield	=	$\frac{5.05}{13.00} X \frac{100}{1}$	=	32.79%

DIAGRAM OF EQUIPMENT SETUP



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## DECLARATION

I, ABUTU FAITH OJONE, declare that this research work presented for the award of B.Eng. in Chemical Engineering, (extraction of cottonseed oil for the production of hair cream) to the best of my knowledge has not been presented anywhere either partially or fully for this same purpose.

~~ABUTU~~

10/11/05

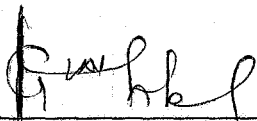
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**ABUTU FAITH OJONE.**

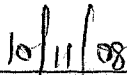
**DATE**

## CERTIFICATION

This is to certify that this project "Extraction of oil from cottonseed for the production of hair cream" is the original work of Abutu Faith Ojone. carried out wholly by her under my thorough supervision and submitted to the department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Minna.



Engr. Abubakar Garba Isah  
Supervisor



Date

Dr. M.O Edoga  
(H.O.D. Chemical Engineering)

Date

External Examiner

Date

## DEDICATION

I dedicate this project work to God my heavenly father for his divine inspiration, guidance and protection during my years of studies. I also want to dedicate it to the entire members of my family, the family of Mr. and Mrs. Christopher M. Abutu for their tireless contribution by encouraging and sponsoring me all through these years.



## ACKNOWLEDGEMENT

My work on this project will not be complete if I fail to acknowledge the contributions of people who in one way or the other assisted me in the course of my studies.

My profound appreciation goes to my Head of department, Engr. Dr. M. Edoga, my project supervisor Engr. A. G. Isah for going through the work and making all the necessary corrections and contributions.

I cannot forget the members of my immediate family for their support both financially and otherwise. Special thanks go to the following families for their care and support in the course of my studies. The families of Mr. & Mrs. Abel Ocheme, Mr. & Mrs. M. K. Ibrahim, the families of Late Mr. Vitalis Ehieme, Late Mr. Benjamin Ohiemi, Late Mr. & Mrs. Joseph Y. Agbo, Mr. & Mrs. Thomas Abutu and Mr. & Mrs. Adegbe.

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## CHAPTER ONE

### 1.0 Introduction

Cottonseed oil is a vegetable oil extracted from the seeds of the cotton plant after the cotton lint (cotton fibres) has been removed. It must be refined to remove gossypol. A naturally occurring toxin that protects the cotton plant from insect damage. Unrefined cottonseed oil is therefore sometimes used as a pesticide. In its natural un-hydrogenated state, cottonseed oil, like vegetable oils has no cholesterol and also contains no trans fatty acid. However, it does contain over 50% omega-6 fatty acid and only a trace amount of omega-3 fatty acid and the imbalance is considered unhealthy if not used in moderation or balanced elsewhere in the diet. Clack, (2007).

Cottonseed oil is rich in palmitic acid (22-26%), oleic acid (15-20%), linoleic acid (49-50%) and 10% mixture of arachide acid, behenic acid and lignoceric acid. It also contains about 1% sterculic acid and malvalic acids in the crude oil. The cyclopropene acids are undesirable components but they are largely removed during refining, particularly deodorization, and also during hydrogenation. They are not considered to present any health hazard in cottonseed oil. (Canola, 2007)

Cotton plant is a perennial plant that grows in many places on the earth but it has been known about, cultivated and put to use by people of many lands for centuries. Harvesting the cotton by hand is another limitation of productivity. Scientists and historians have found shreds of cloth or written reference to cotton dating back at least seven thousand years. The oldest discovery was made in a Mexican cave where scientist unearthed bits and pieces of cotton bolls and cloth. Excavation in Mahenjo-Doro, Sind, Pakistan (Indus valley) revealed the

