ANALYSIS OF BUSH PEAR AND ITS OIL

BY

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CERTIFICATION

This is to certify that the project “ANALYSIS OF BUSH PEAR AND ITS OIL” was carried out by OnyemetuIfeoma Jane in accordance with the relations governing the presentation of the project for the award of bachelor of engineering in degree (B. ENG) in chemical engineering, Caritas University Enugu

APPROVAL PAGE

This is to approve that this project was carried out by OnyemetuIfeoma Jane, CHE/2007/144, in partial fulfillment of the requirement for the award of Bachelors of Engineering (B. Eng) in chemical engineering, Caritas university.

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DEDICATION

This work is dedicated to God Almighty, the giver of wisdom, the great protector, the great provider, hope restorer and author my life, to my beloved parents Mr.Onyemetu John c. and Mrs.OnyemetuMargreat for their prayers, financial and moral support throughout the tough time.

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ABSTRACT

The research project studies the analysis of pear and its oil. The mesocarp from edible African pear "DacryodesEdulis" were evaluated for their oil yield. The pulp from this pear were oven dried at 100^{o}C-105^{o}C to a moisture content level of 29%.The mesocarp was subjected to proximate analysis to determine the percentage of the moisture, ash, fat, crude fibre, crude protein and carbohydrate content which resulted to the values of 29%, 2%, 19.6%, 25.5%, 11.9%, and 12% respectively. Then the dried sample was pulverized by using hammer mill and the oil was extracted by solvent extraction using n-hexane. The oil extracted were analyzed for the chemical properties i.e. (Acid value, saponification value, peroxide value, iodine value) etc. the values obtained are respectively 8.41gm/KOH/gm, 185.1gm/KOH/gm, 2.8gm/KOH/gm 3.96gm/iodine/gm and Physical properties i.e. (Refractive index, Ph value, specific gravity) which the values obtained are 1.469brix, 5.7 and 0.92. and the The percentage oil yield content is 51.57%. This physio-chemical characteristic and fatty acid composition of this oil show that they have industrial potentials.

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CHAPTER ONE

1.0 INTRODUCTION

In the major world, one major source of protein and vegetable oil is from oil seeds /fruits (Williams M. A. 1996). Oil constitutes a well defined class of naturally occurring substance. It is greasy, being soluble in organic solvents but insoluble in polar solvents such as water. Oil is a liquid at room temperature. Commercially, oil as well as fats is sourced from certain plant groups mostly seeds and nuts and some parts of animal within which they occur in relatively large quantity in an easily available form (McGraw-Hill, 1997). The existence of oil in certain plants has been known for century of years (Ogbu 2005).Oil can be grouped into edible and non-edible oil depending on the amount of unsaponified matters and impurities contained therein. Edible oil extracted from African pear, bread fruits, cashew nut, peanut etc. are examples of vegetable oil which are naturally occurring esters of higher fatty acids and glycerol, and are predominantly triglycerides with traces of mono and diglycerides, sterples, anti-oxidants, vitamins, saturated and unsaturated free fatty acids and other minor constituents. They are widely distributed in nature and were first consumed as food. Later, oils were discovered to be used as renewable raw materials for variety of non-food production. For instance; soaps, creams, disinfectants, paints, enamels, inks etc.

Due to the oil boom in the early 70's, agriculture was abandoned for petroleum and its product, but recently, things are taking a new turn in Nigeria over dependence on petroleum for virtually everything has not really helped matters. The economic situation in the country is bad and the general standard of living is getting poorer with each passing day, Hence, the need for a restructure of the economic system with an agricultural bias.

Most agriculture products such as these oils extracted from local seeds and nut, if properly monitored and harvested can be very useful for us down here and even exported for foreign exchange, hence the need for this project which deals on the extraction and analysis of African pear oil.

Extraction of oil from various vegetable resources is of ancient origin. In fact, the natives from different tropical regions of the globe have long been extracting oil from numerous oil-bearing plants. Humans, since the ancient times have known how to extract fats and oil from their natural resources. Historically, oils had been extracted by wrapping nuts in clothes and then using devices operated by stones and levers to exert pressure on them. But now, an improved form of mechanical device, which allowed considerably more pressure to be exerted, is now in use in form of hydraulic operated ram. This type of press is developed into a motorized hydraulic pump system that pressed the nut bag and then released a pressed cake. The next improvement in extracting oil is the screw press or expeller which is been driven by electric

motor.Because most press or expeller processes leads to over-heating of the meal and leave too- much of the high value oil in cakes, better methods of extracting the oil with solvent was developed. Bush pear was processed by solvent methods alone in this project. This process can be accomplished by a variety of ways but as might be expected, its efficiency depends to a great extent on attaining intimate contact between the liquid solvent and the solid containing the solute. The type of solvent available for this process include n-hexane, petroleum ether, benzene, n-heptanes, acetone etc.

1.1 HISTORICAL BACKGROUND OF THE STUDY

The generic name “Dacryodes” was derived from the Greek word “Dakruon” meaning [tear] referring to resin droplets on the bank surface of its member while “Edulis” means edible emphasizing the importance of nutrients fruits in the plants cultivation .The plant belongs to the family Burseraceae whose members are characterized by an ovary of 2to 5 cells, prominent as inducts in the bark, wood, and intrasteminal disk (Chunduff, 1984). The genus Dacryodes consist of about 10 species (Verheji, 2002). However (Rehn, 1984) indicated 80 species to encompass sub species of varieties, form and cultivars. Two varieties are recognized; Var-parvicarpa and Var-edulis whose conical fruit is smaller with the pulp. Var-edulis exhibit verticulate or sub-verticulate branching while

the branching is slender and opposite or bifurcate in var-parvicarpa (Okafor et.al 1983).

Dacryodesedulis is an indigenous fruit in the Gulf of Guinea and central African countries(Troupin, 1950), but due to the popularity of the nutritious fruit for consumption, the plant is widely cultivated, extend its area of distribution to Sierra-Leone, Uganda, Angola, Zimbabwe and Nigeria. It rarely grows wild, thus the natural area of distribution is obscure (verheji,2002). Lam gave four synonyms viz to the Dacryodesedulis :CarnaruimedulieHook.f, CarnaruimsaphuEngl, Pachylobusedulis (G.don) Hook. F. and PachylobussaphuEngl (Burkill ,1985,National research council,1996).

However, these synonyms have long been considered as the most unambiguous synonyms (Boutelje,1980).The common names are in English, African pear, African pear tree, Bush butter, Bush butter tree, Bush fruit tree, Eben tree, Native pear (Kapseu and Tchiegang, 1996) and in French, Safoutier (Burkill, 1985). The oil of fruits of DacryodesEdulis is a rich source of amino acids and triglycerides. The fatty acid composition of fruit pulp oil of two cultivars of bush pear [cultivar1 and cultivar2] grown in Nigeria were determined. The oil is found in the pulp which is made up of 48% of oil and a plantation can produce 7.8 tons of oil per hectare. It is also rich in vitamins and a rich source of amino acids triglycerides (Derbyshire.et al 1976).

Bush pear oil is one of the most important rated versatile vegetable oil. This is due to its uses in various spheres of life, most especially as a very healthy food ingredient.

1.2 AIMS AND OBJECTIVES

This project is aimed at the analysis of bush pear and its oil.The main objective of this study is to carry out proximate analysis and physio-chemical properties of African pear oil extracted by solvent methods. This physio-chemical properties determined are specific gravity, refractive index, ph value, boiling point, acid value, iodine value, peroxide value, and saponification value. To achieve the objectives of this project, it is important to:-

a) Select the best suited solvent for optimum yield.

b) Characterize the extracted oil for compositions and properties.

c) Test the suitability of the oil.

1.3 STATEMENT OF THE PROBLEM

This research work involves the analysis of African pear and its oil though the food crop African pear potential is rated one of the highest oil producing fruit crop yet it begs the question of its potential.

Furthermore, this project will answer the following questions;

i) Solvent extraction by solvent method

ii) What is the optimum yield of the particle size using n- hexane?

iii) Is there significant difference in the characterization of the extracted oil as compared to theoretical value in terms of;

1) Chemical properties (Acid value, iodine value, saponification value and peroxide value).
2) Physical properties (specific gravity, density, viscosity, refractive index).
3) Chemical composition (protein crude, fibre, carbohydrate and moisture.

1.4 SIGNIFICANCE/ECONOMIC IMPORTANCE OF THE STUDY

Characterizing the potentials of African pear/African pear oil for many purposes has several implications. Communities in the West African countries are significantly dependent on financial gain from agrarian enterprise.

It is hope that from the project, optimum extraction parameters which are quality of the oil would be established, the result would add to the data bank that could help potential industrialist who intends to go into vegetable table oil production from African pear. A crop that experiences a post harvest loss in excess of 40% in areas where malnutrition is prevalent is a problem for potentials to reclaim the lost percentage of either food or other purposes is advantageous for producers and consumers alike.

