

**EXTRACTION OF ALMOND SEED AND CHARACTERIZATION**

*(Terminalia Catappa)*

**BY**

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**FACULTY OF PHYSICAL SCIENCES**

**UNIVERSITY OF BENIN**

**BENIN CITY.**

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**A ONE YEAR PROJECT PRESENTED TO THE DEPARTMENT OF CHEMISTRY  
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**SUPERVISED BY**

**PROF OGBEIFUN**

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## **DEDICATION**

This project work is dedicated to the Almighty God who without Him I am incomplete. IT is an honor also to dedicate this work to my awesome parents, Mr. & Mrs. Grace Amaja, whose support and encouragement has made this work to be a reality.

## **ACKNOWLEDGEMENT**

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## ABSTRACT

The oil was extracted from tropical almond seed using soxhlet apparatus and n hexane .The oil obtained was characterized. The moisture obtained was 21.60% , saponification value 216mgKOH/g, acid value 16.4mgKOH/g , peroxide value 6mEq 03/kg, Ash content 4.8%, iodine value 86mg/100g, Density value 0.87, pH8.04 and free fatty acid 8.2 . The chemical properties of oil showed that the oil contain high saponification value with low iodine value hence the oil is not recommended for soap making but for cosmetics industries, essential ingredients for body development.

# CHAPTER ONE

## 1.1 INTRODUCTION

### 1.1.1 BACKGROUND OF PROBLEMS

Tropical almond (*Terminalia catappa*) is a large spreading tree now distributed throughout the tropics in coastal environment, the tree is tolerant of strong wind, salt, spray and moderately high in the root zone. It grows principally in freely drained well aerated, sandy soils. The species has traditionally been very important for coastal communities, providing a wide range of non-wood products and services. It has a spreading, fibrous root system and plays a vital role in stabilization. It is widely planted throughout the tropics, especially along sandy seashores for shade, ornamental purposes. The timber makes a useful and decorative general purposes hardwood, fruits are produced from about 5 years of age, and the nutritious tasty seed kernels may be eaten immediately after extraction. Selected cultivators of the species warrant wide commercial planting for joint production of timber and nuts, there is also a need for experimental work to develop propagation techniques and more efficient including drying. Soil texture prefer light to medium soils, (sands, Sandy loams, and Sandy clay loams).

Tropical almond is easily propagated from seed, and is fast growing and flourishes with minimal maintenance in suitable environment, *Terminalia catappa* also known as (Ebeleboh) by whom to have originated in Malaysia, the seed oils can be used for both human consumption and for industrial application.

Seed oils are vital sources of nutritional oils, and are of industrial, pharmaceutical importance. They have composition and no oil from single sources can be suitable for all purposes (Mohammed and Jorf-Thomas, 2003). consequently with increasing awareness of important of these seed oils there is a need for application, most seed oil extraction have been performed with organic solvent such as hexane but studies have shown that this solvent is rapidly increase costs and uncertain, According to international journal and engineering research volume 7 issued 5<sup>th</sup> May 2016 say that due to the increased ability of solvent to overcome forces that binds liquid within the sample matrix (lumely et al, 1991). It is possible to extract components such as unsaturated fatty acids and avoid any toxic solvent residue in the products (king and list 1994). the oil can be used for cooking for its health benefits and in cosmetics (hand and body lotions, shampoos (Aremu et al, 2006a) as it is valued for its regenerative and moisturizing properties. in Africa and some developing countries, manual processes, where hard objects are used to crack the shells are popular, once the nuts are cracked, the seed been removed dried either by sun or an oven, then by doing extraction process.

Seed oils are extracted either by mechanical pressing, or squeezing, or with solvents. Before the 1940's, mechanical pressing was the method used. According to Aremu et al, (2015) the oil is stable for a long period of time and protect against peroxide and have presence of natural antioxidant, the seed have vitamins as well as other phytochemicals and acid valued and it help in the purification of blood. According to Foildl et al, (2001), the oil externally applied for curing skin diseases due to the antimicrobial ,antiseptic and anti-inflammatory properties content in it (Ruttarattanamongkol et al, 2014). extraction can be defined as a separation process consisting in the separation of a substance from a matrix; **oil is extracted from**, which is the nut for cooking .and the work focuses on the extraction of oil from the **seed using a solvent apparatus**, determine the physiochemical **properties of the oil content**, pour point ,**specific gravity, viscosity**, refractive index, iodine value, acid value and peroxide value. This oil study was to establish the suitability of oils for domestic and industrial uses. The plant is also called Indian almond, is a large spreading tree now distributed throughout the tropics in coastal environments. The seeds are often of small sizes and **difficult to extract from the nuts**,These factors have contributed for its lack of use **in many area (Adu et al 2013)**.The tree is tolerant to strong winds ,salt spray and moderately high salinity in the root zone if grow principally in freely drained and well aerated Sandy soils. There is an Increasing need to search for oils from non - conventional sources and also meet specific application (Kyari ,2008). Ajaji et al, **Oderinde,V.O**

