TITLE PAGE

EFFECT OF PARTICLE SIZE ON OIL YIELD USING SCENT BEAN SEED ('OZAKI').

BY

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CERTIFICATION

This project on the extraction and characterization of scent bean (Ozaki) was written and compiled in partial fulfillment of the requirement for the award of a bachelor of Engineering (B. ENG) degree in Chemical Engineering has been approved by the undersigned.

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DEDICATION

This project is dedicated to the Almighty God who had made it possible for me to survive throughout my stay in school. My lovely parents and to all my brothers for all their support and encouragement throughout this project.

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A maximum thanks that is sincere goes to my project supervisor Engr. Mrs. Otegbulu for her intense and transparent contributions which has lifted me academically and otherwise.

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ABSTRACT

This project was done to extract and characterize bean oil according to their particle sizes. The experiment was carried out using scent bean (i.e. 'Ozaki', 'Ijilizi'or 'Azamu') as sample. The oils were extracted by solvent extraction /leaching extraction using n-hexane. Proximate analysis was carried out to obtain percentage moisture content, ash content, total oil content, protein content and carbohydrate content of the extracted oils. From observation, it was noticed that as the diameter of the sieve decreased, the quantity of oil obtained increased

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CHAPTER ONE

1.0. INTRODUCTION

1.1. BACKGROUND OF THE STUDY

There has been an increase in the world production of oilseeds over the last thirty years (Murphy, 1994); this would appear to be related to the increasing demand for oilseed products and by-products as oilseeds are primarily grown for their oil and meal.

Oils from most edible oilseeds are used in the food industry, though there is growing emphasis on industrial utilization as feedstock for several industries with about 80% of the world production of vegetable oils for human consumption. The remaining 20% utilization is between animal and chemical industries (Murphy, 1994).

According to Rajagopal *et al.* (2005), bio-oils from oilseeds are used as Straight Vegetable Oil (SVO) or as biodiesel (trans esterified oil) depending on type of engine and level of blend of the oil; scent bean oil i.e. Ozaki, Ijiliji, or Azamu is found mainly in the South-East of Nigeria and is not an exception. This phenomenon has created a school of thought that it is better to use oilseeds as biofuel, which will lessen the competition for fossil fuels, which are not renewable. Fossil fuels are not only costly in terms of price but are also costly to the environment as they degrade land, pollute water and cause a general destabilization of the ecosystem with global warming as an end result. Furthermore, crude oil wields socio-political power that often dictates the pace of economic growth in specific locations, especially non-oil producing nations.

Nevertheless, the petroleum industry requires a greater quantity of oil to meet its demand.

Demand, however, by the food industry alone is not secure for many developing countries like Ghana that depend on imports of vegetable oil and fossil fuels. In order to meet the required amounts needed by all industries, these fats and oils must be available in large quantities locally with an effective extraction process at an affordable cost. The ability of a particular oilseed to fit into the growing industries depends on its utilization potential, rate of production, availability and ease of the processing technology. Thus while some oilseeds are being largely utilized in the oil processing industries, quite a number of oilseeds are underexploited.

Generally, oils and fats from seeds and nuts constitute an essential part of man's diet. Fats and oils, together with proteins, carbohydrates, vitamins and minerals, are the main nutrients required by the human body. Fats and oils are rich sources of energy, containing two and a half times the calories of carbohydrates (per unit

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weight). In addition to being a source of vitamins A, D, E and K, fats and oils also contain essential fatty acids. These essential fatty acids are not manufactured by the body and must be obtained from diets, with linoleic, oleic and linoleic acids as examples of unsaturated fatty acids (NRI, 1995).

Modern processing of vegetable oils yields valuable products such as oleo chemicals. Oleo chemicals are now largely being used in the manufacture of many industrial products, namely building auxiliaries, candles, detergents and cleaning agents, cosmetics, fire-extinguishing agents, flotation agents, food emulsifiers, insecticides, lubricants, paints, paper, medicine and chemicals. The meal or cake is used in the formulation and preparation of livestock feeds and food additives.

The production of oil plants takes third place in the world production in terms of value, after starchy plants and fruits, and ahead of beverages and stimulants. Edible seeds and nuts noted for their oil contents include palm nut, coconut, soya bean, olive, groundnut, sunflower seed, and cottonseed, while non-edible seeds and nuts include jatropha seed, neem seed, and castor bean. Moreover, bean oil has strengthened its dominant role among fats and oils produced based on its quality and nutritional grade. Bean oil contains linoleic, oleic and linoleic acids that are found in many plant oils. Shortage of these fatty acids leads to deficiency symptoms especially in growing children and animals. Bean oil has the highest content of lecithin (1.1-3.2%) which is a surface-active compound used as an

emulsifier in the food and pharmaceutical industries, and other industries (Sigmund and Gustav, 1991).