Furthermore, the development of crops with indigenous appeal can strengthen the agricultural and energy sectors of struggling economics, identifying the oils fuels quantities, whether favorable or not, will help to inform future crop and industry development.

1.5 SCOPE AND LIMITATION OF THE STUDY

In this project work, we intend to analyze and extract completely African pear and its oil. Many research work has been done in giving a detailed composition found in African pear. But this research account on the optimum route to:

i) Extraction of vegetable oil from bush pear.

ii) Separation of pure oil from the solvent.

iii) Characterization of the African pear.

iv) Characterization of the oil extracted.

Due to the low yield of the above method (mechanical method) of extraction, soxhlet and cold method of extraction are used i.e. solvent extraction.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 INTRODUCTION

This chapter represents the theoretical valuable information on bush pear and its oil, the method of extraction, its analysis and scientific information needed to support the research or study. In chemical engineering practice, materials handled are often contained on a solid matrix or contaminated with impurities in some cases and hence require separation and purification respectively in order to bring it to a state convenient for chemical operations.

Vegetables oils are water-insoluble substances of plant origin, which consists predominantly of long-chain fatty acid esters derived from the single alcohol, glycerol, $HOCH_2CHOHCH_2OH$, and are known as simple triglycerides. A triglyceride is known as simple triglycerides, if all the fatty acids are identical (Derbyshire.et al, 1976).

Although, fats and oils are predominantly triglycerides as noted earlier, there are a number of minor components which are present in both crude and refined fats and oil, the important ones are phospholipids, sterols, vitamins and their precursors, antioxidants, pigments, free fatty acids and some impurities[Ajewole K.1996].These components affects color, odor, and other qualities of the oil.

African pear are fruits on-seeded with pulpy perical about 5mm thick and thin cartilaginous endocarp, seeds are oblong ellipsoid up to 5.5cm long, whereas the flesh is pale to light green [FAO, 1982].The pear is broken to remove the seeds, the pulp was dried in an oven (Galen camp at 100-105^{o}c). The dried pulp was grounded to coarse powder, Hence is further processed to extract the oil in a process known as extraction (McGraw-Hill, 1977). The extracted oil was stored in sample container ready for analysis.

2.2 EXTRACTION

Extraction involves using two immiscible phases to separate a solute from one phase into the other, therefore extraction is a separation process which involves the removal of a solute from a mixture by chemical or physical method. Oil extraction is the isolation of oil from animal by-products, fleshy fruits, olive palm, oil bearing seeds and nuts.

Extraction depends on five basic factors: Preparation, solvent, contact, Time, and Temperature.

a) **PREPARATION**: All kinds of extractors require proper preparation of the oil seed to achieve good results and one often hears the phrase “it all begins in preparation” The preparation area/steps includes cracking, dehulling, flaking etc.

b) **SOLVENT**: Pure, good quality solvent, with enough flow rate for the tonnage processed, and with a sufficient number of effective counter current stages.

c) **CONTACT:** correctly prepared material with high surface area, good porosity, and a machine providing a good contact for effective washing and drainage.

d) **TIME**: Perhaps 30-50mins for pear depending on bed depth, thickness of the pulp and other variables to allow the dissolving and flow processes to occur.

e) **TEMPERATURE**: Hot enough to maintain rapid extraction in the time available (Williams, M. A; John Wiley and sons, 1996).

Extraction is of two major types: Mechanical /physical method and Chemical/solvent method.(Combination of both methods are being practiced in our various industries).

2.2.1 MECHANICAL/PHYSICAL METHOD OF EXTRACTION

This is a physical method that involves a mechanical process. It is an early means of separation of oil from oil seeds using physical pressure to squeeze the oil out, this process leaves intact the beneficial nutrient that is naturally contained in the seeds or flesh of the fruit and eliminate any possibility of solvent residual in the oil. The efficient method is the modern screw press, more than half of the oil is easily removed but perhaps 7% or 8% residual oil is left in

the solids, the process uses considerable horse power, there is considerable wear and maintenance (Joe Givens, Robert Jorhed Hein and George Anderson 1977).

2.2.2 CHEMICAL/SOLVENT METHOD

This is a separation process, which involves a substance usually a liquid that dissolves or is capable or dissolving the component of a solution present in greater amount. This liquid is known as a solvent, Solvent is any liquid in which another substance can be dissolved (Mosby's 2009, sanders, 2007). This method can also be called LEACHING in oil extraction. Examples of the solvent are benzene, carbon tetrachloride, isopropyl alcohol, ethanol, acetone, and n-hexane.

In this process, the oil is extracted from organic solvent, a modern process largely displacing extraction by pressure. The resulting cake or meal may be toxic example trichloroethylene extracted by soya bean (Joe Givens, RobertJordhein; George Anderson 1977). Distillation is used to recover the oil and last traces of solvent are removed with the aid of stream. In this method of extraction, the yield of oil is higher compared to the mechanical method, and the cost efficiency of this method is high compared to mechanical method.

Generally, extraction is essentially a mass transfer operation, therefore it is important to study the kinetics of the operation and processes. Extraction

operation is expressed in the different industries by various words such as leaching, washing-decantation.

Generally Extraction is essentially a mass transfer operation therefore it is important to study the kinetics of the operation and processes Extraction operation is expressed in the different industries by various words such as leaching washing decantation etc.

2.2.3 BASIC PRINCIPLES OF SOLVENT EXTRACTION

In this project work,solvent extraction was the method applied therefore more attention is needed.

Extraction by solvent involves the dissolution of the soluble constituents from a solid material by a means of a suitable solvent known as leaching. This type of extraction is a LIQUID-SOLID EXTRACTION. It is a unit operation in which components are separated by dissolving them in liquids.

The solute may be a solid or a liquid dispersed through the insoluble solids coating their surfaces dissolved in a liquid adhering (Mc Cade W. I. Smith, M. Craw Hill 1985) or entrained in the solids located within their cellular structure.

In extraction the method used is determined by the:

- Properties of soluble constituent present.
- Its distribution throughout the solid

- The nature of the solid.
- The particle size.

The solvent contacts the solute absorbed in solid and dissolves it and it therefore provides the product mixture as liquid rich in the solute. Though the process was accomplished in a variety of ways but its efficiency depends to a very large extent on attaining intimate contact between the liquid solvent and the solid containing the solute.

Three steps are involved in solvent extraction;

a) Change in the solute by dissolving in the solvent .The solute in the solid changes into liquid as it dissolves in the solvent.
b) Diffusion through the pores of the solid with the solvent by the solute to the surface of the solid.
c) The movement from the surface of the solid to the bulk of the solution. This method is either used to concentrate a solid valuable product or remove impurity from the solid such as pigments. In designing system for solvent extraction to produce a given amount of extract from a given quality of solid materials, this is generally accomplished using a material balance on the various components involved in the process.

2.2.4 CHOICE OF SOLVENT/SOLVENT PROPERTIES

The choice of solvent for use in the extraction of particular oil depends to a large extent on the following properties of the oil and the solvent;

- The solubility of the oil in the solvent.
- The inert condition of the solvent with the oil.
- The difference between the boiling point of the oil and solvent.
- The polarity condition of the oil and solvent.
- The end use of the oil.

-The vapor pressure and the flash point of the solvent as cited in the journal of Purdue food crop of Avocado oil, 2008.

For higher yield to be achieved the solvent must selectively dissolve the oil in preference to other components of the seed oil and the solvent must not react chemically. Also the viscosity of the solvent must be low enough not to hinder the diffusion processes before and after the oil dissolution[Bird et al,1990].The solids undergo mechanical and often thermal treatment before extraction to make the solute fit for solvent action. These pre-treatment can be of grinding, crushing, roasting, rolling, steaming etc.

2.2.5 APPLICATIONS OF SOLVENT EXTRACTION.

As applied in this research work, it is used in the extraction of vegetable oils from seeds/fruits such as African pear, breadfruit, soybeans etc by leaching with organic solvent such as n-hexane, acetone, and petroleum ether.

i) it plays an important role in the metallurgical processing of aluminum cobalt, manganese, nickel and zinc.

ii) Perfumes flavoring extracts and essential oils can be obtained from natural sources primarily through solvent extraction process in our industries.

iii) Soluble coffee is obtained from its bean and soluble tea from its leaf by hot water extraction and the caffeine can be removed and recovered from such materials by the use of the proper solvents.

iv) Solvent extraction has been applied on a commercial basis in the removal and recovery of oils and fats from raw wool garbage and a number of packing house by-products (McGraw-Hill, 1977).

Extraction operations are used extensively throughout the chemical and allied industries and have actual or potential applications whenever impure solids are processed.