**Taiwo**,et al) evaluated the short term toxicology of three kinds of oil seeds in rats ,and found that oil obtained from Terminalia, catappa seeds **had the least deleterious effect**, and was more suitable for consumption **,In addition J.T.A .Oliveria, I.M .Vasconcelos, L.C.N. M Bezerra**,et al. Indicate that the oil content of Terminalia catappa (583.0g1kg dry matter ) is comparable to that of other oil seeds.

### **1.1.2 STATEMENT OF PROBLEM**

Since almost part of the tree as seeds, leaves, and roots have benefits, research were done in order to study the characteristics, properties, yield and uses of the products obtained. The research focuses more on the extraction of oil from terminalia catappa [almond] seed, to achieved high yield of the oil, and characterization, the fascinating thing about these oils is that it can be applied directly to the skin without any refining or bleaching. In Nigeria and other parts of the world, there been increasing cases of after shave burns and skin related illness. Studies have shown that the almond oil (ebeleboh) helps the skin in related issued. Oil is commonly used in cooking, use as skin moisturizers, helping with dry skin. This project work is designed to assay for the extraction of an almond oil (ebeleboh), the benefits and health impacts and the guide to solving other skin issued.

### **1.1.3 JUSTIFICATION /RELEVANCE OF THE RESEARCH WORK**

In Africa, Nigeria and even Benin City there has been a rise in skin related diseases such as acne, swollen skin, and dry skin amongst others. But these problems or skin diseases can be reduced to the minimum, if the almond oil is used in our day to day life to moisturize the skin and prevent other skin related diseases. Therefore, if this project work is done accordingly and measures are set in place, the occurrence of skin diseases will be reduced to the barest minimum.

### **1.1.4 SCOPE OF WORK**

The research work covers feasibility study and acquisition of the necessary raw materials required for the production .the analyses how the almond oil (Ebeleboh seed) can be produced using extraction techniques. The focus of this study is to extract oil from the seeds gotten from south south part of Nigeria; another important factor is to ensure that the physiochemical analysis of almond oil correlated with the standard values.

### **1.1.5 AIM(S) AND OBJECTIVES**

1. To study the physiochemical properties of the oil and determine oil content
2. To study the extraction of T. catappa oil using heptane as solvent in Soxhlet extraction process.
3. To characterize the T.catappa seeds used in the extraction study.

### **1.2.0 LITERATURE AND REVIEW**

*Terminalia catappa* (ebeleboh) is a tropical edible fruit that belongs to the family of combretaceae and wind tolerance and produce fruits (5-10cm long ) with a thin fresh surrounding, large fibrous nut. The tree are large to the extend grows well in tropical diameter, the seed can be eaten raw, grow to a height of 35m with an upright symmetrical crown and horizontal branches. *terminalia catappa* is known for its nutritional values and possess medicinal benefit as well (Ajala and Adeleke, 2014; Aremu et al, 2015) . oil seeds crop are major sources of lipids, and which have shown various health benefits fats and oil can be used in food, according to (international journal of scientific and engineering research, volume 6. Issued 10, October 2015). The *terminalia catappa* are in green turning brown to purple when ripe, is good for smoothing skin and brain power according to Aremu et al, 2015. stable for a long period of time and protect against disease and peroxidant in presence of natural vitamin such as A and C as well as other phytochemicals, (Remu et al, 2006a) says it can be used also for shampoos, acid valued is higher reported by Bello and Agge and the peroxide value obtained for the oil slightly higher reported by Barku et al, the moisture content of tropical seed was unknow ,this value is lower than that was reported by Mastos, el al. antiplasmodial activity of new Caledonia and Vanuatu traditional medicines pharm boil, 2011 apr; 49 [4]: 369-76. Almond seed originates at the Middle East, India subcontinent and North Africa. it can resist the temperature under 20 degree and the oil are volatile, natural complex compounds characterized by a strong aroma ,and are also promising sources of