Among the industries that use oils and fats from oilseeds, apart from the food industry, are the beauty, pharmaceuticals, aromatherapies, building and construction, and the petroleum industry.

1.2. Problem Statement

Many plants have been identified as sources of oil, with some of the plant species and their oil extracted and used as medicines and food. However, very few of these species have their oil characteristics determined.

Because of the high demand of oils for various purposes including medicinal, perfumery, soap making, insecticides et al. Imported oils are very expensive to meet the demands of our local consumer industries; therefore, it becomes necessary to source and synthesize these oils locally. Since these oils can be produced locally, it gives no reason for their importation or at least should reduce the rate at which these oils are imported and give attention to local production.

1.3. OBJECTIVES OF THE STUDY

The purpose of this study is to

- a. Find the percentage composition of oil in the bean seed
- b. To determine the effect of particle size on the yield of the oil.

1.4. SIGNIFICANCE OF THE STUDY

Exploitation of fruits and seeds as a source of oil can help to reduce oil costs by diversifying the sources for this commodity. Data generated from this study will benefit industries for production of oils for various purposes.

In addition the content and composition of fatty acids of plant seed oils can serve as plants taxonomic markers.

1.5. JUSTIFICATION OF THE RESEARCH

Some factors and benefits of bean ("Ozaki, Ijiliji or Azamu") oil make the research worthwhile;

1 The bean is readily available.

2 Oil from this particular bean is medicinal and applicable in pharmaceutical industries.

3 Small scale industries coming up as a result of oil extraction can reduce unemployment.

4 It can attract foreign exchange earnings for Nigeria.

CHAPTER TWO

2.0. LITERATURE REVIEW

2.1. PREAMBLE

Scent bean is also known as 'Ozaki', 'Ijiliji' or 'Azamu' in ogbo dialect. It is a leguminous plant found manly in the South-Eastern part of Nigeria.

The term *bean* originally referred to the seed of **the broad or fava bean**, but was later expanded to include members of the New World genus *Phaseolus*, such as the common bean and the runner bean, and the related genus *Vigna*. The term is now applied generally to many other related plants such as Old World soybeans, peas, chickpeas (garbanzos), vetches, and lupins.

Unlike the closely related pea, beans are a summer crop that needs warm temperatures to grow. Maturity is typically 55–60 days from planting to harvest. As the bean pods mature, they turn yellow and dry up, and the beans inside change from green to their mature colour.

Plant seed oils have a wide variety of structures, because oils do not occur in nature as single pure entities, but rather as complex mixtures of molecular species in which various fatty acids (FAs) and glycerin are present in different combinations. There are many different kinds of fats, but each is a variation on the same chemical structure. Triglycerides are the main constituents of vegetable oils. All fats consist of FAs (chains of carbon and hydrogen atoms, with a carboxylic acid group at one end) bonded to a backbone structure, often glycerol (a "backbone" of carbon, hydrogen, and oxygen). Chemically, this is a tri-ester of glycerol, an ester being the molecule formed from the reaction of the carboxylic acid and an organic alcohol. Oils are usually from plants while fats are from animal origin. Wikipedia, (2013).

2.2. Importance of oils

Many plant oils are used in food, in medicine, cosmetics and as fuels. They are consumed directly, or are used as ingredients in the preparation of food. Fat and oil are the most concentrated kind of energy that humans can use. They provide 9 kilocalories per gram of oil while the other two types of energy that humans can use i.e. carbohydrates and proteins provide 4 kilocalories per gram each. The Food and Agriculture Organization (FAO) and the World Health Organisation (WHO) have listed the important functions of dietary oils as a source of energy, cell structure and membrane functions, source of essential FAs, vehicle for oil-soluble vitamins and for control of blood lipids. Yaniv *et al.* (1999) made an assay of Citrullus colocynthis utilized for oil production, especially in Nigeria. Its oil contains a large amount of linoleic acid (C18:2) which is important for human nutrition (Yaniv *et al.* 1996). Such oil composition resembles safflower oil and is very beneficial in human diets. For treating some conditions, such as rheumatoid arthritis or diabetic neuropathy, one may try oils high in gamma linoleic acid, such as primrose oil. Here the oil is used as a medication to treat symptoms of a disease with both positive and negative effects. Consuming oils high in polyunsaturated FAs can lower blood cholesterol levels and thereby decrease the risk of cardiovascular diseases (Dagne and Jonsson, 1997). Some oils have medicinal properties while others can make excellent excipients in pharmaceutical and cosmetical preparations.

