Sudan University of Science and Technology College of Graduate Studies

Extraction And Characterization Of The Oil, Biodiesel From

Balanites Aegyptiaca Seeds And Biodiesel Blends With Diesel And

Ethanol

استخلاص وتوصيف الزيت والوقود الحيوي من بذور الهجليج ومخاليط الوقود الحيوي مع الديزل والايثانول

A Thesis Submitted in Fulfillment of the Requirements for a PhD Degree in chemistry

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الاستهلال

بسم الله الرحمن الرحيم

(ان الله فالق الحب والنوي يخرج الحي من الميت ومخرج الميت من الحي ذلكم الله فاني تؤفكون)

سوره الأنعام الايه95

Dedication

TO

My father,

My mother,

wife,

Brothers and Sister.

Acknowledgment

Praise to AllaH who gave me health and patience to pursue this project to completion

Iam mostlyindebted to my supervisors Prof . Mohammed Elmubark Osman also Dr. ElfatihAhmmed Hassan fo r theirgridance and follow up throughout this work.

Thanks are also, due to the National Trees Seeds Center, (Algadarif), petroleum laboratories research and studies for their technical support.

my thanks also extended to, my friends for their moral support

Abstract:

This study was conducted on the oil, Biodiesel and Biodiesel blendsfrom *Balanitesaegyptiaca*seeds, the oil content of seeds was 53.9% w/w.

The physicochemical properties of oil ,the moisture content was (0.27%), the viscosity was (25.87cp), refractive index was (1.475), density of oil was $(0.88kg/m^3)$ at $40\mathbb{C}$, the color index was yellow (2.6) and red (1.1), free fatty acid value was (1.067%), fat content was (54.7%), the Acid value was (2.1mgKOH/g), Peroxide value was (6meq/kg), saponification value was (239.35mgKOH/g) and the iodine value was (74.34 I2/100g oil).

the Biodiesel physiochemical analysis showed that the viscosity was(5.041) cp at 40Cand (1.916) at 100C, the density of Biodiesel was (0.885kg/m³) at 40C, the cloud point of Biodieselwas (13.5), copper corrosion was (1a), flash point was (130)C, color was (0.7), cetanenumber was (54.6), sulfur m/m% was (0.011).

The physicochemical characterization of the Diesel were determined, where, the viscosity was (3.3511) cpat 40**C**, and (1.307) at 100**C**the density of diesel was (0.841kg/m³), the cloud point of diesel was (5.1), copper corrosion was (1a), flash point was (77)**C**, color was (0.5), cetanenumber was (53.9), sulfur m/m% was (0.011).

The physicochemical properties of biodiesel /diesel/ ,blend(20:80)show that viscosity of blends at 40Cwas (3.855) cp, at 100C was (1.702) cp, the density of blend was (0.848kg/m³),the cloud point of blends was (5.6)C, copper corrosion was (1a) ,flash point was (77.5)C, color was (0.7), cetanenumber was (55.2), sulfur m/m% was (0.011).

The physicochemical properties of biodiesel /diesel/ethanol(20:60:20), show that the viscosity was (3.011) cpat 40C, at 100C was (1.53), the density of mixture was (0.845kg/m³), the cloud point of mixture was (15.7)C, copper corrosion was (1a), the flash point was (40.5)C, the color was (0.5), the cetanenumber was (54.6), the sulfur m/m% was (0.011).

The 0il of *Balanitesaegyptiaca* seeds characteristic spectrum in FT-IR the absorption bands 3006.65(C-H Stretching vibration), 2925.35(C-H for CH3),

2856.85(C-H for CH3symmetric), 1745.56(C=O for ester), 1457.26(C-H for CH2), 1162.43(C-C Stretching vibration) and 721.58 (CH2) $_n$

Biodieselsample were analyzed using PerkinElmer GC –MS in total scan mode to identify the fatty acid methyl ester composition.