natural medicine products (Maedeh,M.et al 2011). The composition of oil is mainly oleic acid (68%) ,Almond oil has been researched for several health promoting properties, including, prevention of cardiovascular diseases (Damesceno et al,2011;Ros, 2009). Studies also showed that both whole almond and almond oil consumption caused similar reductions in plasma cholesterol and LDL (4% and 6% respectively ) as well as a 14% decrease in fasting plasma triacylglycerol's (Hyson et al,2002). Extraction of whole almond seed, brown skin ,shell ,and green shell cover (Hull) bear potent free radical -scavenging capacities (Sirwardhana Wijeratne et al, 2006; Moure et Al. 2007; Sfahlan et Al .2009).These activities are related to the presence of Flavonoids and other phenolic compounds recalling the need of the molecular composition approach ( Shahidi et al.2009) produce by several methods which can be generally categorized into dry methods (Bawalan & Chapman,2006).

In contrast, the dry method, the kernel is dried by controlled heating to remove the moisture, preventing microbial invasion from occurring, fatty acid content, free acid, Iodine value, peroxide value, saponification value, and viscosity In addition total phenolic content and antioxidants capacity and the composition of oil are known to affect its rate of oxidation, nutritional value, preservation properties and shelf life (Murillo et al, 1999). Several review on the quality have been reported (Amri, 2011, Belitz & Grosch, 1999, Gopala Krishna et al 2010, Marina Cheman & Amin 2009) and its physiochemical properties have been standardized by the Asian. All parts of the plant that is, leaves, kernel bark

root wood and fruit are used for medical purposes. The leaf and Bark extract of the tree possesses Anti-carcinogenic, Anti- HIV, Hepatoprotective, Anti - Diabetic and Liver regenerating effects. The leaves are Anti sickling in nature, it is beneficial for liver detoxification and support the Immune system .The antioxidant property of leaves prevent the breakdown of the chromosomes and Anticlastogenic effect. The kernel of the almond tree possesses Aphrodisiacs property, it cures sexual dysfunction and it aids healthy reproductive system in men. The fruits is helpful in the treatment of Leprosy, Headache and reduced Nausea associated from traveling, it used extremely for healing wounds and ulcers. It stops bleeding during Foot extraction. It is used to treat infection of mouth, throat and intestines caused by yeast infection. The species is known to form natural root graft and has natural pruning characterization and use for cooking in South America (Adu et al,2013).The size large 25-40m(82-130ft) tall, and subtropical maritime climates with annual rainfall generally 1000 to 3500mm (40-140m) elevation below 300-400m and the soil adopted to a wide range of higher texture soil types. Tropical almond is easily propagated from seed, and is fast growing and flourishes with maintenance in suitable environments. Soil acidity acid to neutral / midly alkaline, soils (pH4.0-0.5). It is also known as sea -almond and false kamahi ,it is referred to as Ebeleboh in Benin .The branches offering clusters of foot long ,obviate leaves that turn pink - red to red yellow before falling. Some of the pigments responsible for this are: violaxantin, Litein and zeaxanthin. The young tree display a characteristics

Pagoda form, with a single bole and monopodia horizontal branching in regular false whorls of 4-5 branches, along each lateral, new branches are formed in a bifurcating pattern.

The treed crown become flatter with wide spread branches in older specimens ,In Tonga he reported fruiting season varies between island groups eg , September to December in the South (Tongatapu and Eua ) and February - May further north (Ha'apai and Vava'v ) .The rind of the fruit is a light, pithy, or corky tissue that enables the fruit to float and be dispersed by sea currents .Tree are also away from coasts due to fruits being carried inland and dropped by frugivowers birds and bats ,and as a result of deliberate planting by humans ,two fruits types have been reported from the Mussau Islands, Bismarck Archipelago ,Papua New Guinea (Lepofsky 1992) one type has a soft endocarp that can easily be broken with the teeth ,while the other has a hard endocarp that must be hit with a stone or cut with a knife to extract the nut. The almond oil can be taken from both the bitter almond and the sweet almond varieties. The tree principally is drained, well aerated sandy soils and it product lots of seeds seasonally that are usually consumed minimally as edible fruit, with little attention given to the utilization of the kernels, (Liu et al, 2009 ; *et al*, 2010 ; Sritiet al; 2011; Sulaim et al, 2013 ). It is popularly known as Indian-almond, sea –almond, tropical-almond and false kamani. It is often referred to as Ebeleboh in Benin .The seed can be eaten alone or for garnishing dishes.