Although many plant oils have good structural values, it is not yet clear whether they can be safely consumed because toxicity has been associated with some oils. Even the most common commercial plant oils, such as canola (rapeseed), soybeans, cottonseed or castor oils in their crude form are not fit for human consumption without further processing. These processes include filtration, neutral and physical refining, fractionation, bleaching and deodorizing.

Refining removes undesirable impurities of oil whereas fractionation separates oils and fats on a commercial scale into two or more components. Fractionation

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increases oils range of use, shelf life and adds value. The deodorizing makes possible the production of neutral flavor food product i.e. tasteless and odorless oil while the colorless oil production is the obvious result of bleaching.

Plant oils are used to make soaps, skin products, candles, perfumes and other cosmetic products. High unsaturated oils are suitable as drying agents, and are used in making paints and other wood treatment products. They are also increasingly being used in the electrical industry as insulators since they are non-toxic to the environment, biodegradable if spilled and have high flash and fire point.

Many plant oils have similar fuel properties to those of diesel fuel and may substitute for this fuel, most significantly as engine fuel or for home heating oil. Crop plant oils already used as fuels include canola, sunflower, soybean and palm oils. They can be used in pure form but they are often blended with regular diesel.

2.3. Proximate Composition of oil

The key parameters in oilseeds are fats and moisture. Proximate determination provides information on the basic chemical composition of foods/feeds. The components of food or materials are moisture, ash, fat, protein, crude fibre and carbohydrate. These components are fundamental to the assessment of the nutritive quality and quantity of foodstuff being analysed.

2.3.1. Moisture Content

Moisture content is the measure of water in a material. The moisture content of foods is of great importance for many scientific, technical and economic reasons. Moisture determination is important in many industrial applications, for example, in the evaluation of material balance or processing losses. It is important to know the optimum moisture content when processing foods. The moisture content of food gives an indication of its shelf life and nutritive value, low moisture content is a requirement for long storage life. Compounds that volatilize under the same physical conditions as water also would be included; however, these are usually negligible (Aurand *et al*, 1987).

2.3.2. Ash Content

The ash content of a foodstuff is the inorganic residue remaining after the organic matter of the material has been burnt. The ash composition is not considered the same as the mineral content as there may be some loss due to volatilisation. It is a measure of the total mineral content in the foodstuff; the ash content can provide an estimate of the quality of the food product, since high levels may indicate contamination.

2.3.3. Crude Protein

Protein is composed of a large group of nitrogenous organic compounds that are essential constituents of living cells; they consist of polymers of amino acids and are essential for growth and repair of tissues. Generally, samples that do not contain unusually high concentrations of non-protein nitrogen-containing compounds may be analysed by simply determining the percentage of nitrogen (NH3) and making the assumption that nitrogen was released from protein during digestion. Soya bean protein provides all the essential amino acids needed to fulfil human nutritional requirements for growth, maintenance or physical stress (Robert and Nemat, 1998).

2.3.4. Crude Fat

Crude fat is the term used to refer to the crude mixture of fat-soluble materials present in a sample (Aurand *et al*, 1987). The fat content (sometimes called the ether extract, neutral fat or crude fat), which may be considered as consisting of the free lipid constituents, comprising essentially neutral fats and free fatty acids. The total lipid content of food is commonly determined by organic solvent extraction methods (Nielsen, 1994).

2.3.5. Crude Fibre

Crude fibre is a measure of the quantity of indigestible cellulose, pentosans, lignin and other similar components found in food (Aurand *et al*, 1987). Crude fibre consists of hemicelluloses, cellulose and lignin. Lignin comprises polymers of phenolic acids and hemicelluloses consist of heteropolymers of polysaccharides. Crude fibre is the insoluble and combustible organic residue after the treatment of a sample. Treatments include consecutive dissolution of the sample with light petroleum, boiling sample with dilute sulphuric acid and boiling sample with dilute sodium hydroxide (Kirk and Sawyer, 1991). Treatment with the solvents sulphuric acid (H2SO4) and sodium hydroxide (NaOH) removes all the protein and carbohydrate from the sample.

2.3.6. Carbohydrate

Carbohydrate is an essential structural component of living cells and source of energy; it includes simple sugars with small molecules as well as macromolecular substances. Classification of carbohydrate is based on the number of saccharide groups they contain as monosaccharide, oligosaccharides and polysaccharides (Pomeranz and Meloan, 1987). Carbohydrates are the most abundant and widely distributed food components in the world especially among roots and tubers, and cereals and legumes.