The components in the sample such as hydrocarbons —ester —phenol — alcohol and others. The fatty acids 80% is unsaturated and the 20% is saturated. The major components are 9,12- Octadecadienoic acid ,Hexadecanoicacid,methyl ester, 9-Octadecenoic acid methyl ester , methyl stearate, Cyclopropaneoctanoic acid and Eicosanoic acid methyl ester constitute about 95%.

المستخلص

أجريت هذه الدراسة على بذور الهجليج التي جمعت من الحواته ولاية القضارف في عام 2016.

تماستخلاص زيت بذور الهجليج عن طريق الاستخلاص بالمذيب وكانت نسبة الاستخلاص 53.9%.

تم اجراء الاختبارات الفيزيوكيمائية علي الزيت المستخلص وكانت الذتائج كالآتي: كانت قيمه الرطوبة. (0.27%) واللزوجة (25.87%) وقيم معامل الانكسار (1.475) والكثافة (0.88kg/m³) at 40°C واللون (1.475) والكثافة (25.87%) واللون (2.60%) والمحتوي (1.067%) والقيمه الحمضية (1.067%) وكمية الأحماض الدهون (54.7%) والقيمه الحمضية (2.1mgKOH/g) وقيمه النسرة (54.7%) وقيمه الأسترة (239.35mgKOH/g oil).

وكانت الصفات الغيزيوكيمائية للوقود الحيوي كالاتي: اللزوجة (5.041 عند 40 درجه مئوية و1.91 عند 100 درجه مئوية), والكثافة (0.885kg/m3) عند15 درجه مئوية), ونقطه التحسب (13.5) ودرجه تأكل شريحه النحاس (1)، نقطه الوميض (130) درجه مئويه ، واللون (0.7) ، ورقم السيتان (54.6) ، ومحتوي الكبريت (m/m0.011).

ونجد ايضاالصفات الفيزيوكيمائية للديزل كالاتي: اللزوجة (3.3511 عند 3.3511 عند 3.3511 عند 3.3511 درجه مئوية), ونقطه التحسب 3.3511 ودرجه تأكل درجه مئوية), ونقطه التحسب 3.3511 ودرجه تأكل شريحه النحاس 3.3511 نقطه الوميض 3.3511 درجه مئويه ، واللون 3.3511 ومحتوي الكبريت 3.3511 الكبريت 3.3511 ومحتوي الكبريت 3.3511

وكانت الخصائص الفيز وكيميائيه للبايوديز ل(20:80):

اللزوجة (3.855عند 40 درجه مئوية و 1.702 عند 100 درجه مئوية، والكثافة (0.848kg/m³ عند15 درجه مئوية), ونقطه التحسب (5.6) ودرجه تأكل شريحه النحاس (1)، نقطه الوميض (77.5) درجه مئويه، واللون (0.7)، ورقم السيتان (0.7)، ومحتوي الكبريت (0.01).

وكانت الخصائص الفيزيوكيميائيه للخليط (الديزل/البايوديزل/الايثانول20:60:20)

اللزوجة (3.011 عند 10 درجه مئوية و (1.053) عند 100 درجه مئوية، والكثافة (3.011 عند 100 درجه مئوية), ونقطه التحسب (3.01) ودرجه تأكل شريحه النحاس (3.01)، نقطه الوميض (3.01)، واللون (3.01)، ورقم السيتان (3.01)، ومحتوي الكبريت (3.01).

تم اخضاع زيت بذور اللالوب الي جهاز الاشعه FT-IR وكانت النتائج:

حزم الامتصاص 3006.65(C-H المترازات الشد 2925.35(C-H for CH3), 2856.85(C-H for CH3), 1745.56(C=O المترازات (اللاستر 745.56(C-H for CH2), 1162.43(C-C المترازات 721.58 (CH2) $_{\rm n}$

وايضا تم عمل GC-MS للبايوديزل للتعرف علي المكونات من الاحماض الدهنية والاسترات والهيدروكاربون والكحول ووجد ان 80% من االاحماض غير مشبعه و 20% مشبعه ومن المكونات المهمة وو2 اوكتاديناويك , هكسادينويك, ميثايل استر , وايكوسانويكميثايل استر , سايكلوبروباناوكاتونيك وايكوسانويكميثايل استر