### 1.2.1 ALMOND OIL (TERMINALIA CATAPPA)

Almond oil is an edible oil obtained from fresh and matured kernel of almond seed (Marina et al, 2009), a tropical plant belonging to Combretaceae family. Almond oil is colorless to brown yellow with the aroma of fresh almond, and it has been largely consumed for many purposes in cooking, bakery, confectionary, infant foods, and cosmetics. In cosmetics, the almond oil is utilized as a substance to enhance beauty, to promote the growth of hairs, and to improve and moisture skin. Another emerging application of the almond oil is the health supplement area, due to health benefits of medium chain fatty acids contained in the almond oil. Medium chain fatty acids such as lauric, palmitic, stearic and linoleic acids are easily digestible (DebMandal & Mandal, 2011). The lauric acid component in the almond oil, was reported to show potential for anti-obesity treatments (Assuncao et al., 2009; Nevin & Rajamohan, 2004; St-Onge & Jones, 2002). The almond oil is rapidly gaining popularity because of high stability and various health advantages (Carandang 2008). The almond oil also possesses antioxidant properties that boost the immune system. Therefore, consumption of the almond oil does not undergo any hydrolytic and atmospheric oxidation as confirmed by low peroxide value as well as very low free fatty acid content (Marina et al, 2009c: patil et al. 2016). Despite the vast impact of almond plants as a whole and its health important to humanity, most people still lack the basic knowledge in this plant and relatively few studies has been done to ascertain its health impact.

### **1.2.2 FATS AND OILS**

Fats and oils are obtained from a huge assortment of plant and animal sources. The difference between fat and oil is simply that the former is plastic or semi-solid at room temperature while the latter is liquid (Manley, 2000b). Edible fats and oils are composed of triacylglycerols. The type of fatty acid at every position profoundly affects the physical behavior of fat and oil to their performance stability. The important of fats and oils commences from their functionality which is due to their chemical composition and structural aspects (Belton, 2000). Fats and oils have a great impact on the texture of the final products .According to (deMan, 1998) the functional properties of commercial fats are strongly allied to their fatty acid and triacylglycerol composition. The words fats, oils and lipids are all used to refer to fats with different characteristics especially physical state. Word fat is generally used to refer to fats which exist as solid at normal room temperature and word oils is used to refer to fats which exists as liquids at normal room temperature while word lipids refer to both liquid and solid fats (Anther, 1993). Like most organic materials, oils and fats are made up of three elements :

1. Carbon
2. Hydrogen
3. Oxygen

### **1.2.3 METHODS OF EXTRACTION**

Various techniques such as mechanical extraction, solvent extraction, traditional extraction and super critical fluid extraction are used to obtain the oil from the seeds. The solvent has become the most popular method of extraction of oil because of its high percentage of oil recovery from the seeds. Solvent extraction bridges the gap between mechanical extraction which produces oil with high turbidity metal and water content and supercritical fluid extraction which is very expensive to build and maintain its facilities. Hexane is often used as solvent for oil extraction due to its lower boiling point for easy separation after extraction, its non –polar for extraction oils which generally nonpolar, yet hexane has been categorized as hazardous air population (HAP) by the US Environment Protection Agency and is included in the list of toxic chemicals (NIOSA, 2007). The maximum permissible limit for hexane in oil and the meal are 5ppm, and 10ppm, respectively (PFA, 1934). These problems have attracted researchers to find a suitable alternative solvent. A number of solvents and their mixtures such as n-hexane, alcohols etc. Several parameters such as particle size, volume of solvent, operating temperature and extraction time has been found to affect the percentage yield of oil from seeds. Hence, the determination of the effect of this parameters on oil yields from seeds is important to minimize the of oil, the cost of oil extraction in general and the amount of energy expended over time.

#### **1.2.4. TRADITIONAL EXTRACTION OF SEED OIL**

Traditionally, the commonest way of oil extraction is the water flotation process; oilseeds are thermally treated, crushed and milled into a container. With the aid of simple domestic utensils, oil is extracted by hand kneading. Water is added and mixture stirred and kneaded by hand until the oil separates to the top and sides of the utensils being used for the kneading. Water plays a vital role in hydrolyzing, which displaces oil from hydrophilic surface. This method is used, however, on a small scale, as it is labour-intensive, slow and tedious in operation compared to other methods but is assumed to produce high oil quality.