2.4. Concept of Vegetable Oil Extraction

Vegetable oil is a group of liquid edible fats and oils that are obtained from plants and seeds, and the extraction of vegetable oil is the processing of oilseeds and plants to remove oil for human consumption and industrial purposes. Edible vegetable oils and fats are components of foodstuffs, which are composed primarily of glycerides of fatty acids obtained only from plant sources. They may contain small amounts of other lipids such as phosphatides, unsaponifiable constituents and of free fatty acids naturally present in the fat or oil. Methods often used for the extraction process are mechanical and solvent methods. In most cases, the process involves two or more combinations of these methods to ensure optimum oil recovery. Traditionally, oil is extracted though oil recoveries are very low and are often for home consumption. Among the factors enhancing optimum oil yield in oilseeds are, moisture content and temperature.

2.4.1. The Role of Moisture and Temperature in Oil Extraction

Oil and water can each wet the solid components of oilseeds, though the two differ in affinity for the hydrophilic surfaces of particles. Water has a higher affinity and wets the surface of particles at a faster rate than oil due to its polarity and absorption ability. As such, the surface tension on the particles and water interface is insignificant while that of the oil interface is considerable. Research has proven that particles are selectively wetted by liquids with lower surface tension at the interface; hence water will tend to displace oil from the surface of particles. At a certain moisture content all the surface of the particles will be saturated by water and the oil will flow freely from the molecular forces. Thus, moisture increases the flow of oil through the pores of the press cake, hence reducing the amount of oil entrained in the cake and increasing the oil yield mostly in mechanical expression. High moisture content stops oil flow possibly because the structures of the finely milled particles have been altered (high aggregation). Moisture lubricates the pulp during pressing and causes a slower pressure increase and reduces oil yields (Sefah, 2006).

Temperature is increased for oilseeds after pre-treatments such as cracking, dehulling, and milling by heating, roasting and steaming of oilseeds prior to extraction and is termed thermal treatment of oilseeds. Better extraction is achieved by heating, which reduces the oil viscosity and releases oil from intact cells, and also reduces moisture in the cells. Temperature plays an active role in the seed treatment for mechanical extraction and ensures an effective solvent process by heating the solvent which hastens the extraction process. At the right temperature and moisture content, the individual oil droplets unite to form a continuous phase and flow out maximising oil yield.

2.4.2. Traditional Extraction of Vegetable Oil

Traditionally, the commonest way of oil extraction is the water flotation process; oilseeds are thermally treated, crushed and milled into slurry (paste). With the aid of simple domestic utensils, oil is extracted by hand kneading. Water is added to the slurry and the 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 the paste, which displaces oil from hydrophilic surfaces in the slurry.

Under the traditional method, there are two ways of extracting oil; wet and water assisted extractions. This method is used, however, on a small scale, as it is labourintensive, slow and tedious in operation compared to other methods but is assumed to produce high oil quality. In the wet extraction method, water is used in relatively large amounts to suspend the oilseeds such that the extracted oil floats on the top of the suspended oilseeds. Hot water flotation method of edible oil extraction is traditionally used in the rural areas of many developing countries. The waterassisted method involves the addition of small quantities of water to the slurry before the oil is extracted by manual kneading. The slurry is suspended in boiling water and boiled for at least 30 min with liberated oil floating on the surface. Further quantities of water are added after boiling to replace the lost water that occurred during evaporation, and to facilitate the floatation of the oil to the surface. The oil is carefully scooped from the surface of the water and boiled.

2.4.3. Solvent Extraction of Vegetable Oil / leaching method

Solvent extraction is the use of chemicals as solvents 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 (0.25-0.30 mm) with hexane is commercially practised to extract oil. Commercial solvent extraction does not include any pre-pressing operation due to the relative disadvantages of low oil content and slower oil recoveries. Becker (1978), and Johnson and 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°-69°C), high stability, low corrosiveness, low greasy residual effect, and better aroma and flavour productivity for the milled products.

2.4.3.1. Solvent Characteristics

In the search for a replacement for hexane, isopropyl alcohol and petroleum ether were chosen for this research. These solvents were selected on the basis of their availability, ability to extract oil from soya beans with as much recovery as hexane, and affordability. Below are the descriptive characteristics of the solvents.