2.2.6 METHODS OF LEACHING OPERATIONS.

Extraction may be accomplished in either a batch or continuous operation. Batch operation is employed where a large distribution ratio for the desired separation is readily obtainable. A small number of batch extractions readily remove the desired component, It may be carried out in simple separating funnel or stopper flask. Conversely when distribution is low, continuous methods are used. This procedure makes use of continuous flow of immiscible solvents through the solution until no more oil can be recovered by standard method of seed/vegetable oil estimation.

2.2.7 SOLVENT EXTRACTION THEORY.

This involves two different mechanism postulated for explaining solvent extraction of oils, they are; i) Molecular Diffusion Theory.

ii) Undissolved oil Theory.

i) **MOLECULAR DIFFUSION THEORY:** This theory was first postulated by Boucher and later verified by Osburn and ket and recently by others. Boucher and others derived the equation for diffusion extraction of oil in the following ways that when a porous solid containing a liquid is with a solvent, liquid inter diffusion of the molecules follows. This theory by Boucher and others was obtained from ficks law and some assumptions are made.

Thus $\frac{dc}{dt} = \frac{Dd^2c}{dc^2}$.....(1)

Where d=Diffusion constant defined by the amount of material which passes a plain of unit area in unit time under unit concentration gradient.

C=concentration of solute [oil] at point distance from the origin.

T =time (s).

Some assumption which the theory is based on is:

(a) The diffusion co-efficient must be constant and independent of thickness.

(b)The structure of the fruit must be reasonably homogenous and isotropic.

(c)The distribution of the oil in the cell must be uniform.

(d)The thickness of the slab must be small compared with the dimensions of the surface and diffusion via the edge of the slab must be neglible.

(e)The thickness of the slab must be the same. With these conditions, Boucher and others gave the solution of equation (1) as:

$$\frac{(C - C_1)}{(C_o - C_1)} = \frac{8}{\Pi^2} \frac{1e^{-(2n+1)_2}}{(2n+1)^2} \frac{Dt}{\Pi^2 (Z_1)^2} \text{----------(2)}$$

Where Z1=thickness of slab, D=Diffusion constant, C=conc. (weight per unit volume of oil at a point on the slab, C_0=Original uniform concentration in the slab, $C_{1=}$concentration of liquid solution into which the slab is immersed.

From the equation (2) above, $\frac{C - C_1}{C_o - C_1}$ may be represented by E.

Where E=Extractable oil content at anytime ÷Initial extractable oil content.

Also, Extractable oil=Total oil content-Equilibrium oil content.

Equation (2) reduce to:

$$E = \frac{8}{\Pi^2} \times E^{-\Pi 2} - \frac{Dt}{(Z_1)^2}$$

=From this equation, Osburn and Katz concluded by suggesting that the large portion of readily available oil was derived from ruptured cells, whereas the smaller portion of difficult extractable oil was in cells that remained intact.

ii) **UNDISSOLVED OIL THEORY**

This theory was proposed by Coasts and Karnosy, and according to the theory, the oil acts like a slowly dissolving material, then rate of solution of which is independent of the miscella concentration.

The theory is supported by the fact that there are slow dissolving constituents in the extractable that may inhibit that dissolution of oil. The theory also assumes that the diffusion through the cells is rapid compared with the rate of dissolution of oil, so that the mis-cella in the voids has the same concentration as the extracting solution.

The un-dissolved oil theory is further supported by the fact that the last portions of the oil are removed much more of the seeds which are first given a ‘soaking’ period even in relatively small miscella and that there is no difficulty encountered recovering the last portions of the oil from seeds. In effect, the complete theory of extraction seems to be a combination of diffusion and solution of slowly soluble extractable material (Shukla S.D).

2.2.8 FACTORS AFFECTING THE RATE OF EXTRACTION.

The selection of the equipment for an extraction process will be influenced by the factors which are responsible for limitating the extraction rate. thus, if the diffusion of the solute through the porous structure of the residual solid is the controlling factor, the material should be of small size, so that the distance the solute has to travel is small. On the other hand, if diffusion on the solution is sufficiently slow to control the process, a high degree of agitation of the fluid is called (Gooding 1985).Three major factors are to be considered:

1. **TEMPERATURE :**In most cases, the solubility of the material, which is being extracted will increase with temperature to give a higher rate of extraction, further, the diffusion co-efficient will be expected to increase with rise in temperature, and this will also improve the rate. In some cases, the upper limit of temperature is determined by secondary consideration, such as the necessity of preventing denaturing of oils from seeds.

2. **PARTICLE SIZE:** The particle size influences the extraction in a number of ways. The smaller the size, the greater is the interfacial areas between the solid and the liquid and therefore the higher is the rate of transfer of materials. It is generally desirable that the range of particle size should be small, so that each particle requires approximately the same time for extraction and in particular, the production of large amount of fine material should be avoided as it may wedge in the interstices of the large particle and impede the flow of the solvent.
3. **SOLVENT:** The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely. Generally, a relatively pure solvent will be used initially, but as the extraction proceed, the concentration of solute will increase and the rate of extraction will progressively decrease, first, because the concentration gradient will be reduced, and secondly, because the general solution becomes more viscous.

2.2.9 PROPERTIES OF SOLVENT

Not all solvents exhibits all the properties necessary for desirable extraction but should be selected based on certain qualities in which if neglected would result to ineffective leaching operation.

The following properties are considered;

- SOLUBILITY: It should be able to dissolve component of the mixture required to be separated.
- STABILITY: The solvent should be chemically stable when in contact with metallic surface.
- SEPARABILITY: The solvent should be able to be separated from the mixture.
- CHEAPNESS: The solvent should be affordable.
- TOXICITY: The solvent should be non-toxic (non-poisonous).
- CORROSIVENESS: A solvent should be non corrosive.
- RECOVERABILITY: A solvent should exhibit high volatility for effective and low cost recovery by distillation for re-use.

Obviously, no solvent fulfils all these conditions, but for practical purposes, only few solvents are feasible for use of which normal-hexane is of outmost importance.

2.3.0 EXTRACTORS

These are equipments used for extraction. Examples of these equipments are;

Boll man extractor, Bonalto extractor, Screw conveyor extractor, Kenedy extractor, Hildebrandt extractor and Soxhlet extractor.

But for this project, the soxhlet is extractor employed.

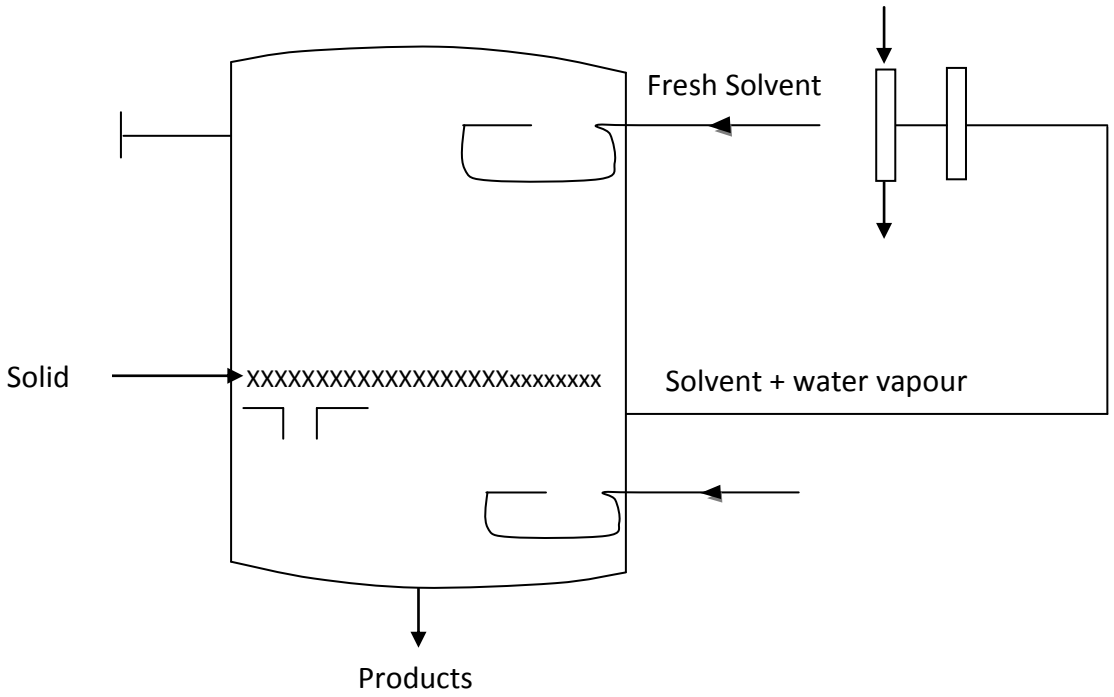


Fig. 1: A Diagram Of The Soxhlet Extractor

The fig above consists of a vertical cylindrical vessel divided into two sections by a slanting perforated floor. The upper section is charged with seeds which are sprayed with fresh solvent from a distributor. The solvent percolates through a bed of solids and drains into the lower compartment where together with any water extracted from the pear is continuously boiled off by means of a steam coil. The vapor are taken to an external condenser and the mixed liquid is passed to a separating box from which the solvent is continuously fed back to the extractor and the water is run to waste. In this way, a concentrated solution of the oil is produced.