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**A PROJECT REPORT ON THE EXTRACTION OF OIL FROM COTTONSEED FOR**

**THE PRODUCTION OF HAIR CREAM**

**BY**

**ABUTU FAITH OJONE**

**2003/14912EH**

**A PROJECT SUBMITTED TO**

**THE DEPARTMENT OF CHEMICAL ENGINEERING ,**

**SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY,**

**FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA.**

**IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF THE  
DEGREE, BACHELOR OF ENGINEERING (B.ENG.) IN CHEMICAL ENGINEERING**

**NOVEMBER, 2008**

occurrence of cotton in the form of strings and fragments of cloths covering the household articles. (Gulati and Turner, 1982)

Cotton has been grown for its fibres for several thousand years. Its cultivation and manufacture into cloth developed independently in both the eastern and western hemispheres. An old record of cotton textiles dating back about 5,000 years was found in the Indus River Valley (Pakistan). Cotton fabrics have also been found in the remains of some of the ancient civilization of Egypt. The desire for cotton and cotton fabrics was one of the factors that led to European exploration during the 15th and 16th centuries (Slater, 1780).

Although cotton has been grown for its fibres 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 centuries, however, the use of cottonseed did not develop much beyond that crude stage and was confined to local areas. The first cottonseed oil known to have been produced in America was exhibited before the American philosophical society in 1768. It was produced experimentally on a very small scale and was generally regarded as a curiosity. Little effort appears to have been made to produce additional oil until after the invention of the cotton gin (a machine used to separate seeds from fibres) in 1793. The increase in cotton production that followed the invention made the increasing quantities of cottonseed a challenge. (Whitney, 1793) and (Nathaniel, 2002).

Cottonseed oil has superior nutritive qualities, it has no cholesterol, and it is high in poly-unsaturates, moderate in mono-unsaturates and low in saturated fats. Cottonseed oil has 3:1 ratio of unsaturated fatty acids. This meets the

recommendations of many health professionals. Cottonseed oil is used in the production of margarine, shortenings (production of baking and drying fats) and as a cooking fat. Winterized cottonseed oil is used as a salad oil.

Non-food uses of cotton seed oil include; soaps, emulsifiers, pharmaceuticals; insecticides, fungicides, pesticides, cosmetics, rubber, plastics and finishes for leather, paper and textile. With advances in technology such uses may expand. Since cottonseed oil is an edible product, it is ideal for use in dust control applications in the food, feed and bedding industries. Other uses of cotton includes; animal feed (cotton seed hulls) and the short fibres (cotton seed linters) removed from the seed undergo mechanical and chemical conversion and enter into a wide range of uses. Goura, (2001).

In the solvent extraction mills, the cooked meat are flaked to about the thickness of paper and exposed to live steam and high pressure to make the oil cells accessible for the solvent extraction process. The prepared meats are conveyed to the extractor and washed with hexane (organic solvent that dissolves out the oil) removing up to 98% of the oil. The oil-hexane mixture (miscella) is pumped out of the extractor and distilled by boiling the hexane from the mixture and condensing it with cooling water. The crude cottonseed oil is then processed further. The deoiled meats are subjected to live steam for removal of residual hexane. The meats are then dried and ground to produce 41% protein livestock feed. (Joseph, 2007)

## **1.2 Aim and Objectives**

The aim of the project is to extract oil from cottonseed and to test its suitability in the production of hair cream

The major objective is to provide an alternate source of oil to other existing oil and to make it more accessible for domestic and commercial uses.

Also, to provide livestock feed of a high nutritive value.

### **1.3 Justification**

With the increasing demand for vegetable oil, the need for cottonseed oil for household and industrial uses are of greater importance. Cottonseed oil that can serve as a better substitute for other vegetable oil can also be used to produce a more effective product such as hair cream that increases hair growth and prevents the hair from attacks such as dandruff etc.