2.4.3.1.1. Isopropyl Alcohol

Isopropyl alcohol (IPA, isopropanol, propan-2-ol, 2-propanol, C3H8O) produced from the combination of water and propene, like acetone, dissolves a wide range of non-polar compounds. It is relatively non-toxic and dries quickly. Isopropyl alcohol vapour is heavier than air and is highly flammable with a very wide combustible range. Isopropyl alcohol has a density of 0.786 g/cm3, melting point of -89oC, a molar mass of 60.10 g/mol, and is miscible in water. It should, however, be kept away from heat and open flame. When mixed with air or other oxidizers, it can explode through deflagration and is reported to form explosive peroxides (Wikipedia, 2008). Isopropyl alcohol is described as an efficient and advantageous extraction solvent for soya beans and other oilseeds as an attractive alternative to extraction grade hexane (Lusas *et al*). IPA extraction produces high quality oil that requires less refining, produces high quality meal that requires less toasting, uses less energy and is safe, and is less toxic. IPA has a higher vaporization temperature (boiling point 82.3oC) than hexane, only a small portion of the total IPA in the system requires vaporization and energy savings should result. Owing to the significant amount of trypsin inhibitor inactivation achieved, the desolventising- toasting (D-T) cycle is considerably shorter, producing a high quality meal. Gandhi et al. (2003) obtained the highest degree of purity in soya bean oil for extraction with IPA and reported that IPA extraction process is equally effective when compared with hexane.

2.4.3.1.2. Petroleum Ether

Petroleum ether (benzine, naphtha, petroleum naphtha, petroleum spirit, X4 or ligroin), is a group of various volatile, highly flammable and irritant, liquid hydrocarbon mixtures used chiefly as non-polar solvents. Petroleum ether is obtained from petroleum refineries as the portion of the distillate which is intermediate between the lighter naphtha and the heavier kerosene. Petroleum ether has a specific gravity of between 0.625 - 0.660 g/cm3 depending on its composition and is not water soluble (Wikipedia, 2013). It has a boiling point of 20°C - 75°C, melting point of -73°C, and a molar mass of between 87-90 g/mol. Petroleum ether being a petroleum product also faces occasional scarcity and

fluctuation in price depending upon supply and demand of gasoline (Johnson and Lusas, 1983, Seth *et al.*, 2007).

2.4.3.1.3. Mechanism of Mass Transfer in Solvent Extraction

The transfer of soluble material from a particle by the actions of a solvent is termed leaching or percolation. Leaching is a complex mechanism involving the transfer of the solvent to the surface of the solid particles, penetration of the solvent into the solid, dissolution of the solute into the solvent, diffusion of the solute into the solvent and transfer of the solute to the bulk solvent (Adu-Amankwa, 2006). Based on the different phenomena for the leaching process, it becomes virtually impractical to use any one theory to explain or describe the leaching activity. The dissolution of a sample from the solid to the liquid phase depends on the rate of mass transfer from the solid surface to the solvent as the controlling factor.

The mass transfer rate of a solute A being dissolved in a solvent of volume V (m3) is

$$N_{A} = k_{L}a(C_{AS} - C_{A}) \tag{1}$$

where,

N_A is the kg/mol of A dissolving to the solution per second,

a = Inter-surface area of the particles (m2),

 k_L = Mass transfer coefficient (m/s),

 C_{AS} = Saturation solubility of the solute (kg/mol/m3)

 C_A = Time dependent concentration of the solute.

The rate of accumulation of A into the solvent by material balance is:

$$\frac{Vd_{CA}}{dt} = k_L a (C_{AS} - C_A)$$
(2)

By integration of equation (2) for t = 0, $C_A = C_{A0} = 0$, t = tf, $C_A = C_{AS}$, we have;

$$\frac{C_{AS} - C_A}{C_{AS} - C_{AO}} = \ell^{-(k_L)t}$$
(3)

These equations aid in the understanding and calculation of the process of leaching of oil or liquid from a solid substance using a solvent.

2.5. Mechanical Expression of Vegetable Oil

The main applications of mechanical expression are in the extraction of oils and juices. Expression is often combined with size reduction to maximise the yield of product. Components are extracted from plant parts either for direct use or for use in subsequent processing such as refining. In oil-bearing seeds, the oil is found inside cells in small droplets (10-80µm) in diameter. 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 shape, storage and handling practices, and the proportions of hulls in different oilseeds are factors influencing yield and quality of vegetable oil expressed.

Expression is achieved either in two stages (size reduction to produce pulp or slurry, 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 of expression is more effective. The degree of effectiveness varies with the kind of oilseed and method of oil expression (Akinoso, 2006).