2.3.1 DISTILLATION AS A SEPARATION TECHNIQUE.

Distillation is the separation of mixture cases with different boiling points into various individual components. It is applied to cases where all components are present in the same phase. This is the key operation in the chemical and allied industries. A feature common to all distillation processes is the tendency for the concentration of the more volatile component in the vapor phase, when the two phases are in contact.

Methods of distillation for a two component mixture include;

- Differential distillation.
- Flash/equilibrium distillation.
- Rectification.

Of the three, rectification is the most important because the vapor is condensed and retained as liquid in the distillation column whereas vapor are either removed or condensed as product in other two methods, while for multi-component mixtures, method of distillation are; Extractive distillation and Azeotropic distillation .

Therefore, in this research work, in order to reduce cost, this process must be carried out to recover the solvent for re-use.

2.3.2 CLASSIFICATION OF OILS

The degree of un-saturation determines the extent to which oils and fats could be used. Oils can be classified into: Drying oils, Non-Drying oils and Semi-Drying oils.

a) DRYING OILS. This refers to those oils with iodine number varying between 150 and 200.They have greater un-saturations. They absorb oxygen more or less readily when oxidized they are converted to a hard non-sticky film. The fatty acid comes in form of linoleic acid and their isomers. The important groups of drying oil include: linseed, tungperilla, dehydrated castor oil, and soy bean. These drying oils are consumed in large quantities in paints varnishes, plasticizers, lubricants, and brake lining (Codd L. W. 1984).

b) NON-DRYING OILS: This refers to those that are slowly attacked by the atmospheric oxygen, but remains liquid which considerable increases in viscosity even when oxidation at ordinary temperature occurs. The non-drying oils have low unsaturated as it contains less than 20% of linoleic acid. Coconut and olive are members of this group. Oils of greater un-saturation, example soy beans oils, may be used in similar products, if the unsaturated is lowered by hydrogenation.

Unlike drying, non-drying oils do not change to have solids, on exposure to air. Example include castor oil, coconut oil, palm oil, groundnut oil etc (Codd.L.W 1984).

c) SEMI- DRYING OILS: Oils classified as semi-drying are intermediate between the drying and the non-drying oils, in properties and contain about 40-60% linoleic acid. The iodine value ranges from 85-130.they exhibit a similar characteristic like the drying oils by absorbing more or less atmospheric oxygen thereby making the final product to be shift, tacky and semi-gelatinous which in actual fact, do not yield by any means completely hard and rigid dried or oxidized oil film. Examples of this class include soya bean which can be deodorized and inhibited with anti-oxidant to make an edible salad oil or hydrogenised to make plastic shortenings.

2.3.3 PROPERTIES OF OIL

It is of great importance to analyze oil in order to check its quality, purity, and also for identification. The identification of the oil in question means to precisely know the number of carbon molecule per molecule. However, during the literature survey, it was not possible to acquire work or analysis on edible oil like one from African pear; hence work is to be done in this respect.

I. IODINE VALUE: This is the weight of iodine absorbed by 100 parts by weight of sample. Glycerides of the unsaturated fatty acid present

(particularly the oleic series) unite with a definite amount of halogen and the iodine value is therefore a measure of degree of un-saturation.

Iodine value = 260x÷ 45

X is the average volume of the Hubi's iodine used

II. SAPONIFICATION VALUE: This is the number of milligram of potassium hydroxide necessary for saponifying one gram of the oil. it is related to the average molecular weight(mw) of the fatty acids present by the expression. The process of the saponification is the hydrolysis of triacylglycerol into glycerol and the potassium salt of the fatty acids, using a solution of (KOH) in alcohol. Oils with low molecular weight fatty acid have a high saponification value and vice versa. For example, butter with its unusually high percentage of butyric acid has high saponification value (Nielson 2002).

$$\text{Saponification value} = \frac{56.1N\ (V_o - V_1)}{M}$$

III. ACID VALUE: This is defined as the number of milligram of potassium hydroxide required to neutralize the free acid in 1g of the sample. The result is given as percentage of free fatty acid (FFA).

Acid value $=56.1N(v_o - v_1)/m$

IV. PEROXIDE VALUE:This is the measure of the peroxide contained in the oil. This depends on the reaction of potassium iodide in acid solution with

the bond oxygen followed by titration of the liberated iodine with sodium thiosulphate(McGraw-Hill, 1987).

$$\text{Peroxide value} = \frac{1000\ (V_1 - V_2) \times N}{\text{Weight of sample}}$$

V. UNSAPONIFICATION VALUE: These are those substances that are not soluble in water after saponification of fatty bodies, they are classed under the general term unsaponifiable matter.

VI. MOISTURE CONTENT OF OIL:This is the amount of percentage of water present in the oil. It is always necessary to determine the moisture content of oil, normally for ordinary edible oil or fat, it is between the range of 0.05% and 1%. (Codd L.W, 1984).

VII. REFRACTIVE INDEX: It is a physical property of oil which involves measuring the angle via which a beam of light is bent when passing through the oil or fat medium. It is normally done using refractometer. Generally, the more viscous a quantity of oil is, the more the angle of deviation of the oil.

VIII. RELATIVE DENSITY: The density of oil is the mass of unit volume of the oil. The relative density is the ratio of the weight of a given volume of the substance to the weight of an equal volume of water at the same temperature (0^oC).The density of the substance at a given temperature is equal to the Relative density of water at that temperature. Density or specific is measured using density bottle as hydrometer.

IX. BOILING POINT: This is the temperature at which the oil boils. It helps in determining the quality of the oil.

X. COLOR, SOLUBILITY, AND VISCOSITY: The color of oils and fats is important in judging quality and determining the degree of bleaching. The solubility of fats and oils plays a part in determining the immiscibility curve of oil or fat in various solvents. These curves may be used for checking purity (Robert, 1990).The viscosity of fatty oils is of very little significance for their analysis and classification. In general, it increases slightly with increase in the average molecular weight and in the degree of un-saturation of the fatty acids. The actual differences are however very small (Robert 1990).

XI. SPECIFIC GRAVITY: For most fats and oil, this lies between 0.90 and 0.94 at 20℃. In general; it increases with increase in degree of unsaturation and decrease in the mean molecular weight of the fatty acids. It is thus expressed in the following empirical equation.

The table below shows the properties of vegetable oil from literature value.

TABLE 2.1: Some chemical property of vegetable oil.

Oil	**Relative density**	**Refractive index**	**Saponification value**	**Iodine value**	**Unsaponified value**
Cotton seed	0.918 – 0.93	1.46-1.47	189 - 196	99 - 119	≤ 15g /g
Maize	0.917 - 0.93	1.458 -1.47	187 - 195	103 -128	≤ 28g/g
Soya bean	0.919 -0. 93	1.466 – 1.47	189 - 195	120 - 143	≤15g/g
Groundnut	0 .914 – 0.92	1.460 – 1.47	187 - 196	80 - 106	≤ 10g/g
Palm kernel	0.894 - 0.91	1.449 – 1.45	245 - 255	14 - 20	≤108g/g
Palm oil	0.891 – 0.90	1.447 – 1.46	195 - 205	45 - 58	≤10g/g
Coconut oil	0.915 – 0.92	1.448 – 1.45	250 - 264	7 – 10.5	≤6g/g
Cashew nut oil	0.910 – 1.00	1.466 –1.47	———	96 - 106	———

Source ;(Standard organization of Nigeria from vegetable oil production journal).

2.4 AFRICAN PEAR

HISTORY: African pear is an indigenous fruit in the Gulf of Guinea and central African countries but due to popularity of the nutritious fruit for consumption, the plant is widely cultivated, extending its area of distribution to sierra-Leone, Uganda, Angola, Zimbabwe, and Nigeria. It rarely grows wild, thus the natural area of distribution is obscure (Verheji, 2002). The common names are in English, African pear, African pear tree, Bush butter, Bush fruit tree, Native pear and in French it is called “safoutier” (Burkill, 1985).