### **1.4 Scope of the Study and Limitation**

The scope of this work covers the research on extraction of oil from cottonseed and the effectiveness of the oil in production of hair cream. The expiring date could not be determined within two months of production.



## CHAPTER TWO

### 2.0 Literature Review

Wren (1995) documented that, prior to the civil war, surplus cottonseed was only utilized as feed for cattle or as fertilizer on depleted cotton and cornfield. Rotting cottonseed became such a problem in the south that several states passed law to regulate its disposal, during the 20<sup>th</sup> century, this supposedly worthless cottonseed became the second most previous cash crop in the south and this was due to the manufacture of cottonseed into marketable commodities.

### 2.1 Processing of Cottonseed

Jones and Bling (1993) in their work stated that cottonseed is cleaned by a variety of screening methods, linter fibres are removed mechanically using cotton seed delinter machines. Lint must be removed in order to improve the yield of oil. Hulls are removed from the seed to improve the protein quality of the meal produced and prevent oil sorption by the mulls. Reduction involves flaking or reducing the size of the meat once the hulls are removed in order to simplify oil removal.

Ward (1976) investigated that the seeds are cooked in steam-jacketed kettles in two separate phases, the first phase utilize a temperature of 88<sup>o</sup>C while the second phase utilizes a temperature range of 11<sup>o</sup>C-132<sup>o</sup>C. The final moisture content of the seeds is between 3-6% depending on which extraction method is use. Cooking breaks down cell walls which allows the oil to escape, reduces oil viscosity, control moisture content, inactivates enzymes, kills microorganism, detoxifies gossypol and binds phosphatides. Three methods are used in the industry to extract oil; screw press, solvent method or a combination of the two. The solvent method, which uses hexane, is the most common.

## 2.2 Manufacturing of Cottonseed Oil

Wren, 1995, stated in his work that, depending on the intended use of the oil, cottonseed oil can be winterized or hydrogenated. Cottonseed oil must be winterized before being marketed as a salad oil to remove the cloudiness caused by stearine. Stearine solidifies at refrigerator temperatures due to its saturated fat content. To produce a clear cottonseed oil, the oil is cooled until the stearine precipitates.

Salunkhe et al, 1992, describe that, the purpose of hydrogenation is to reduce the potential for oxidative damage, increase the stability of the oil and to solidify the oil. The process increases the melting point of oils and helps retard oxidation and flavour deterioration. Hydrogenation not only adds hydrogen to the double bond, it also results in migration of double bonds and reconfiguration of some of the cis double bonds into trans double bonds. Trans fatty acids are so named because the hydrogen atoms on the carbon atoms involved in the double bond are on opposite sides of the carbon chain.

Puri (1981) in his work wrote that oils that require hydrogenation to increase stability are usually high in polyunsaturated and monounsaturated fatty acids. Polyunsaturated fatty acids such as linoleic and oleic acids that have a tendency to oxidize and become rancid are reduced during hydrogenation, which prolongs storage time. Oils that are higher in saturated fats are more stable; oil must meet a certain quality standard in order for hydrogenation to be effective. The requirements are that the oil contain <0.1% free fatty acids, <1.5ppm soap, in addition, it should have a low colour and a peroxide value < 10 meq/ kg because polar pigments and oxidized ions can act as catalyst poison.

### **2.3 Refining of Cottonseed Oil**

Jones and King (1993) reviewed in their work that, raw cottonseed oil must be refined to remove impurities before it can be utilized as a human food source, which ensures desirable flavour, colour and stability characteristics. Refining is the most important step in the purification process for oil and involves removing the non-glycerid components such as phospholipids, colour, and trace metals and free fatty acids. The process mixes alkali and oil to form soaps. The non-glycerid components contain in the soap is then removed by using centrifugation and hot water washings.

They stated further that, bleaching furthers the purification process that involves the removal of colour bodies, trace metals, and soaps by using bleaching clays that absorb the impurities.