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Screw expellers are observed to be more efficient in oil extraction. This is due to their ability to combine size reduction (milling) and oil expression at the same time. As mentioned however, mechanical expression gives relatively low oil yields in bean seeds. De-oiling of beans follows pre-treatments such as dehulling, cracking, grinding or flaking, heating or steaming to enhance the quantity of oil extracted.

2.6. QUALITY OIL ASSESMENT

The physical characteristics of an oil or fat are dependent on such factors as seed source, degree of unsaturation, length of carbon chains, isomeric forms of the fatty acids, molecular structure of the glycerides and processing.

Fats that are liquid at room temperature tend to be more unsaturated than those that appear to be solid. The degree of unsaturation can be expressed in terms of the iodine value of the fat. As the chain length of the fatty acid increases, the melting point also increases. Thus, a short chain saturated fatty acid such as butyric acid and will have a lower melting point than saturated fatty acids with longer chains and even some of the higher molecular weight unsaturated fatty acids such as oleic acid. Most with longer chains is solid appearing at about 78°F (25.6°C).

The molecular structure of triglycerides can also affect the properties of an oil. A single triglyceride will have a sharp melting point. A mixture of triglycerides, as is typical of lard and finished vegetable oil will have a broad melting range.

A mixture of several triglycerides has a melting point lower than the average of the melting points of the individual components. The mixture also has a broader melting range than any of its components.

2.6.1. OBJECTIVE METHOD OF ASSESING OIL QUALITY

The most prevalent cause of poor flavour in oils is oxidation, which leads to aldehydes, ketones, alcohols and other compounds that affect flavor. Methods were therefore designed to measure the degree of oxidation or certain by-products.

- Peroxide value (PV): A measure of the primary products of oil oxidation, the peroxide. The peroxides themselves have no flavor and breakdown readily. Correlation of measurement and flavor is variable.
- Anisidine value: Determine level of aldehydes, primarily 2-alkenals that are derived from peroxides. The totox value equals the anisidine value plus twice the PV. This sum is felt to be a measure of past and current oxidative history.
- Thio-barbituric acid test: measures malonaldehyde, which has been shown to correlate well with PV of oils containing fatty acids of three or more double bonds.

- Conjugated diene and triene: Double bonds conjugate as oxidation occurs, of value; more with oils of known composition.
- Volatiles: Direct measurement by gas-liquid chromatography of responsible compounds. This approach continues to receive considerable attention.
- Predictive test: Oil quality predictive tests that have been applied include active oxygen method (AOM), American Society for Testing and Materials (ASTM), oxygen bomb, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA).

2.6.2. PROPERTIES OF OILS

FLAVOUR: The flavour of a product is considered the prime determinant in its acceptance. It also needs to be realized that poor or off flavor will cause product failure. The major cause of consumer complaints to the food industry is off flavor, and products are withdrawn from the market because of its defect. Oxidation can be auto-oxidation, photo-oxidation, and enzymatic oxidation. Auto oxidation proceeds through free radical chain reactions via the attack on the alpha methylene to the double bond. Photo oxidation is much faster reaction that catalysis the attack at the double bond by singlet oxygen formation. Enzymatic oxidation of lipids is caused primarily by the lipoxygenases. Plant

lipoxygenases produce hydro peroxides that produce the secondary products important to flavor of many food products besides oils.

Plasticity: From melting point, it was seen that a shortening that appears solid to the eye at room temperature is really composed of both solid fats and liquid oils. At 21°C, a typical shortening that appears solid contain 15-20 percent solids and hence, 80-85 percent liquid oil. As temperature drops, more of the oils solidify and the shortening becomes progressively firmer. On the other hand, as the temperature rises, more solid fats melts and the shortening becomes progressively softer until it has practically no 'body' or plasticity at all, and eventually it becomes completely melted.

OILINESS: Fatty materials feel oily and they have the ability to form oily or lubricating films. In preparing some food products, such as grilled foods, thus lubricating action is very important.

VISCOSITY: Viscosity of oils decreases slightly with an increase in unsaturation, therefore, viscosity is increased slightly by hydrogenation. Oils /fats containing a greater proportion of fatty acids of relatively low molecular weight are slightly less viscous than ones of an equivalent degree of

unsaturation but containing a higher proportion of high molecular weight acids. The viscosity of highly polymerized oils is much greater than that of normal oils.