2.4.1FEATURES OF THE AFRICAN PEAR

It is dioecious shade loving specie of non flooded forest in the humid tropical zone.

a) THE TREE: It is a medium sized evergreen tree reaching a height of 18-40m in the forest but not more than 12m in the plantations (Hutchinson and Daizel, 1958). It is generally, low branching with deep dense crown. The hole is 50-70cm in diameter, short fluted and shallow and more or less sinuous. The trees grow between January and April and bears fruit between May and October.

b) THE BARK: The bark is yellowish grey to pale grey, often rough with lenticels and horizontal folds exuding white aromatic resin.

c) LEAVES AND FLOWERS: The leaves are compound/glossy in pari-pinnate with 5-8pairs of leaflets, they are glossy and pubescence disappearing with age. The flowers are yellow and about 5mm across. They are arranged in a large inflorescence.

d) THE FRUIT: The fruit is on-seeded with pulpy perical about 5mm thick and thin cartilaginous endocarp, Seeds are oblong ellipsoid up to 5.5cm long, whereas the flesh is pale to light green (FAO, 1982).This is the most important to which the tree is widely cultivated, domesticated, and commercialized. (Leakey 1999).

2.4.2 TAXONOMY OF THE AFRICAN PEAR PLANT

Below is a table showing the classification of African pear botanically known as DACRYODES EDULIS.

CLASSIFICATION OF DACRYODES EDULIS: This involves;

KINGDOM	Plantae.
PHYLUM	Angiosperms.
ORDER	Sapindales.
FAMILY	Burseraceae.
GENUS	Dacryodes.
SPECIES	Edulis.
COMMON NAMES	Bush butter tree, African pear.

The generic name “DACYODES” was derived from the Greek word “DAKRUON” meaning “TEAR” referring to resin droplets on the bark surface of its member, while “Edulis means Edible, emphasizing the importance of the nutrients fruits in the plant’s cultivation.

Two varieties are recognized:

- Var- edulis
- Var –parvicarpa.

Varedulis exhibits verticulate or sub verticulate branching while the branching is slender.Varpanicarpa has a smaller conical fruit with the pulp.

2.4.3 CHARACTERISTIC AND COMPOSITION OF THE AFRICAN PEAR African pear usually eaten raw as desert, boiled in water, roasted in hot ash or grilled in oven. The mesocarp softens to form a kind of butter which is eaten in conjunction with boiled or roasted corn.

The high oil concentration of African pear (Dacryodesedulis) which can reach up to 25.2% and 40% respectively on wet basis is its main feature, and on the variety orchard location and harvesting time. (Manson, 1981;Olacte et al, 1986). As reported by Buisson 1965, Burkill 1985, Omoti and okiyi 1987), African pear oil comprises the following fatty acids: Oleic acid, Lineoleic acid, Palmatic and stearic acid with the following composition listed in the table below:

PARAMETERS	PERCENTAGES (%)
Oleic acid.	18 - 60%
Lineoleic acid	15 - 24 %
Palmatic acid.	30 - 62 %
Stearic acid.	1.3 - 5.5 %

From the fatty acid profile shown, a saturated fatty acid content of 50.85% and unsaturated fatty acid content of 49.14% (Lam et al 1986).

African pear also contains some unsaponifiables, like: Protein, Fibre, Carbohydrate, Fats and oil and Moisture. As reported by lam et. al, the mesocarp was found consisting of the following parameters:

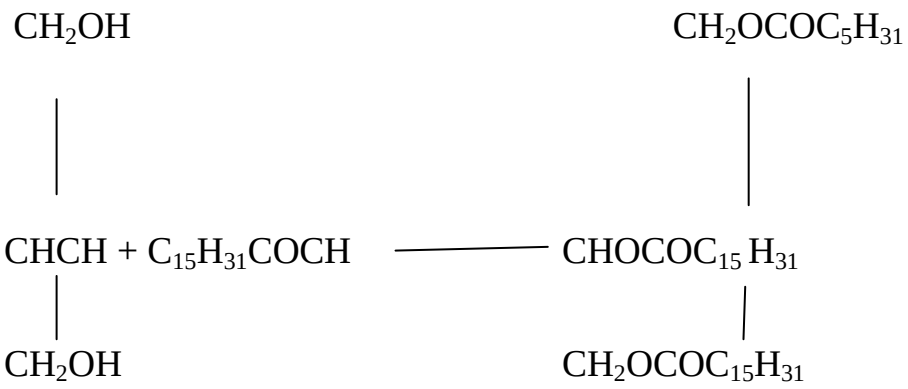
Parameters	**%**
Moisture	36.50- 53.82%
Crude protein	11.09 – 19.19%
Fats	18.81 – 38.36%
Fibre	17.9%
Carbohydrate	13.5%

Courtesy;Lam et al, (1986).

2.5.0 OIL FROM AFRICAN PEAR

Chemically, the constituent of oils and fats are fundamentally glycerides esters of fatty acids. Each fat or oil constitute of glycerides derived from numerous carboxylic acids that varies from one source of oil to another.

The molecular formula of glycerides is given by the product of the reaction shown below:



Sourced from (Codd L.W, 1984).

The African pear oil contains 18-60% of oleic acid, and 15-24% of Lineoleic acid and 30-62% of palmtic acid as reported by (Busson 1965, Burkill 1985, Omoti and Okiyi 1987).

African pear oil is extracted from African pear like other vegetable oils, African pear is an ideal diet as contains bioactive compounds comprising saponin, Alkaloids, Tannins, Flavonoids, and phenol compounds. It also contains bioactive constituents with anti-oxidant activity that may have some medicinal

properties(Koudou, et al 2008). All the fruits contain 8.82 and 12.66% moisture, 30.18 and 85.64% carbohydrate, and 0.40% fats (Downtown et. al, 1975).

In this project, further research was carried out on this same study of proximate analysis to ascertain the transparency and accuracy of data in this quest of knowledge.

2.5.1 USES OF AFRICAN PEAR.

1. An article appearing in the October 1996 issue of "Bioresource technology" investigated the uses of oil extracted from the African pear by a process known as soxhlet extraction named for its inventor, the process is used to isolate lipids or fats from raw materials, therefore making it possible/suitable for the preparation of raw materials. As a vegetable oil, African pear oil can be used for Polish, Paints, Wood varnish, Skin cream and Resin (Burkil 1985).
2. IT IS AN ALTERNATIVE SOURCE OF DIESEL OIL: African pear oil derived from seeds has been investigated as a potential alternative to diesel fuel, to reduce the effects of air pollution. The January 2000 issue of "Energy and fuels" documented the properties of African pear seed oil and compared them to the energy content of diesel fuel. It was observed that the two had similar properties and the pear seed oil could be used as an alternative fuel source (Ekong and okogun, 1969).

3. IT IS USED FOR MEDICINAL PURPOSES.

DacyodesEdulis is a versatile plant in African ethnomedicine as its various partare employed to treat several diseases (Walker and Silans 1961). i) African pear oil contains power antioxidant which may be helpful in gastric ulcer prevention, blood lipid, and cholesterol lowering potential and bioactive compounds such as saponins, tannins, flavonoids, alkaloids etc. which is used for traditional medicine to cure ringworm, wound, scabies, skin diseases and inflammation (Okwu and Nnamdi, 2008).

ii) The oil contains monoheptulose which is a special type of sugar which depress the secretion of insulin(Daquet, 2000).

iii) The oil contains Beta-sit sterols which is reported to reduce the uptake of both testosterone and DHT (DiHydrotesterone) and also inhibits the conversion reaction (Daquet, 2000).

2.5.2 ECONOMIC VALUES OF AFRICAN PEAR

DacryodesEdulis is a versatile plant as its various parts serves many purposes ;The tree wood is heavy, elastic and is found suitable to make axe (Dalziel 1973; Walker and Silans 1961).

-The branches of the tree are used as firewood (Ayuk et al, 1999).

-The twig serves as chewing sticks (Ajibesin, 2005)

-The resin is use as pitch on the inner surface of Calabashes and mending earthen ware (Burkil 1985). It can also serve as fuel (Ekong and Okogun, 1969).

The fruit for consumption

The fruit is roasted or cooked raw in boiling water, which is eaten with corn. It is rich in lipids, proteins, minerals and vitamins which make it an excellent source of nutrition to consumers stimulating its increase in production and commercialization decades (Silou, 1996; Kenmgne et al. 1997): the fruit yields oil found suitable for food.

Safou fruit oil when incorporated into foods can boost their nutritional value thus making them more marketable. So highly traded are the safou fruits that transactions now cut across local and international boundaries, the fruits are marked in specialized markets in Europe (Awono et al 2002). The farm- level value of fruit production may reach USD 161 a year per grower/collector (Ayuk et al 1999). Whereas, the other medicinal parts of the plant such as leaves, stems and roots are sold in the herb section or domestic markets, the fruit are ubiquitous in every section of the market. The average price of the fruit in the markets in nigeria where home consumption accounts for about 70% ranges from USD 300-700/ton of fruits. Between Jan-June 1995 almost 600 tons of fruit valued at USD 224,000 were traded in the humid low lands of Cameroun (Verheji 2002). However, the marketed volume of safou fruit increased to 2,324 tons at a value of USD 1.5 million in 1999, when nine markets were surveyed in

different parts. (Awono et al, 2002). The trade is so active in Cameroun that it is extended to countries such as Gabon.