Gavin (1978) stated that deodorization is an essential step in the refining process. The process removes volatile compounds that can produce off-odours and off-flavours, leaving the oil with 0.01-0.03% free fatty acid content and a zero peroxide value. David Wesson perfected this process in the twentieth century who exposed the oil to superheated steam in a vacuum. Along with free fatty acids, aldehydes, ketones, alcohols and hydrocarbons associated with undesirable flavours and odours produced by autoxidation are removed. Cottonseed oil can be deodorized at lower temperatures, resulting in less loss of tocopherols that are natural antioxidants that help retard oxidation.

### **2.4 Compositions of the Oil and Uses**

Weiss (1983) in his work stated that, cottonseed oil has been the preferred oil for frying potato chips in the southern U.S.A in 1995/1996, 497 million pound of cotton seed oil in the form of shortening, margarine, salad and cooking oils were used in food. Cottonseed oil contains higher proportion of saturated fatty

acids (24.6%) as compared to soybean oil (14.7%) and canola oil (6.1%). However, the saturated fatty acid content reported for canola oil and soybean oil was obtained from the unhydrogenated oil.

Cottonseed oil contains (40-5%) linoleic acid, 20-25% palmitic acid, 2-7% stearic acid, 18-30% oleic acid and small amounts of myristic and arachidonic acids and approximately 0.5-2% cyclopropanoid acids. Cottonseed oil contains virtually no linolenic acids that considerably strengthen its stability for frying. (Salunkhe et al. 1992).

Eskin et al (1996) summarizes the fatty acid composition as in table 1a below.

Table 1a. Characteristics of cotton seed oil, canola oil, and soybean oil.

Parameters	Cottonseed oil	Canola oil	Unhydrogenated
Free fatty acid	Unhydrogenated	Unhydrogenated	Unhydrogenated
Oleic	Not more than 0.05%	Not more than 0.05%	Not more than 0.05%
Iodine value	99-119	110-126	120-143
Peroxide value	Not more than 1 meal kg	Not more than 1 meal kg	Not more than 1 meal kg
P-Anisidine value 2	<10	<10	<10
Linolenic acid	Not more than 2.1%	Not more than 1.4 %	Not more than 1.5%
Water	Not more than 0.1%	Not more than 0.1%	Not more than 0.1%

Salunkhe et al. (1992) describe the vast utilization of cottonseed, cottonseed hulls are used as roughage in livestock feeds and are used for a variety of other purposes such as fuel for oil mills, insulation material and soil conditioner. Cottonseed meal is used in the textile industry and is also used as an adhesive. Cottonseed proteins are used as nutrition's protein source for human consumption. The manufacture of salad and cooking oils, shortenings and margarine comprise almost the entire market for cottonseed oil. The remainder of the oil is used in the manufacture of non-edible items such as soap and it is also used to pack fish and cured meats.

Wrenn (1995) wrote that cottonseed oil was used to produce the first hydrogenated-all vegetable shortening product for the retail trade. Crystallized cottonseed oil was

introduced in the early 1900s by Proctor & Gamble who had a patent on the hydrogenation process. Once hydrogenation became accessible to other manufacturers, less expensive, lower grade oils were able to compete in the market.

Nationally, cottonseed oil competes with soybean oil, corn oil, peanut oil, sunflower oil,

safflower oil, and some animal. Cotton seed oil must compete internationally with coconut oil from Southeast Asia, palm oil from Malaysia, African peanut oil, olive oil from the Mediterranean basin, European sunflower oil and Canadian rapeseed oil.

The dominant position of cottonseed oil in the early 1900's started to decline secondary to the advent of improved processing methods (hydrogenation and deodorization) that greatly improved not only the quality of cottonseed oil but other oils as well. In addition, the increased use of cottonseed for cattle feed has

decreased cottonseed oil production (Jones and King 1993). Cottonseed oil remains fifth in world production due to the competition from these other oils.