SOLUBILITY: Oils and fats are almost completely insoluble in water. When they are held together in systems such as cake batters, these systems require the use of food emulsifiers and mechanical means such as homogenizers. These same oils, fats, and fatty acids are completely miscible in many organic solvents, such as hydrocarbons, ethers, esters, and so on. This is specially true at temperatures above the melting points of the fats or oils in question. With increased temperature, the rapidity and completeness of solubility increases. Hexane is a very important solvent, it is used to extract vegetable oils from their oil seeds.

SPECIFIC GRAVITY: is a comparison of the weight of an oil with that of the same volume of water. The specific gravity of vegetable oils is usually about 0.910-0.920 at 25°C. As the temperature increases, the S.G of the oil or fat decreases. As oils/fats are higher than water (specific gravity less than 1000), when mixtures of water and oils/fat separate, the oil/fat will be on top of the water.
CHAPTER THREE

3.0. MATERIALS AND METHODS

In order to achieve the objective of this project "extraction and analysis of oil from the bean seed" certain laboratory work was carried out. Therefore, this chapter shall focus on the equipments/apparatus used and the procedures involved in the experimentation, thus, the following stages/steps shall be our guide:

- i. Pre-processing and processing
- ii. Extraction and separation
- iii. Proximate analysis.

3.1. RAW MATERIALS AND EQUIPMENTS USED

All the necessary equipment required for carrying out this experimental work was provided by Caritas University Laboratory, The chemicals and reagents were bought from the chemical market while the bean seed was purchased at Ogbete market Enugu, Enugu State.

The apparatus and chemicals used for the experiment are listed below:

3.1.1. EQUIPMENTS

Electric oven, laboratory grinder, electrical thermostatic waterbath, electronic weighing scale, stop watch, sieve shaker SS20, measuring cylinder, conical flask (250ml), beakers (250ml), Whatman filter paper (125mm⁴), sieves of various sizes.

3.1.2. REAGENT

Reagent used was N-hexane for extraction.

3.3. OIL EXTRACTION AND SEPERATION EXPERIMENTS

The extraction of oil from this local seed was done using solid-liquid extraction otherwise known as leaching.

PROCEDURE:

The seed is weighed with the weighing balance to get d weight in grams of the sample to be used. The sample is then placed inside the electric oven for an hour at temperature of 70°C.when removed, the sample is grinded and placed inside the sieve that seperates the bean into various sizes according to the diameters of the sieves of different layers which have diameters of 60,30,25,22,14,12,8 and final diameter. All the eight samples obtained are weighed individually using the weighing balance. The sample with the least weight is used as the reference weight for the other samples to be measured. When other samples are weighed on the

same value as that of the reference sample, they are all placed inside a filter paper and tied up and placed inside the conical flask. 200ml of n-hexane is measure and poured in each of the tied bean samples with a foil used to cover the flasks to avoid evaporation of the n-hexane and kept inside a waterbath at a temperature of 40°C and allowed to stay for a period of two hours in order to ensure sufficient extraction of the oil from the bean seed. After two hours duration, the mixture of the n-hexane and the extracted oils was separated from the solid chaff by filtration using the funnel and the filter paper. Thus, the n-hexane solvents were separated from the extracted oil by gently heating of the mixture to evaporate the solvent. During the heating, the n-hexane solvents were separated from the extracted oil in the conical flask.

CHAPTER FOUR

4.0. RESULTS AND DICUSSION

The results of the various experiment carried out in this research work are reported and discussed in this chapter.

4.1. EXPERIMENTAL RESULTS

Weight of seed sample used = 100g

Waterbath temperature = 40° C

Volume of n-hexane for each sample = 200ml

Time interval for the extraction = 8-10 hours

Weight of reference sample : 14.70g

The yield of the oil are expressed as the percentage of the extract from the seed:

• % oil yield = W<u>eight of oil extracted (g) \times 100</u>

Weight of seed sample used (g) 1

% oil yield

For 60 = 246mm: 11.77%

For 30 = 500mm: 12.11%

For 25 = 600mm: 12.38%

For 22 = 699mm: 12.59%

For 14 = 1204mm: 13.06%

For 12 = 1405mm: 13.47%

For 8 = 2057mm : 14.29%

PAN sieve: 15.03%

After grinding of the beans sample, they are placed inside a sieve that seperates the grinded beans into apartments inside the sieve according to their sizes. Ranging from 246mm diameter of the sieve to the PAN layer. The samples are collected from each layer of the sieve/shaker and weighed. The sample with the least weight is taken as the reference sample in which other samples are weighed and used for extraction. **Table 1** below show the weight of the samples obtained from the different sieve layers.