The major snag of safou international trade, however is the perishability of the fruit. Inspite of this, DacryodesEdulis has become the main source of food cash Incomes, employment and enhanced live hood for subsistence farmers and traders.

CHAPTER THREE

3.0 CHARACTERISATION OF DACRYODES EDULIS

3.1 EXPERIMENT PROCEDURES

i. Collection of the fruits

ii. Dehulling

iii. Drying

iv. Characterisation of the sample proximate analysis

v. Size reduction to various size ranges

vi. Extraction of the oil

vii. Characterisation of the oil (physical and chemical analysis).

3.1.1 Material/Equipment

1) Weighing balance
2) Electric blender
3) Knife
4) Hand gloves
5) Round bottom flask
6) Beaker
7) Conical flask (250ml) & (500ml)
8) Measuring cylinder
9) Filter paper

10) Glass funnel
11) Cellotape
12) Pipette
13) Burette
14) Density bottle
15) Stop watch
16) Thermometer
17) Burnsen burner
18) Platinum crucible
19) Distillation apparatus
20) Reflux condenser
21) Electric heater
22) Refractometer
23) PH electrode/PH paper
24) Water bathe
25) Petri – dish
26) Oven
27) Clamp stand
28) Dessicator
29) Hot – plate
30) Kjedahyl flask
31) Refrigerator

32) Zinc metal

3.1.2 Reagents

1) Normal hexane
2) Distilled water
3) Standard 0.1m NaOH
4) H_2SO_4
5) Boric acid
6) Methyl/red indicator
7) Phenolphthalein indicator
8) Standard 0.5m koH
9) Ethanol
10) Ether
11) Oxalic acid (O.IN)
12) Chloroform
13) Hubi’s Iodine
14) Starch indicator
15) Acetic and
16) Ethanolic petroleum

3.2 PRE-TREATMENT PROCESSES

In the extraction of oil from African pear preparation were made before the extraction of the oil. This preparation is the pre-treatment process which involves:

- Collection of the fruit
- Dehulling of the fruit
- Drying.

(i) COLLECTION OF THE FRUIT

The fruit consisting of the pulp and seed were collected from one Emene-market in Emene, Enugu state.

(ii) DEHULLING OF THE FRUIT

Dehulling was carried out using a sharp knife, the fruit is carefully cut into two making it easy to pluck out the stone seeds within. After this, the sample was cut into different sizes and weighed, then readings were obtained.

(iii) DRYING AND SIZE REDUCTION

The sample was oven dried at the temperature of 100^{o}c before grinding so as to reduce the moisture content, thereby enhancing extraction and distillation process. During this oven drying, appropriate care was taken to prevent the oil from attaining its melting point. After this, the dried pulp was grounded using hammer-mill. During grinding, care was taken not to grind to very fine particles

as this will prevent free flow of solvent during extraction which could lead to reduction in the yield of the oil.

3.3 EXTRACTION OF THE OIL

The oil was extracted by solvent extraction method using n-hexane. This process is a solid-liquid extraction.

PROCEDURE

The pounded sample was dried into five different portion and was poured into round bottom flask, 100ml of the solvent (n-hexane) was measured and transferred also into the round bottom flask and was covered with a metal foil which was sealed with a cellotape to avoid the evaporation of the solvent. The sample was soaked for 24hrs(1440mins).

EXTRACTION.

About 225g ground sample was measured using a weighing balance and transferred into five different conical flask of 50g each, also solvent quantity of 100ml was measured using a measuring cylinder and transferred into each of the five conical flask containing the ground sample. The conical flask was well shaking and sealed with a metal foil and a cellotape to avoid the vapourising of

the solvent. The five flask were then allowed to stay for 24hrs or equilibrium time. After this time, the mixture in each of the five conical flasks were filtered using a filter paper inserted on a glass funnel into a beaker. The miscellawere then heated to evaporate the n-hexane leaving the oil behind.

3.4 CHARACTERIZATION OF THE OIL

3.4.1 TO DETERMINE THE PERCENTAGE YIELD OF THE OIL

This determines the oil content which predicts the profitability of the plant.

Material weighing balance

Procedure: - weigh the sample and take reading.

- Weigh the extract (oil extracted from the sample) and take reading.

The percentage yield in determined by dividing the weight of the oil with the weight of the sample multiplied by 100

i.e. $\frac{weight of oil}{weight of sample} x \frac{100}{1}$

3.5 Proximate Analysis OfDacryodesEdulis

In this analysis are determine the % constituents content of the moisture content, Ash content, fat content, Crude fibre and protein contents.

EXPERIMENT 1

3.5.1 To Determine The Moisture Content Of the Sample

The moisture content is the loss of weight of the soil when heated at a specific temperature.

Materials: Petri-dish, oven, stop watch, weighing balance etc.

Procedure:

- Weigh 1g of sample into a Petri-dish
- Weigh the Petri-dish + sample before drying and note the weight
- Put in the oven and dry for 1hr, note the weight, then oven-dry for another 30mins.
- Continue drying until a constant weight is achieved.

Set the oven at least at 100^0C

Calculation

$$\% \text{ moisture content loss} = \frac{W_1 - W_2}{1\text{g}} \times \frac{100}{1}$$

- $W_1 - W_2 =$ moisture content

- Weight of crucible = 23.25g
- Weight of crucible + sample before drying (W_1) = 24.20g

	Heating	**weight**
1.	10mins	24.15g
2.	20 mins	24.10g
3.	30 mins	23.96g
4.	40 mins	23.92g
5.	50 mins	23.91g
6.	60 mins	23.91g => W_2

EXPERIMENT II

3.5.2 To Determine The Ash Content Of The Sample

The ash content is the percentage of inorganic residue remaining after ignition of the fitered and non-filtered oil.

Materials: platinum crucible, Bunsen burner, weighing balance

Procedure: =>

- Wash empty P.crucible, dry and note the weight.
- Weigh 1g of the sample in the platinum crucible and place in the Bunsen burner and burn for 1hr.
- Heat until it turns to ash

- Cool the sample after burning and weigh

Calculation

- Weigh the empty P. C + sample before drying (W_1)
- Weight of P.C + sample after drying (W_2)

$$\%\ ashcontent\ = \frac{W_1 - W_2}{1g} \times \frac{100}{1}$$

EXPERIMENT III

3.5.3 To Determine The Fat Content Of The Sample (Lipid – Fat & Oil)

Extraction of fat using soxhlet extraction method. The soxhletextractor consists of reflux condenser, hot plate and n – hexane as the solvent.

Materials used: Soxhlet extractor, filter paper, electric heater, conical flask, beaker, desiccators weighing balance.

Procedure:

- Weigh 5g of sample and wrap very well in a filter as put in soxhlet extractor.
- Apply a heating mantle below a conical flask with n-hexane evaporates, condenses and goes back into the conical flask.
- The system in recycled 8 – 9 times to achieve maximum yield of oil.

- After the recycling, the extractor is disconnected and a distillation apparatus is set up to separate the solvent (n-hexane) from the oil. This is done so that the solvent can be recovered.
- An empty beaker was weighed and the sample containing oil and traces of the solvent after distillation was transferred into the weighed beaker and heated so that the remaining hexane escapes leaving only the oil.
- Allow to cool in a dedicator and weigh the beaker again.

Calculation

Weight of beaker

Weight of beaker + oil

Weight of oil

$$\%\, fatcontent = \frac{\text{Wt of oil}}{\text{weight of sample}} \times \frac{100}{1}$$

EXPERIMENT IV

3.5.4 To Determine The Crude Fibre Of The Sample

Materials used: 200ml conical flask, hot plate, PH paper, filter paper, Bunsen burner, dessicator, weighing balance and platinum crucible.

Procedure:

- Weight of sample = weight of the sample into a 200ml conical flaks
- Soak in 200ml of 1.25% H_2SO_4. i.e. 1.25% in 100ml of H_2O (increased 7.5g of pellets to 600ml of H_2O).
- Heat for 30mins on a hot plate, this is called acid treatment.
- Filter and wash the residue with hot H_2O until it is no more acidic using PH paper.
- Re-soak the residue with 200ml of 1.25% NaOH
- Heat again for another 30mins
- Filter in a noted weight of filter paper and dry in an oven and weigh again.
- Weigh an empty platinum crucible
- Transfer the paper containing the residue into the weighed P.C.
- Burn to ash using a Bunsen burner.
- After ashing, cool in desiccators and weigh again.

EXPERIMENT V.

3.5.4 TO DETRMINE THE CRUDE PROTEIN OF THE SAMPLEW

Reagents used:

Na_2SO_4, H_2SO_4, $CuSO_4$, distilled water, NaOH, boric acid and methyl red indicator.