List Of Abbreviations:

- (SOD) Super oxide dismutase
- (GPx) Glutathione peroxide
- (CAT) Plasma catalase
- (ASTM) The society for testing and materials
- (EPA) Environmental protection agency
- (EGR) The exhaust gas recirculation system
- (DPF) Diesel particulate filter
- (HDPE) High density poly ethylene
- (PVC) Poly vinyl chloride
- (EN) European union
- (JEP) Jatropha evaluation project
- (CFSEF) The center for sustainable energy farming
- (UV) Ultraviolet

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Chapter One Introduction And Literature Review

CHAPTER One

Introduction and Literature Review

1.1 Introduction:

The reserves of petroleum-derived fuels are diminishing with their increasing demand. Moreover, the combustion products that result from burning these fuels are considered harmful to the environment. Several factors such as depletion of petroleum derived fuel, climate change, and increase in the price of petroleum products have generated interest in discovering alternative energy sources among researchers (Palashetal, 2013).

In recent years, many researchers worldwide have searched for new alternative energy sources that are available, technically feasible, economically viable, and environmentally acceptable (Liaquat, 2010). Biodiesel is considered one of the best alternative energy sources because of its

potential to reduce dependency on fossil diesel fuel, capacity to decrease environmental pollutant output, and application in compression ignition (CI) engines without modification (Tan etal, 2012).

Biodiesel is non-explosive, biodegradable, nonflammable, renewable, Nontoxic, environment friendly, and similar to diesel fuel (Mofijuretal, 2013).

The main advantages of biodiesel include the following: it can be blended with diesel fuel.it can be used in CI engines with no modification; it does not contain any harmful substances; and it produces less harmful emissions to the environment than diesel fuel (Shahabuddinetal, 2012).

Biodiesel can be obtained through transesterification of vegetable oils, animal fats, waste cooking oil, and waste restaurants greases (Costaetal, 2013). It originates from edible and nonedible sources. The most common edible oils of biodiesel include palm oil, rapeseed oil, sunflower oil, coconut oil, and peanut oil, whereas the nonedible oil sources of biodiesel are Jatropha, neem, cotton, jojoba, rubber, Moringa, Mahua, castor, and animal tallow (Atabanietal, 2012).

1.2 Broduction of Biodiesel from Balanites aegyptiaca Seed Oils:

The fossil fuel global politics and its high cost have mandated the investigation and production of an environmental friendly, cost effective and renewable fuel source

(biodiesel) which is obtained from animal fats and vegetable oils. The oil has so many attractive features over petro diesel most which are:

Biodiesel is a renewable, biodegradable and environmentally friendly fuel for use in diesel engines. It can overcome the problem associated with fossil fuels such as its non-renewability, polluting nature and its global politics which is a matter of concern to many nations.

The biodiesel has so many attractive features over petro diesel most which are: Biodiesel is biodegradable 'biodiesel improves engine performance 'biodiesel is essentially non-toxic and is not a skin irritant. It has higher cetane number than ordinary diesel and therefore does not need improper additives as in premium petro diesel. It has a higher flash point than ordinary diesel and so is not flammable. It reduces net CO₂ emission by 70% compared with ordinary diesel cutting green house gases that lead to global warming. It is much more lubricating than ordinary diesel reducing engine wear. It is produced from local feedstock, reducing the need for foreign imports, while boosting the local economy and supporting the agricultural community biodiesel replaces the exhaust odour of ordinary diesel with a more pleasant smell of popcorn or French fire, biodiesel blends of 20% with 80% ordinary diesel can be used in unmodified diesel engines. Biodiesel can be used in its pure form but may require certain engine modifications to avoid performance problemsbiodiesel maintenance and requires virtually additives. The advancements of biodiesel quality are being developed globally to maintain the quality of the end product and to ensure better criteria of biodiesel storage and feedstock for consumers' confidence and successful commercialization of biodiesel. Since biodiesel is produced from quite differently scaled plants of varying origins and qualities, it is necessary to install a standardization of fuel quality to guarantee an engine performance without any difficulties (Balat, Balat.2010). Austria was the first country in the world to define and approve the standards for rapeseed oil methyl esters as a diesel fuel. The guidelines for standards and the quality of biodiesel have also been defined in other countries such as in Germany, Italy, France, the Czech Republic and the United States (Meher, VidyaSagar, Naik. 2006). Currently, the properties and qualities of Biodiesel must comply with the international biodiesel standard specifications. These specifications include the American Standards for Testing Materials (ASTM 6751-3) or the European Union (EN 14214) Standards for biodiesel fuel (Atadashi, Aroua, Abdul Aziz. 2008). However, there are some other standards available globally such as in Germany), Austria and Czech republic.