When the beans samples have been tied and placed inside a conical flask containing 200ml of n-hexane. The flask is covered with a foil to avoid evaporation and each sample of the tied bean was placed inside a waterbath set at a temperature of 40°C. After a duration of about 8-10 hours inside the water bath, the samples were collected and rinsed with n-hexane before weighing. Table 2 below show the result of the bean samples after they had been leached.

After leaching, the weights of the beakers containing the oil extracts were measured. The oil extracts are then poured into a separate flasks while the weight of the beakers are obtained using a weighing balance so as to obtain the exact value of oil extracted from the beans samples. Table 3 shows detailed information about the values obtained.

s/n	Seive	Weight of	Weight
	size/diameter	sample(g)	of
	(mm)		sample(g)
			2
1	60 = 246	22.25	22.24
2	30 = 500	22.20	22.29
3	25 = 600	14.47	14.64
4	22 = 699	66.05	66.06
5	14 = 1204	37.30	37.07
6	12 = 1405	306.01	306.04
7	8 = 2057	16.67	16.67
8	PAN	15.61	15.60

Table 2:	Weight	of Sample afte	er Leaching
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s/n	Seive	Weight	Weight (g)2
	size/diameter	(g) of	of sample
	(mm)	sample	
1	60 = 246	14.35	14.35
2	30 = 500	12.86	12.29
3	25 = 600	14.13	13.94
4	22 = 699	15.10	13.66
5	14 = 1204	15.20	14.22
6	12 = 1405	15.80	14.29
7	8 = 2057	14.86	13.85
8	PAN	13.63	13.63

Table 3: Weight of Oil Obtained

s/n	Seive	Weight of oil +	Weight of	Weight of
	size/diameter	Beaker (g),A	Beaker (g),B	oil, A-B (g)
	(mm)			
1	60 = 246	85.29	83.55	1.73
2	30 = 500	84.73	82.95	1.78
3	25 = 600	134.63	132.81	1.82
4	22 = 699	85.06	83.21	1.85
5	14 = 1204	109.79	107.87	1.92
6	12 = 1405	84.43	82.45	1.98
7	8 = 2057	86.18	84.08	2.10
8	PAN	84.43	82.22	2.21

CHAPTER FIVE

CONCLUSION AND RECOMENDATION

The extraction of oil was done using the solvent extraction at the same conditions of temperature and time, and the same quantity of solvent (N-Hexane). Also the proximate analysis experiment portrays that the oils has no content of the ash, protein and carbohydrate contents. But they have very small amount of moisture.

The beans oil can be used for culineary purposes (in the food industry and domestic cookings). It is also applied in making of soaps, skin products, perfumes, and other personal care and cosmetic products. The viscosity of the oil bean is found to be very small, hence it can be applied in making engine greese and other lubricating oils. Also due to its high stability, it can be used for many industrial applications.

This research work was a very task full one. During the period of this research work, we were faced with lots of challenges ranging from lack of information due to poor library facilities and inadequate equipment and apparatus in the laboratory. Another close challenge that faced us during the time of the research work was lack of fund as most of the required materials and reagent for the project were very expensive. Despite all the challenges, the research was a huge success because we were able to successfully obtain all our results from the experiments done. Having carefully and keenly taken part in the research work and taken note of the percentage yield of the oil from the bean seed, I hereby strongly recommend the government, investors, industrialists and other business individuals with the capital to invest into the extraction of vegetable oil from these local seeds including these bean seed. This will help to increase the availability of these oils in the market, provide employment and improve exportation of vegetable oil from Nigeria, thereby making our economy an industrialist economy.

APPENDICES

APPENDIX A:

The percentage of oil yield

% oil yield : <u>Weight of oil extracted (g)</u> \times <u>100</u>

Weight of seed used (g) 1

FOR 60 = 246mm diameter seive

% oil yield = 1.73×100

14.70

= 11.77 %

FOR 30 = 500mm diameter seive

% oil yield = 1.78×100

14.70

= 12.11 %

FOR 25 = 600mm diameter sieve

% oil yield = 1.82×100

14.70

= 12.38 %

FOR 22 = 699mm diameter sieve

% oil yield = 1.85×100

14.70

= 12.59 %

FOR 14 = 1204mm diameter sieve

% oil yield =
$$1.92 \times 100$$

= 13.06 %

14.70

FOR 12 = 1405mm diameter sieve

% oil yield =
$$1.98 \times 100$$

14.70
= 13.47 %

FOR 8 = 2057mm diameter sieve

% oil yield = 2.10×100

14.70

= 14.29 %

PAN sieve

% oil yield = 2.21×100

= 15.03%

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