#### **1.2.5 MECHANICAL EXTRACTION OF SEED OIL**

The main application of mechanical expression are in the extraction of oils and juices. Expression is often combined with size reduction to maximize the yield of oil. Components are extracted from plants parts either for direct use or use in subsequent processing such as refining. In oil-bearing seeds, the oil is found inside cells in small droplets (10-80µm) in diameter (fellows, 1998). However, a single type of equipment is not suited to all oilseeds owing to variation in oil content, moisture content, porosity and solidity of the material, applied pressure, heating temperature, heating duration, particle size and shade, storage and handling practices, and the proportion of hulls in different oilseeds are factors influencing yield and quality of the oil expressed (waisis, 2000). Expression is achieved either two stages ( size reduction to produce ,

followed by separation in a press) or in a single stage, which both rupture the cells and express the oil. In general, the single –stage operation is more economical, permits higher throughputs and has lower capital and operational cost but not suitable for hard nuts as the two stage expression is more effective. The degree of effectiveness varies with the kind of oilseed and method of oil expression (Akinoso, 2006).

#### **1.2.6. SOLVENT EXTRACTION OF SEED OIL**

Solvent extraction is the use of chemicals as solvent in the extraction of oil from oilseeds. Solvent extraction is known for its high yielding oil output, ease and swiftness to carry out; relatively cost effective, high overhead cost, and hazardous effects during and after operations. The use of this method requires a complete refining process to ensure traces of the solvents are removed totally. Solvent extraction of cleaned, cracked, dehulled and conditioned thin soy flakes with hexane is commercial practiced to extract oil.(Becker, 1971; 1978, Galloway, 1976). Commercial solvent extraction does not include any pre-pressing operation due to the relative disadvantages of low oilcontent and slower oil recoveries. Becker (1978), Johnson & Lusas (1983) indicated that hexane, a petroleum –derived product has been extensively used as solvent for the oil extraction of soya beans and other oilseeds because of its low vapourisation temperature ( boiling point 63-69c), high stability,

low corrosiveness, low greasy residual effect, and better aroma and flavor productivity for the products.

### **1.2.7 EXTRACTION OF TROPICAL ALMOND SEED OIL**

The ground tropical almond seeds were wrapped in filter cloths woven from horse –hair in small batches of 400g each. They manually loaded into perforated, horizontal boxes below the head block and above the ram of the hydraulic press. The boxes were pressed together using upward hydraulic pressure on the ram. The hydraulic press type used was a commercial Mikel-Mexicol hydraulic press with a thermal casing, maximum pressure capacity of 68.65 Mpa, 150 mm piston diameter, 1000watt commercial strength at 127v and data logger system which had capacity for cold press and hot press extraction method. The ability of the hydraulic press was due to the presence of a k-type thermocouple capable of temperature – controlled oil extraction process. The thermocouple was maintained at a specific temperature during the extraction process in order to maintain the natural state of the extracted oil ( active principle) with minimal effects on oil quality. The oil was pressed out through the filter cloths and spent cakes were manually removed from the hydraulic press. The residual oil in each spent cake for each batch operation was less 5 %. On completion of the extraction process, the oil was allowed to stand undisturbed for 24 hours in a dark room so solid particles could settle to the bottom of the container, Thereafter, the oil was filtered using a fine

cloth and heated to 40c to drive off traces of water and destroy any bacteria present. Additionally, to ensure high purity and quality of the oil, the resulting oil was severally centrifuged at 1200 rpm for 20 minutes using a centrifuged (Mark iv, Auto Bench, Baird and Tatlock Ltd, London, UK) to further remove any containments present. The packaging and storage was done in clean dry containers were sealed against moisture air, and light to protect the oil from going rancid and kept in dark boxes to help increased the shelf-life.