1.2.1 Biodiesel From Algal:

From 1978 to 1996, the U.S. experimented with using algal as a biodiesel source in the "Aquatic Species Program.(Charles and Guy,1979). A self-published article by Michael Briggs, from the biodiesel Group, offer estimates for realistic replacement of all vehicular fuel with biodiesel by utilizing algae that have a natural oil content greater than 50%, which Briggs suggests can be grown on algae ponds at wastewater treatment plants.(Donkor,1986). This oil-rich algae can then be extracted from the system and processed into biodiesel, with the dried remainder further reprocessed to create ethanol.(Haroldetal,1990).

The production of algae to harvest oil for biodiesel has not yet been undertaken on a commercial scale, but feasibility studies have been conducted to arrive at the above yield estimate. In addition to its projected high yield, algaculture unlike crop-based biofuels — does not entail a decrease in food production, since it requires neither farmland nor fresh water. Many companies are pursuing algae bioreactors for various purposes, including scaling up Biodieselproduction to commercial levels.(Nasco,1994).

Prof. Rodrigo E. Teixeira from the University of Alabama in Huntsville demonstrated the extraction of biodiesel lipids from wet algae using a simple and economical reaction in ionic liquids. (Nasco, 1994).

1.2.2 Biodiesel FromBongamia:

Millettiapinnata, also known as the *Pongam* Oil tree or *Pongamia*, is a *leguminous*, oilseed-bearing tree that has been identified as a candidate for non-edible vegetable oil production.

Pongamia plantations for biodiesel production have a two-fold environmental benefit. The trees both store carbon and produce fuel oil. *Pongamia* grows on marginal land not fit for food crops and does not require fertilizers. The oil producing tree has the highest yield of oil producing plant (approximately 40% by weight of the seed is oil) while growing in malnourished soils with high levels of salt. It is becoming a main focus in a number of biodiesel research organizations. The main advantages of Pongamia are a higher recovery and quality of oil than other crops and no direct competition with food crops. However, growth on marginal land can lead to lower oil yields which could cause competition with food crops for better soil.(Nielen, 1994.)

1.2.3 Biodiesel From Jatropha:

Several groups in various sectors are conducting research on *Jatropha*curcas, a poisonous shrub-like tree that produces seeds considered by many to be a viable source of biodiesel feedstock oil. Much of this research focuses on improving the overall per acre oil yield of Jatropha through advancements in genetics, soil science, and horticultural practices. (Pomeranz and Medean, 1987).

SG Biofuels, a San Diego-based *Jatropha* developer, has used molecular breeding and biotechnology to produce elite hybrid seeds of *Jatropha* that show significant yield improvements over first generation varieties. SG Biofuels also claims that additional benefits have arisen from such strains, including improved flowering synchronicity, higher resistance to pests and disease, and increased cold weather tolerance.(Poucher ,1984).

Plant Research International, a department of the Wageningen University and Research Centre in the Netherlands, maintains an ongoing Jatropha Evaluation Project (JEP) that examines the feasibility of large scale Jatropha cultivation through field and laboratory experiments.(Rossel ,1971).