Cottonseed oil is difficult to market because its supply relies on cotton production, which can be variable according to climate conditions. This is a disadvantage to customers who rely on a stable supply of oil to support their finished products.

During the period from the 1940's to the 1960's, the encouragement of soybean production to increase the supply of protein caused cottonseed oil prices to be placed at a disadvantage (Smith 1962). According to the USDA (1988) cottonseed oil is perceived as expensive oil due to its exceptional performance characteristics in food applications, however, it can be priced lower than soybean and other oils. Typically, oil prices around the world are closely related and remain competitive with each other.

Alhassan and Isah (2001) documented the production of biodiesel by transesterification for cottonseed oil with alcohol in the presence of a catalyst (NaOH), with the aim of providing an alternative to fossil fuel, which has adverse effect on the environment.

Hunter and Applewhite, 1991, investigated that mono and polyunsaturated fatty acid are considered "heart healthy", however; the process of hydrogenation rearranges some of the fatty acids from the cis form to the unhealthy trans form. Partially hydrogenated vegetable oils contain approximately 30% trans fatty acids, whereas tallow contains approximately 3%. It has been known for year that dietary fat and cholesterol influence blood cholesterol concentrations. Extensive research has been done not only on fats as a class, but on individual fatty acid and their effect on blood lipid level and lipoprotein concentration. The length of the

chain and the degree of un-saturation of a particular fatty acid molecule contribute to the ability of the fatty acid.

A study by Mensink and Katan (1990) found that consumption of saturated fatty acid and trans fatty acid are equal in their effect on blood cholesterols.

Carr, 1995, wrote that canola oil ranks third among the oilseed crops, behind soybean and palm oil in the production of edible oils. In the years 1995/1996, 319 million pounds was used in food in the form of shortening, margarine, salad and cooking oil. Canola oil is the least saturated oil on the market and has a naturally high content of linolenic acid content (11-12%).

Plant breeders have been able to reduce the linolenic acid content to 2.1%, increase the Oleic acid from 20% to 27%, and increase the oleic acid content from 60 to 85% with the intent of producing a very stable oil. (Eskin et al. 1996).

Soybean oil is the most widely produced oil in the U.S. and the world. In 1995/1996, 11,877 million pounds of oil was used in food in the form of shortening, margarine, salad and cooking oil.

Because of its high linolenic acid content (6.8%), soybean oil must be hydrogenated to produce a stable oil used under frying conditions. In addition, linolenic acid is responsible for the oil's flavor and odour reversion. Light hydrogenation reduces the linolenic acid content to approximately 3% and flavor stability is improved. On average, unhydrogenated soybean oil contains 22.8% oleic, 50.8% linoleic, and 6.8% linolenic .(Eskin et al. 1996).

## CHAPTER THREE

### 3.0 Experimental Work

This chapter contains: pre-treatment process, experimental procedure, flow chart, diagram of equipment set up, the list of equipments and material reagents for the experiment.

### 3.1 Pre Treatment Process

The pre-treatment process was divided into a number of stages that had to be carried out before the actual procedure of the experiment. The operation includes the following.

- i. Seeds collection
  - ii. Cleaning of seeds
  - iii. Drying of seeds
  - iv. De-hulling
  - v. Size reduction
  - vi. Size classification
- i. **Seeds collection:** - this involves the collection of the seeds from cotton plant and the drying. The drying process stimulates the opening of the capsule to release the seeds embedded inside. The seeds were then removed from shaft and other impurities.
  - ii. **Cleaning of seeds:** - particles associated with the cottonseed that are not really cottonseeds were handpicked during cleaning of the seeds.
  - iii. **Drying of seeds:** - having selected the seeds, the seeds were dried in an oven for one hour at 100<sup>0</sup>C. Drying is a physical process whose objectives is to reduce the moisture content of the seeds and enable size reduction to be carried out with ease.