Physiochemical characterization of the tropical almond seed oil (TASO)

The TASO obtained after extraction and clarification was subjected to physiochemical characterization and fatty acid profiling according to various standard methods (AOAC, AOCS, EN and ASTM) to determine the properties. Acid valued was determined using ASTM D664 method with a limits of 0.8mgKOH/g, kinematic viscosity at 40 c was determined by ASTM D44 method with limit of 1.9-6.0 using a Rheomat viscometer (Mettler Toledo, USA), iodine value was determined by ASTM D554 and saponification value was determined by ASTM D558. Oil color was measured using a colorimeter (CR 300, Konica Minolta, Japan ) according to ASTM D1500-12 standard. The fatty acid composition or profile of the TASO was determined by GC method in accordance with ASTM D6584, EN14214 and EN14105 using a Thermo scientific Trace GC Ultra AS 3000 Auto-sampler gas chromatography to a flame

ionization detector. And moisture content was determined, peroxide value and refractive index.

## **CHAPTER TWO**

### **MATERIAL(S) AND METHODS**

#### **2.1 MATERIALS**

Seeds (*Terminalia catappa*)

Blender

Burette

Pipette

Beaker

Conical flask

Oven

##### **2.1.1 Chemical reagent**

Diethyl ether

Potassium hydroxide

Phenolphthalein indicator

Potassium iodide

N-hexane

Ethanol

Wij's reagent

Carbon tetrachloride

Distilled water

## **2.2 DESCRIPTION OF THE STUDY AREA**

The experiment was carried out in the main laboratory of the faculty of physical science, department of chemistry, University of Benin, Ugbowo campus, Benin city, Nigeria. University of Benin is located between latitude 6°30'N of the equator and longitude 5°40' and 6°E of the Greenwich Meridian in the forest zone with average temperature of 27.6C (NAA, 2014)

## **2.3 SOURCES OF ALMOND SEEDS**

Fruits of almond seeds (*TERMINALIA CATAPPA*), were collected from the faculty of Arts of University of Benin, Edo state. The fruits were sun-dried after which the seeds inside were removed mechanically. The seeds were then sun-dried for another two weeks after which they de-coated. The samples were grounded into powder using a manual grinder, packed in an air tight container and stored in a dessicator, ready for further

analysis. The climate in this area is characterized by high humidity, precipitation up to 4000mm per annum and relatively high temperature, averaging 28°C, all the chemicals reagent and solvent used in the experiment were of analytical grade and were products of sigma chemical ( St. Louis, Mo).

## **2.4. PREPARATION OF POWDER SEEDS**

The tropical almond seeds (TAS) were obtained from matured tropical almond fruits. The fruits were initially washed and separated into the seeds and the pulp. The seed preparation process included cleaning, drying, size reduction, hull removal, drying and extruding. The seeds were separated from the hull by cracking with a hammer and thereafter dried to reduce the moisture content. This was done to minimize degradation on seed storage. Afterwards, the seeds were crushed and ground using motorized industrial grinding machine.

### **2.4.1. EXTRACTION OF SEED OIL**

This was carried out in a 1000 ml Soxhlet apparatus on a heating mantle. The solvent used was n-hexane. The almond seed powder was packed inside a muslin cloth placed in a thimble of Soxhlet extractor. A round bottom flask containing n-hexane was fixed to the end of the extractor and a condenser was tightly fixed at the bottom end of the extractor.

The flask was heated at 60°C with the use of an electric mantle. The solvent then vaporized and condensed into the evaporator. The mixture obtained (solvent and oil) moved directly into a round bottom flask. The process continues for the specified time. Oil was recovered by distillation process using the same apparatus. The oil obtained was stored in a bottle for further uses.

## 2.5 OIL CHARACTERIZATION

### 2.5.1 Iodine Value

The Wij's method for determining Iodine value was employed. The iodine value of oil was determined by weighing 1g of the oil into a conical flask; 10ml of chloroform (CCL<sub>4</sub>) was added, warmed gently and allowed to cool for 10minutes. 250ml Wij's reagent was added with vigorous but careful swirling and placed in the dark to prevent reaction with sunlight for 30minutes. After the set time, 20ml of 10% KI and 150ml distilled water was added to the mixture and titrated against sodium thiosulphate until the appearance of a yellow colour. The resulting yellow mixture was titrated against sodium thiosulphate (Na<sub>2</sub>SO<sub>3</sub>) using 1% starch solution as an indicator until a change of colour from indigo / black colour to colourless (endpoint). A blank (water) test was carried out under the same condition.

$$\text{Iodine value} = 12.69 * N * (v_1 - v_2) / (\text{mass of oil in (g)}) \dots\dots\dots \text{equ 2.1}$$

Where:

$V_1$  = Volume of  $\text{Na}_2\text{SO}_3$  solution used for the blank test

$V_2$  = Volume of  $\text{Na}_2\text{SO}_3$  solution used for the test oil

$N$  = Normality of  $\text{Na}_2\text{SO}_3$  (i.e. 0.1N)

### **.2.5.2 Saponification Value**

1g of Oil sample was weighed into a conical flask and 25ml of 0.5M ethanolic KOH was added. The resulting mixture was heated under reflux for one hour. After the set time, 0.5ml of phenolphthalein was added and titrated with 0.5M HCL solution until change in colour from pink to colourless. A blank (distilled water) test was carried out under the same condition.