The Center for Sustainable Energy Farming (CFSEF) is a Los Angeles-based non-profit research organization dedicated to Jatropha research in the areas of plant science, agronomy, and horticulture. Successful exploration of these disciplines is projected to increase Jatropha farm production yields by 200-300% in the next ten years. (Sulaiman and Jackson, 1959).

1.2.4 Biodiesel From Fungi:

A group at the Russian Academy of Sciences in Moscow published a paper in September 2008, stating that they had isolated large amounts of lipids from single-celled fungi and turned it into biodiesel in an economically efficient manner. More research on this fungal species; *Cunninghamella japonica*, and others, is likely to appear in the near future. (Usher ,1984).

The recent discovery of a variant of the fungus *Gliocladiumroseum* points toward the production of so-called myco-diesel from cellulose. This organism was recently discovered in the rainforests of northern Patagonia and has the unique capability of converting cellulose into medium length hydrocarbons typically found in diesel fuel. (Wikipedia, 2007).

1.2.5 BiodieselFromUsed GroundsCoffee:

Researchers at the University of Nevada, Reno, successfully produced biodiesel from oil derived from used coffee grounds. Their analysis of the used grounds showed a10% to 15% oil content (by weight). Once the oil was extracted, it underwent conventional processing into biodiesel. It is estimated that finished biodiesel could be produced for about one US dollar per gallon. Further, it was reported that "the technique is not difficult" and that "there is so much coffee around that several hundred million gallons of biodiesel could potentially be made annually." However, even if all the coffee grounds in the world were used to make fuel, the amount produced would be less than 1 percent of the diesel used in the United States annually. "It won't solve the world's energy problem," Dr. Misra said of his work. (Chengetal, 2004).

1.2.6 Exotic Sources:

Recently, alligator fat was identified as a source to produce biodiesel. Every year, about 15 million pounds of alligator fat are disposed of in landfills as a waste byproduct of the alligator meat and skin industry. Studies have shown that biodiesel produced from alligator fat is similar in composition to biodiesel made from soybeans, and is cheaper to refine since it is primarily a waste product. (Pongamiacsheet).

1.2.7Biodiesel To Hydrogen-cell Power:

A microreactor has been developed to convert biodiesel into hydrogen steam to power fuel cells.(Freedman and Pryde,1986)

Steam reforming, also known as fossil fuel reforming is a process which produces hydrogen gas from hydrocarbon fuels, most notably biodiesel due to its efficiency. Reformer is the processing device in which water vapor reacts with the liquid fuel under high temperature and pressure. Under temperatures ranging from 700 – 1100 °C, a nickel-based catalyst enables the production of carbon monoxide and hydrogen:

Hydrocarbon + $H_2O \leftrightarrow CO + 3 H_2$ (Highly endothermic)

Furthermore, a higher yield of hydrogen gas can be harnessed by further oxidizing carbon monoxide to produce more hydrogen and carbon dioxide:

 $(CO + H_2O \rightarrow CO_2 + H_2 \text{ (Mildly exothermic).(Cooks. and Van Rede ,1997).}$

1.3Problems and Potential Solutions OfUsing Vegetable Oils:

The direct use of vegetable oils or blends has generally been considered to be impractical for both direct and indirect diesel engines. The high viscosity, low volatility, acid composition, free fatty acid and moisture content, gum formation due to oxidations and polymerization during storage and combustion, poor cold engine start-up, misfire, ignition delay, incomplete combustion, carbon deposition around the nozzle orifice, ring sticking, injector choking in engine and lubricating oil thickening are the major problems of using vegetable oils. In general, the problems associated with using straight vegetable oil in diesel engines are classified into short term and long term.