Procedure:

1. Weigh 0.5g of the sample in a Kjedahyl flask
2. Add 10g of Na_2SO_4 or K_2SO_4
3. Add about 1g of $CuSO_4$
4. Heat with heating mantle till solution digest completely (changes to bluish green)
5. Allow to solidify for 24hrs. (colour turns to white)

Observation:

1. After costing for 24hrs, the digested sample solidified and whitish colour was got
2. Add 200ml of distilled water to dissolve the solidified sample and allow to cool in a refrigerator.
3. Add 60ml of 40% NaOH to the digested sample and 2 piece of Zinc metal. Connect the mixture to a distillation column.
4. In the set up, add 10ml of 4% boric acid to a conical flask and add 2 drops of screened methyl red indicator.
5. A tight colour appears when boric acid and screened methyl red indicator come in contact.
6. When the whole liquid in the receiver reaches 200ml, the distillation is stopped by dismantling the distillation apparatus.
7. Titrate the absorber (200ml) with 0.1m H_2SO_4.

N/B: Titration is stopped when the colour of the distillate comes to the initial colour of the mixture of boric acid screened by methyl red inidicator pink.

Calculation

$$\frac{100 \times Tv \times 0.0014 \times 6.25}{\text{weight of sample}}$$

Where

0.0014 = A constant – 0.0014 is liberated by 1ml of 0.1N H_2SO_4.

6.25 = protein constant according to Kjedahyl method.

(Tv = Titre value)

Titre value = 6.80

3.5.5 TO DETERMINE THE CARBOHYDRATE CONTENT.

The percentage carbohydrate content of the oil was obtained as the difference from 100 and the % values of the moisture content, protein, crude fibre, and crude protein.

3.6 CHEMICAL ANALYSIS OF THE OIL

EXPERIMENT I:

This involves the determination of the constituents: Acid value, iodine value, peroxide value and saponification value.

3.6.1 Determination Of Acid Value Of The African Pear Oil

The acid value is defined as the number of milligram of KOH (Potassium Hydroxide) required to neutralise the free fatty acid present in one gram of oil or fat.

Reagent

(1) KOH

(2) Fat solvent (Ethanol and ether in 1 : 1 ratio)

(3) Phenolphthalein

(4) Oxalic acid (0.1N)

Equipments:

Beaker conical flask, brush, water bottle, measuring cylinder, clamp stand.

Procedure:

Titration I

(Titration for blank)

Pipette out 20ml of lipid solvent into a clean conical flask, add 2 drops of phenolphthalein indicator and titrate it against KOH taken in the burette. The end point is the appearance of permanent pale pink colour. Repeat the titration for concordant values.

Titration II

For oil

- Weigh out 1g of the oil and transfer into a clean conical flask
- Add 20ml of the lipid solvent to dissolve the oil and shake well.
- Add a few drops of phenolphthalein indicator and titrate against standardized KOH taken in the burette. End point is the appearance of permanent pale pink colour.

$$\text{Acid value} = \frac{\text{strength of KOH} \times \text{Equivalent weigth of KOH} \times 100}{\text{weigth of oil } 1000}$$

EXPERIMENT II

3.6.2 Determination Of The Iodine Value Of African Pear Oil

The iodine value equals the number of mg of iodine required to saturate the fatty acid present in 100mg of oil or fat oils which in saturated fatty acids have low iodine numbers.

In this experiment, Hubi's iodine is added gradually to a fixed volume of the oil dissolved in chloroform as long as double bounds are available, the colour of iodine does not appear in the solution as the iodine absorbed by the double bounds. When all the double bonds are saturated, the colour of iodine appears in the solution.

Materials required:

(a) Chloroform

(b) Oil

(c) Hubi's iodine (dissolve separately)

(d) A 26g of iodine and 30g mercuric chloride in about 250ml of ethanol each, mix the two and make to 1 litre with ethanol.

Equipments:

Burette, clamp stand, funnel, porcelain dish and measuring cylinder.

Experimental Procedures

The Hubi's iodine is filled in a 50ml burette and the initial reading noted.

- Five (5ml) of chloroform is taken in a dry porcelain dish and 2-3 drops of Hubi's iodine after adding, then serves as a control for colour composition. 5ml of chloroform is taken another dry porcelain dish; 0.5ml of oil is added and dissolved by gentle swirling. Hubi's iodine is added slowly from the burette until the colour of iodine matching with the control appears in the solution. The burette reading is noted.
- The experiment is repeated.The specific gravity of the oil is taken as 0.9 to 0.5ml if the oil weighs 450mg.

  Suppose that xml of Hubi's Iodine is used to saturate 0.5ml of the oil, 1ml of Hubi's Iodine, contains 26mg of Iodine. Since 450mg of the oil xml of

Hubi's Iodine contains 96xmg of Iodine, since 450mg of the oil takes up 26xmg of iodine of the oil will take up 26 x 45mg of iodine and 100mg of the oil will take up 260 x 450mg of iodine. Hence, the iodine value of the oil is determined.

Since 0.5ml of pear oil weighs 45mg

1ml of Hubi's Iodine contains 26mg of iodine

Xml of Hubi's Iodine contains 26xmg of Iodine

46mg of oil takes up 26xmg of iodine

1mg of the will take up

$$\frac{26x}{46mg of Iodine}$$

Experiment IV

3.6.3: Determination of the peroxide value of African pear oil

Peroxide value is the measure of the peroxide contained in the oil. This depends on the reaction of potassium iodine as acid solution with the bond oxygen followed by the titration of the liberated iodine with thiosulphate.

Reagents

1) Acetic acid
2) Chloroform

3) Distilled water

4) Starch indicator.

Procedure:

- Measure 0.5g of sample
- Add 25ml of acetic acid and chloroform in the ratio 2:1
- Shake rigorously
- Cover and keep in the dark for I min
- Add 35ml of distilled water + drop of iodine solution
- Add 5ml of starch indicator

  N/B: The colour changes to purple on the addition of starch indicator.
- Titrate with 0.02N Sodium – thiosulphate

Observation

- Colour changes to white

Calculation

$$\frac{1000(V_1 - V_2) \times N}{weight of sample}$$

EXPERIMENT V

3.6.4 Determination Of Saponification Value Of The Oil

Saponification value is the number of milligrams of potassium hydroxide required to completely hydrolyse or saponify 1gram of oil.

Materials: Conical flask, Reflux condenser, Electric heater.

Procedure: Indicator method was used as specific by 1503657 (1988).

- 0.5g of the sample was weighed into a conical flask.
- 50ml of 0.5N ethanolic petroleum hydroxide was then added.
- The content which was constantly thrived was allowed to boil gently for 30mins.
- A reflux condenser was placed on the flask containing the mixture
- Few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5m HCl to the end point. Until the pink colour of the indicator just disappeared. The same procedure was used for other samples and blank.

  The expression for saponification value (S.V) is given by:

$$SV = 56.1N\frac{(V_0 - V_1)}{M}$$

  Where V_0 = The volume of the solution used for blank test.

  V_1 = The volume of the solution used for determination.

N = actual normality of the HCl used.

M = mass of the sample.

3.7 PHYSICAL ANALYSIS OF THE OIL

This is the determination of the refractive index, specific gravity, PH value and boiling point.

EXPERIMENT I

3.7.1 To determine the refractive index of the oil.

This is the measuring of the angle via which a beam of light is bent when passing through the oil or fat medium.

Materials used: Refractometer

Procedure:

- Few drops of the oil was placed on the refractometer
- Reading were taken and recorded

Experiment II

3.7.2 To determine the specific gravity of African pear oil.

This is the ratio of the weight of a given volume of the oil to weight of an equal volume of water at the same temperature.

Procedure

Density bottle was used in determining the density of the oil. A clean and dry bottle of 25ml capacity was weighed (W_0) and then filled with the oil, stopper inserted and reweighed to give (WL). The oil was substituted with water after washing and drying the bottle and weighed to give (W_2). The expression for specific gravity is Sp.gr =($W_1 – W_0$)/($W_2 – W_0$) = mass of the substance / mass of an equal volume of water.

Formula = $\frac{W_3 - W_1}{W_2 - W_1}$

EXPERIMENT III

3.7.3 Determination of the ph value of African pear oil

Materials used: Beaker Hot distilled water, water bath, PH electrode.

Procedure:

2g of the sample was poured into a clean dry 25ml beaker and 13ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was

then cooled in a cold water bath to 25^0C. The PH electrode was standardised with buffer solution and the electrode immersed into the sample and the PH value was read and dried.