Saponification value =  $56.1 \times N \times (v_2 - v_1) / \text{mass of oil}$  .....equ 2.2

Where:

$N$  = factor of 0.5M HCL

$V_1$  = Titre volume of HCL solution used for blank

$V_2$  = Titre volume of HCL used for oil

### **2.5.3. Acid Value**

The acid value is the number of milligrams of the KOH necessary to neutralize the free fatty acid in 1g of the sample. It is the measure of free fatty acid present in the oil. 0.05N of KOH was prepared by dissolving 2.805g of KOH in 1000ml volumetric flask filled with distilled water which was then poured into a burette for titration.

Firstly, 10ml each of ethanol and benzene was measured in a volumetric cylinder and poured into a conical flask to which 2 drops of phenolphthalein indicator was added and titrated using the 0.05N KOH solution present in the burette. This was called a blank (EN 14104 standard method). Another 10ml each of ethanol and benzene was measured and used to dissolve 1g of oil previously measured in a conical flask, 2 drops of phenolphthalein indicator were added to observe an endpoint. The 0.05N KOH solution in the burette was added drop wise to the oil-alcohol-phenolphthalein solution and properly shaken until the solution stayed pink for 20 seconds. The millimeters obtained from the blank and main experiments were inputted into a formula to obtain the acid value as shown below.

$$\text{Acid value} = 0.05\text{N} \times 56.1 \times (v_2 - v_1) / \text{weight of oil} \dots\dots\dots \text{equ 2.3}$$

Where,

AV = Acid Value

$V_2$  = Change in volume of 0.05N KOH with oil

$V_1$  = Change in volume of 0.05N KOH without oil

0.05N = Concentration of KOH (Normality)

Molecular Mass of KOH = 56.1g/mol

Then;

Free fatty acid (FFA) = Acid value/2

Where;

FFA = Free fatty Acid

#### **2.5.4 .Moisture Content**

The moisture content is the weight of water in the oil expressed as a percentage. An empty crucible was weighed in an electronic mass balance. A given quantity of oil was then weighed in the crucible using the electronic mass balance. The crucible containing the oil was then placed in an oven at 80°C. At time interval of 30mins, the crucible with oil was taken out and weighed with a new mass for both oil and crucible obtained. The process was continued till constant weight of the crucible containing oil was obtained.

#### **2.5.5. Density**

Density is the ratio of mass of a sample to its volume. The oil was introduced into a density bottle of known volume, the density of the bottle was weighed initially when it was empty and then the value was recorded, after which the weight of the oil was measured alongside the density bottle and then recorded. The change in weight was noted by taking the difference between both values and recorded to determine the weight / mass of the oil, from which the density of the oil was determined by dividing the mass with known volume of the density bottle, the value was recorded in kg/ m<sup>3</sup> as it density.

Density = mass/volume

#### **2.5.6. Peroxide Value**

The peroxide value is defined as the amount of peroxide oxygen per 1kg of oil. Using 0.1N of sodium thiosulphate in burette, 12ml of acetic acid-chloroform

was measured in ratio of 3:2 into a 250ml conical flask to the acetic acid-chloroform, 0.2ml of saturated potassium iodide was added, it was swirled for 1 minute after which distilled water was added to liberate the iodine from the chloroform layer, then 1ml of starch solution was added and it was titrated against the sodium thiosulphate, until the blue grey colour disappears in the aqueous upper layer, the change in volume of the sodium thiosulphate was taken to be the blank, the procedure was repeated using 2g of the oil sample.