1.4 Economical Viability Of Biodiesel:

Biodiesel is an attractive renewable energy resource. However, there are some challenges that face this vital resource. These challenges include the high cost and limited availability of biodiesel feedstock beside the cheaper prices of crude petroleum. There are various factors contributing to the cost of biodiesel. These factors include feedstock prices, plant's capacity, feedstock quality, processing technology, net energy balance nature of purification and its storage, etc. However, the two main factors are the costs of feed stocks and the cost of processing into Biodiesel. It has been found that the cost of feed stocks accounts for 75% of the total. Therefore, selecting the best feedstock is vital to ensure low biodiesel cost. It has been found that, considering non-edible oils as a feedstock for biodiesel can reduce this cost. In terms of production cost, there also are two aspects, the trans-esterification process cost and by-the product (glycerol) recovery. The continuous transesterification process is one choice to lower the production cost. The foundations of this process are a shorter reaction time and greater production capacity. The recovery of high quality glycerol is another way to lower biodieselproduction cost. Therefore, biodiesel plant must have its own glycerol recovery facility (Kamel,etal,1991).Biodiesel produced with current technology in many countries are still two to three times more expensive than gasoline and diesel. Moreover, a review of many economic feasibility studies around the world shows that biodiesel usually costs over US\$0.5 per liter compared to US \$0.35 per liter for nor-mal diesel. Currently biodiesel cost

is 1.5–3 times higher than the fossil diesel cost in developed countries. Biodiesel is thus currently not economically feasible, and more research and technological development are needed. Thus supporting policies such as tax credits are important to promote biodiesel research and make their prices competitive with other conventional sources of energy (Bailey,1951).

1.5Biodiesel Future:

Acceptance of Kyoto protocol and clean development mechanism will lead to more Biodiesel production around the world. For instance, it is anticipated that this policy will lead to a total bio-fuel demand in EU of around 19.5 and 30.3 million tons in 2012 and 2020 respectively. Biodiesel production is expanding rapidly around the world, driven by energy security and other environmental concerns. Given geographic disparities between demand and supply potential, and supply cost, expanded trade in *Biodiesel* appears to make sense. Global potential in Biodiesel production is very unclear, but in the long run it could be a substantial percentage of transport fuel demand. Currently, Biodiesel can be more effective if used as a complement to other energy sources. With the increase in global human population, more land will be needed to produce food for human consumption. Thus, the insufficient lands could increase the production cost of Biodiesel plants. This problem already exists in Asia where vegetable oil prices are relatively high. The same trend will eventually happen in the rest of the world. This is the potential challenge to *Biodiesel* production. Therefore, non-edible oil, genetically engineered plants and microalgae feed stocks can be proper solutions for this problem and can ensure the sustainability of biodiesel production in the future(Atabanietal, 2007).

1.6 Oil Extraction Methods:

There are three main methods that have been identified for extraction of the oil: (i) Mechanical extraction, (ii) solvent extraction and (iii) enzymatic extraction. Mechanical expellers or presses can be fed with either whole seeds or kernels or a mix of both, but common practice is to use whole seeds. However, for chemical extraction only kernels are used as feed (Palashetal, 2013).

1.6.1 Mechanical Extraction:

The technique of oil extraction by mechanical presses is the most conventional one among other methods. In this type, either a manual ram press or an engine driven screw press can be used. It has been found that, engine driven screw press can extract 68–80% of the available oil while the ram presses only achieved 60–65%. This broader range is due to the fact that seeds can be subjected to a different number of extractions through the expeller. The oil extracted by mechanical presses needs further treatment of filtration and degumming. One more problems associated with conventional mechanical presses are, their design is suited for some particular seeds, and therefore the yield is affected if used for other seeds. It has been found that, pretreatment of the seeds, such as cooking, can increase the oil yield of screw pressing up to 89% after single pass and 91% after dual pression (Mofijur,2013).

1.6.2 Solvent Extraction (chemical extraction):

Solvent extraction is the technique of removing one constituent from a solid by means of a liquid solvent. It is also called leaching. There are many factors influencing the rate of extraction such as particle size, the type of solvent chosen, temperature and agitation. The small particle size is preferable because it allows for greater interfacial area between the solid and liquid. The liquid chosen should be a good selective solvent and its viscosity should be, sufficiently, low to circulate freely. Temperature also affects the extraction rate. The solubility of the material will increase with the increasing temperature. Agitation of the solvent also it increases the eddy diffusion and therefore increases the transfer of material from the surface of the particles. Solvent extraction is only economical at large-scale production of more than 50 ton Biodiesel per day. There are three methods that are used. (Mofijur, 2013).