PH of pear oil = 5.695 ≅5.7

CHAPTER FOUR

4.0 EXPERIMENTAL RESULTS AND DISCUSSION

In the course of this research work, analysis were carried out and results were obtained for the African pear and its oil and recorded accordingly

4.1 TABLES AND RESULTS

Table 4.I (Percentage yield of the oil of DacryodesEdulis)

Weight of the African pear.	225g
Weight of the oil extracted from the sample	116.04g
Percentage yield of the oil	51.6%

TABLE4. 2

A processed table for the results of the proximate analysis of DacryodesEdulis. This fruit crop contains the percentage (%) constituent of the parameters below.

PARAMETER	PERCENTAGE
Moisture content	29%
Ash content	2.0%
Fat content	19.6%
Crude fibre	25.5%
Crude protein	11.9%
Carbohydrate	12.0%

Table 4.3
A processed table for the results of the physic – chemical analysis of the oil of DacryodesEdulis.

PARAMETER	VALUES
Acid value (gm/KOH/gm)	8.41
Iodine value (gm/iodine/gm)	3.96
Saponification value (gm/KOH/gm)	185.1
Peroxide value (gm/KOH/gm)	2.5
Refractive index (brix)	1.469
Specific Gravity	0.92
PH value	5.7

4.2 DISCUSSION

The analysis and discussion of every experimental result is necessary as it gives a clear understanding of the work and possible application of specific results. Determination of oil content in plants is important because it predicts the profitability of given plants as potential source of oil. High oil content in plant seed/fruit implies that processing it for oil will be economical (Ikhuoria et. al, 2007). The oil extracted were liquid at room temperature, this means that they could be classified as oil. The oil was light brown in colour with 51.6% yield which is high but does not favourably compare with that reported in other literature,this may be due to ecological factors affecting seeds/fruits in different geographical locations, or differences in laboratory practice. Most popular plant oils have specific gravity ranging from – 0.910 – 0.940, pretty good number for any cooking oil (Minxangi et. al, 2011). Specific gravity of the oil was determined to be 0.9. Acid number on the other hand indicates the age and quality of the oil.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

DacryodesEdulis is a tree widely cultivated for its fruit. The edible fruit bears high oil content and is a rich source of nutrients as deduced from the Phyto-Chemical properties. From this research work carried out, a reasonable amount of oil was extracted from African pear. The oil from an African pear in an ideal diet. It is more advisable to consume this type of vegetable oil that is low in saturation than the oil from animal product, as it contains no cholesterol which can lead to cardiovascular heart-disease. This study also shows that the African pear can be used as an alternative-substitute base for fats & oil. The Physico-Chemical characteristic composition shows that they have some industrial potentials and utilization of these oil will reduce our dependence on the popular vegetable oils like groundnut, coconut and palm oil for domestic use. The study also shows that solvent extraction method yields more oil.

5.2 RECOMMENDATION

For the fore-going conclusion,vegetable oil is more advisable to be consumed because of its low saturation.In the production of this African pear oil from the fruit seed, this method of extraction process should be adopted in oil extraction for more profit.

Further research should be based on other components of the oil, the fruit skin and the unsaponifiable components in the fruit seed as well as the non-lipid components of the oil.

I also recommend the government investors and other business specialists to invest some of their money into the extraction and analysis of vegetable oil from seed fruits. This project is economical, since it help to provide adequate supply of vegetable oil in the market.

Finally, Nigeria economy will be more suitable, since the companies which engage in extraction and analysis of vegetable oil from seed fruit provide employment to our graduating students.

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APPENDIX I

Percentage yield of the oil (%)

Weight of the sample (plant material) =225g

Weight of the extract (oil extract from the sample) =116.04g

% yield = $\frac{weight\ of\ oil}{weight\ of\ sample} \times \frac{100}{1}$

= $\frac{116.04}{225} \times \frac{100}{1}$

=51.57% ≅ 51.6%

APPENDIX II

i) Moisture content

% moisture content = $\frac{w_1 - w_2}{1g}$ X $\frac{100}{1}$

While $w_1 - w_2$ = Moisture content

W_1=24.20g

W_2=23.91g

Moisture content = 24.20 – 23.91= 0.29g

% moisture content

= $\frac{0.29}{1}$ X $\frac{100}{1}$

=29%

ii) Ash content

% Ash content = $\frac{w_1 - w_2}{1g}$ X $\frac{100}{1}$

Ash content = $w_2 - w_1$

W_2 = 7.99

W_1 = 8.01

Ash content = 8.01 – 7.99 = 0.02

% Ash content = $\frac{0.02}{1}$ X $\frac{100}{1}$

=2%

iii) Fat content

% of oil and fat = $\frac{weight\ of\ oil}{weight\ of\ sample}$ X $\frac{100}{1}$

Weight of sample = 5g

Weight of empty beaker = W_1 = 49.66g

Weight of oil beaker = oil after evaporation (W_2)

Weight of oil alone = $W_2 - W_1$

= 50.64 – 49.66 = 0. 98

% weight of oil and fat

= $\frac{0.98}{5}$ X $\frac{100}{1}$ 19.6%

iv) Crude fibre

% crude fibre = $\frac{weight\ of\ fibre}{weight\ of\ sample}$ X $\frac{100}{1}$

Weight of sample 2g

Weight of filter paper = 1.08g

Weight of residue filter paper after over drying = 1.59g

Weight of residue = 1.59 – 1.08 = 0.51g

Weight of ash = (weight of platinum crucible + ash)-(weight of platinum crucible) = 7.98 – 7.98 = 0

Weight of fibre = weight of residue – weight of Ash

=0.51g – 0 = 0.51g

% crude fibre = $\frac{0.51}{2}$ X $\frac{100}{1}$ = 25.5%

v) Crude protein (protein content)

% protein content = $\frac{1000tv \times 0.0014 \times 6.25}{wt\ of\ sample}$

Where 0.0014 = A constant (0.0014 in liberated by /m/ 000.in H_z504)

6.25 = protein constant according to Kjedahyl method

Titre value = 6.80%

Weight of sample = 0.5g

% protein content $\frac{1000\ X\ 6.80\ X\ 0.0014}{0.5}$ = 11.9%

vi) Carbohydrate content

% carbohydrate = 100-(%protein + %ash + %moisture + %crude fibre + %fat).

= 100-(29 + 2.0 + 19.6 + 25.5 + 11.9)%

= 100-88 = 12.0%

Appendix III

a) Acid value

- Strength of KOH = 0.5ml
- Weight of oil alone = 1g

Initial titre	Final titre	Titre value
10.5ml	10.8ml	0.3ml

- Equivalent weight of KOH = 0.3ml

$$\therefore \text{Acid value} \frac{\text{strenght of KOH} \times \text{Equivalent weight of KOH} \times 1000 \times 56.1}{\text{weight of oil} \times 1000}$$

$$\frac{0.5\text{ml} \times 0.3\text{ml} \times 1000 \times 56.1}{0.1 \times 1000} = \frac{8415}{1000}$$

= 8.41gm/KOH/gm.

b) Iodine value

$$\text{Iodine value} = \frac{260x}{46\text{mg}}$$

usingHubi's Iodine

1mmg of the oil will take up

$$= \frac{26x}{46\text{mg of iodine}}$$

100mg of the oil will take up

$$= \frac{260x}{46}$$

X = 0.7

:. Iodine value =

$$= \frac{260 \times 0.7}{46} = \frac{182}{46}$$

= 3.96gm/iodine/gm.

c) Peroxide value

$$\text{peroxide value} = \frac{2\,X(V_2 - V_1)}{\text{weight sample}}$$

V_1 = Titre value for blank

V_2 = Titre value for sample

1000 = peroxide constant.

Weight of sample = 0.5

:. $V_1 = 0.5$, $V_2 = 1.2$, N = 0.02

$$= \frac{2\,X\,(1.2 - 0.5)}{0.5} = \frac{1.4}{0.5}$$

$= 2.8\text{gm/KOH/gm.}$

d) Saponification value

Saponification value = $56.1N\frac{(V_0-V_1)}{m}$

Where V_0 = the volume of the solution used for be lank test

V_1 = The volume of the solution used for determination

N = Total normality of the HCl used

M = Mass of the sample.

N =0.1, V_0 = 23.00, V_1 = 26.30 and m = 0.5g

$$= \frac{56.1 \times 0.5 \times 3.3}{2g}$$

= 185.1gm/KOH/gm.

Appendix IV

a) Specific gravity

$$\text{specific gravity} = \frac{W_1 - W_0}{W_2 - W_0}$$

where W_1 = weight of specific gravity of bottle = 14.79g

W_2 = weight specific gravity of bottle + water = 70.56g

W_3 = weight of specific gravity of bottle + oil = 66.17g

$$\therefore specific\ gravity = \frac{66.17 - 14.79}{70.56 - 14.79} = \frac{51.38}{55.97}$$

= 0.9213 ≅**0.92.**

b) PH value

PH value = 5.695 ≅**5.7**