The peroxide value can be calculated using the formulae below:

$$\text{Peroxide value} = 10 \times (N_1 - N_2) / \text{weight of oil}$$

Where,

$N_1$  = Volume of sodium thiosulphate used for blank

$N_2$  = Volume of sodium thiosulphate used when 2.0 g of oil was used

Peroxide value	6MEq.O <sub>2</sub> /kg
Acid Value	16.4mgKOH/g
Saponification Value	216mgKOH/g
Moisture content	21.60%
Ash Content	4.8%
Iodine Value	86mgI/100g
Density	0.876
pH	8.04
Free fatty acid	8.2

### CHAPTER THREE

Table 3.1 Show physiochemical properties of Almond seed oil

Oil Extraction: The extracted oils were solid at room temperature. The oil content of Almond seeds is high, it was found to be  $58.30 \pm 2.35\%$  which shows

that the processing of the oil for industrial or edible purposes would be economical. This value is slightly higher than the value reported for the seed of *Sesamum indicum* L ( $57.0 \pm 1.27\%$ ), Nzikou et al., (2009), and also higher than that of *Moringa oleifera* ( $40.0 \pm 1.34\%$ ) by Nzikou et al., (2009).

From the table, the iodine value ( $86.0 \text{ mgI}_2/100\text{g}$ ) was lower than most oil such as Sesame *indicum* oil white ( $103 \text{ mgI}_2/100\text{g}$ ) and red ( $116 \text{ mgI}_2/100\text{g}$ ). The oil with iodine value less than 100 are non-drying oils, therefore, the low iodine value of the almond seed oil indicates that the oil is a non-drying oil and does not make it suitable for making paints and varnishes. The iodine value of oil must not be too high because high value denotes an excessive free fatty acid which causes the oil to turn sour, solidified and decolorized.

From the table, the saponification value of the extracted oil of almond seed was ( $216 \text{ mgKOH/g}$ ) which was higher than those of other oil such as quinoa oil ( $192.0\%$ ), butter fat ( $220-241\%$ ), cotton seed ( $190-200\%$ ) and soy bean ( $190-194\%$ ). The saponification value of greater than  $200 \text{ mgKOH/g}$  indicates high proportion of unsaturated fatty acids. This shows that the almond seed oil has a very high potential for use in soap making and industrial uses.

From the table, the moisture content of almond seed was  $21.62\%$ . This result therefore shows that almond seed has high moisture content hence cannot be preserved for a long time. This value is high when compared to  $5.5$  and  $5.1\%$  for cashew nut (fetuga et al 1974) and African oil bean (osagie et al 1986).

From the table, the ash content of almond seed was 4.6%. Ash content signifies the level of minerals present in the sample. The ash content of almond seed is high when compared to 3.3% recorded for cashew nut 8. (Fetuga et al 1974), it is also higher than the value of 2.7% obtained for African oil bean (Osagie et al 1986).

From the table, the peroxide value (6.00MEqKOH/g) was lower than the maximum acceptable value of 10MEqKOH/g set by the Codex Alimentarius. A high peroxide value indicates a poor resistance of the oil to peroxidation during storage. The low value obtained for the sample indicates that the oil would not easily go rancid when properly stored in a container free from atmospheric oxygen and other contaminants.

From the table, the acid value of almond seed oil was 16.4. This value is high when compared to 2.15 obtained for coconut oil (Peter 1956) and 4.30 for camphor seed oil (Osagie et al 1986). For soap making higher values are required, therefore the oil is useful for soap making with regard to acid values (Divine and Williams 1961). The free fatty acid value of the oil obtained was 8.2, this value is high when also compared to 3.45 obtained for melon seed (Ige et al 1984).

Viscosity is a measure of resistance of a fluid to deform under shear stress. It is commonly perceived as thickness, or resistance to pouring. Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid

friction. The viscosity at 25°C of this oil is given in Table 3. This result (26.92 ± 0.17 mPa.s) is in agreement with that found by Dos Santos, 2008.

## **CONCLUSION**

From the result of the study, it can be concluded that almond tree seed has a higher level of most of the chemical components. It is therefore a very promising raw material for various industries. Also, it would serve as a useful dietary supplement. Therefore, this seed must not be overlooked anymore. The high protein value of the seed and low level of anti-nutrient indicates its potential usefulness in animal and poultry feed supplements. The chemical properties of the almond seed oil shows that the oil contain high saponification value with low iodine value, low saponification value is ideal for soap making, hence the oil is not recommended for soap industry. Almond seed oil can also be consider for recommended for cosmetics industries, essential ingredients for body development. The high oil yield and high degree of unsaturation qualify its usefulness in the industrial manufacture of pharmaceuticals, soaps and cosmetics.

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