- iv. **De-hulling:** - this simple process is the removal of the shaft from the cottonseed leaving the main kernel of meat in readiness for size reduction. The seed is de-hulled to avoid changing in the colour pigment of the cottonseed oil and also to retain all its functional properties after extraction
- v. **Size reduction:** - this was carried out using a mortar and pestle. The seeds were crushed in the mortar with a pestle. Crushing was carried out for a long time to make sure that various sizes can be obtained. This operation ruptures the cell wall and releases the solute for direct contact with the solvent during extraction. The operation also increases the total surface area for the seed.
- vi. **Size classification:** - the pounded sample (seeds) was used in single size using sieve analysis of size 150 $\mu$ m.

### 3.2 Determination of Moisture Content of The Cottonseed.

The cleaned seed was weighed and the mass taken as  $y_1$ , the sample was then dried in an oven and the weight was obtained as  $y_2$ . The percentage moisture in the seed was calculated using the formula below.

$$\% \text{ Moisture content} = \frac{y_1 - y_2}{y_2} \times 100$$

Where  $Y_1$  = weight of sample before drying

$Y_2$  = weight of sample after drying.

### 3.3 Extractions of Oil Form Cottonseeds Using Soxhlet Extractor.

**Procedure: -**

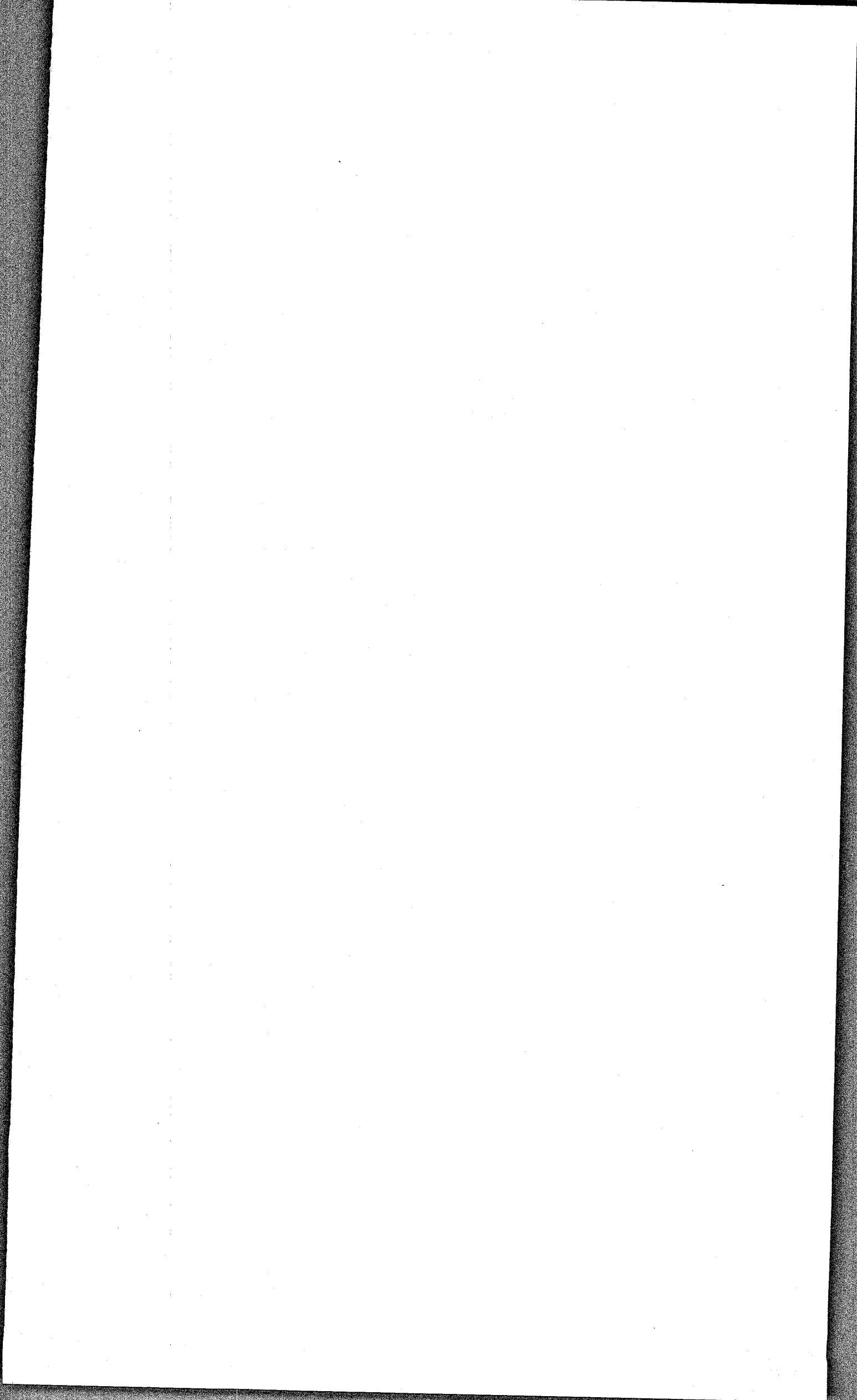
15.0g of the crushed sample were weighed and poured into different thimbles. The thimbles were then covered with cotton wool carefully to avoid particles of the sample entering the oil mixture then it is been put inside the soxhlet extractor.

250ml of n-hexane was poured into the extractor flasks. The soxhlet apparatus was then assembled and heated up using an electromagnet.

After heating and condensing for the required time, the thimbles were brought out and the miscella (which is the hexane oil mixture) in the round bottom flask was distilled using the same soxhlet apparatus.

After recovering a reasonable amount of the solvent, the source of heat was put off. The extracted sample residue inside the thimbles were removed and dried in the oven for about 30mins and then weighed. The remaining solvent entrained in the extract in the round bottom flask was recovered by simple batch distillation process.

The difference between the weights of the sample before and after extraction gives the approximate weight of the oil extracted.



### 3.3.1 Flow chart

The figure below shows the schematic flow diagram of vegetable oil extracted from the cottonseeds.

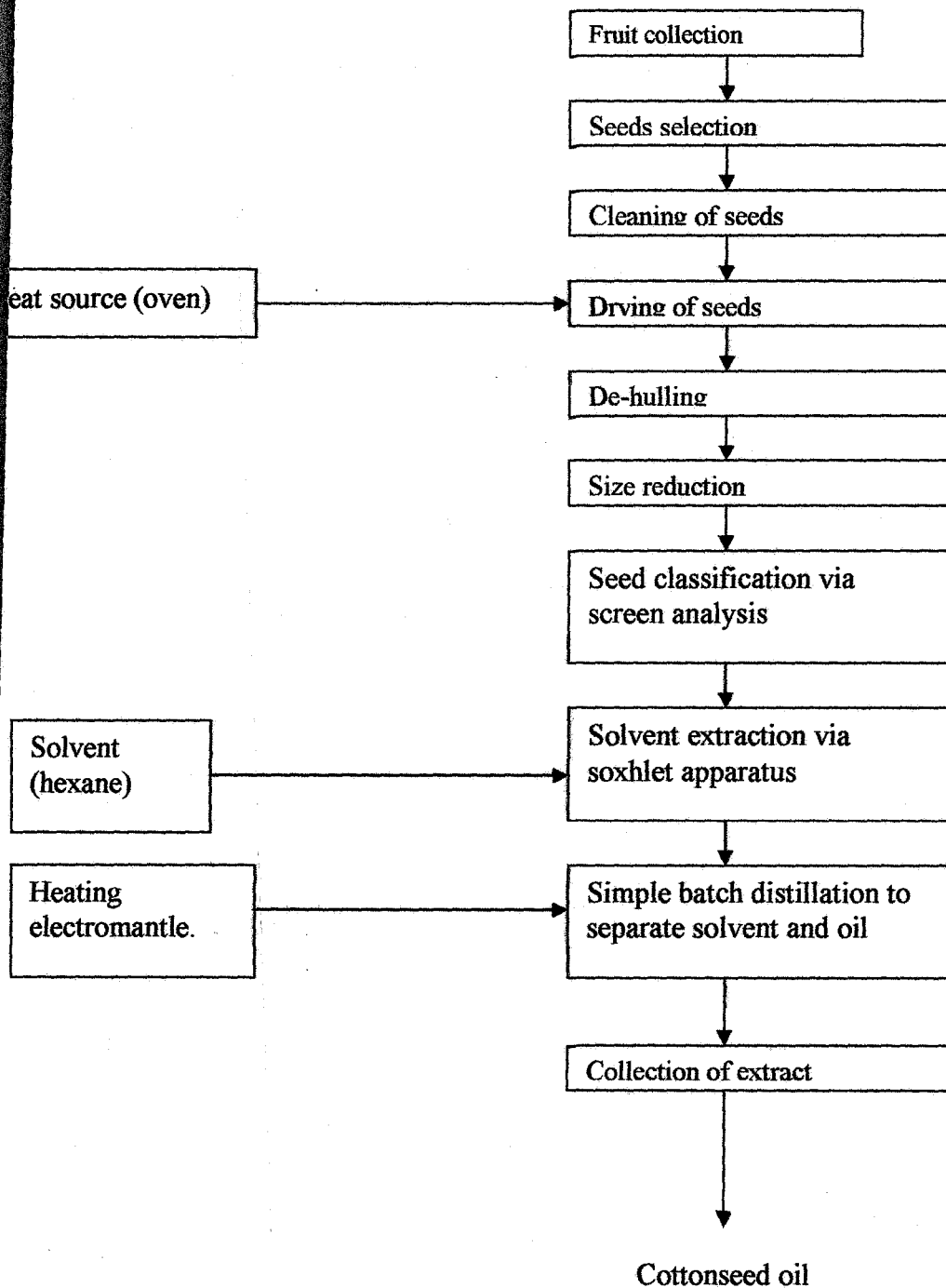


Figure a: flow diagram of cottonseed oil extraction

### **3.4 Characterization of Cottonseed Oil**

**3.4.1 pH determination using pH meter:** the pH meter was switched on for about 30 min to warm up. The pH of the oil was determined by dipping the reference electrode into the oil and a corresponding deflection read off on the meter. The result obtained is shown in chapter 4.

#### **3.4.2 Refractive Index**

Few drops of oil were placed on the surface of a glass prism of a refractometer, thoroughly spread, closed and tightened. Enough time was allowed for the oil and prism to attain a steady temperature. The refractive index was read from the demarcation line.

#### **3.4.3 Specific gravity**

Specific gravity is the ratio of weight of a substance to the weight of the same volume of water both at specified temperature. A 50ml measuring cylinder was weighed ( $W_0$ ). The measuring cylinder was filled with 50ml of water and re-weighed ( $W_1$ ). The measuring cylinder plus oil was weighed ( $W_2$ ), specific gravity (S<sub>g</sub>) was computed with the formula:

$$S_g = \frac{W_2 - W_0}{W_1 - W_0}$$

#### **3.4.4 Odour or smell**

To receive the full spectrum of the aroma of cottonseed oil, hold the bottle containing the oil below your nose, gently move it back and front from the left nostril to the right. This is used also to determine and identify oil.

#### **3.4.5 Acid Value Determination**

About 12g of the oil was taken into a 250ml conical flask. 50ml of a mixture of equal volume of 95% ethanol and diethyl ether was added to it, the whole mixture was agitated and heated to boiling. A drop of phenolphthalein