- (1) Hot water extraction.
- (2) Soxhlet extraction.
- (3) Ultrasonication technique.

1.6.3Enzymatic Oil Extraction:

Enzymatic oil extraction technique has emerged as a promising technique for extraction of oil. In this process suitable enzymes are used to extract oil from

crushed seeds. Its main advantages are that it is environment friendly and does not produce volatile organic compounds. However the main disadvantage of this technique However, this type consumes much more time compared to other types. (Mofijur, 2013).

It has been found that the chemical extraction using n-Hexane method results in the highest oil yield which makes it the most common type. N-Hexane solvent extraction has negative environmental impacts as a result of the waste water generation, higher specific energy consumption and higher emissions of volatile organic compounds and human health impacts (working with hazardous and inflammable chemicals). Using aqueous enzymatic oil extractions greatly reduces these problems. In aqueous enzymatic oil extraction the use of alkaline protease gave better results. Furthermore, ultrasonication pretreatment is a more useful step in aqueous oil extraction (Palashetal,2013).

The term oil is used in generic sense to describe all substances that are greasy or oily fluids at room temperature. They are non-volatile and are insoluble in water but are soluble in organic solvents. Oils from seeds or kernels or nuts along with proteins and carbohydrates, constitute the majority of foodstuffs. They are also found in wide industrial applications, like formulation of soap, toiletries, paints, varnishes, bio-diesels and lubricant.

The criteria for the selection of oil for industrial use are: presence of natural characteristic aroma, clarity, good natural colour, very low moisture content, freedom from solid particles and freedom from flat and rancid (unpleasant) odour (Okoye, 1999).

1.7Transesterification Process:

Is the chemical process which replaces one type of alcohol for another in an ester. An ester is made by combining an alcohol with an acid.

Transesterification is regarded as the best method among other approaches due to its low cost and simplicity (AlAlshaaletal,2010). Biodiesel is the main product of this process. Transesterification consists of a number of consecutive, reversible reactions. In these reactions, the triglycerides are converted step wise to diglycerides, monoglyceride and finally glycerol which sinks to the bottom and Biodiesel which floats on top and can be siphoned off Glycerol is an important byproduct and can be burned for heat or be used as feedstock in the cosmetic industry. In this reaction, methanol and ethanol are the two main light alcohols used for transesterification process due to their relatively low cost. How-ever,

propanol, isopropanol, tert-butanol, branched alcohols and octanol and butanol can also be employed but the cost is much higher (Al Alshaaletal, 2010). In this reaction, 100 lb of fat or oil are reacted with 10 lb of a short chain alcohol in the presence of a catalyst to produce 10 lb of glycerin and 100 lb of biodiesel. As per the transesterification reaction, 3 moles of methanol were required to react with the vegetable oil (Khare, 2007). Generally, transesterification process includes two main processes; catalytic and non-catalytic method. A catalyst is used to commence the reaction. The catalyst is vital as alcohol is barely soluble in oil or fat. The catalyst enhances the solubility of alcohol and thus increases the reaction rate. The most frequently used process is the catalytic transesterification process. Alkaline catalysts include catalysts such as NaOH, NaOCH₃, KOCH₃, KOH, NaOCH₃ and K2CO₃. Most of the Biodiesel producers use sodium hydroxide or potassium hydroxide. Even though some authors reported that sodium hydroxide is better than potassium hydroxide and some are of the view that potassium hydroxide is better than sodium hydroxide. However, most of the researchers reported that both sodium and potassium hydroxide perform equally well. Sodium and potassium methoxides return better yield than all catalysts but they are costly, so they are not very frequently used. Many researchers have found that alkaline cat-alytic method is the fastest and most economical catalyst than other catalysts. An alkaline catalyst proceeds at around 4000 times faster than with the same amount of acid catalyst. Moreover, this method can achieve high purity and yield of biodiesel product in a short time (30-60 min). Therefore, it dominates the current biodiesel production methods. However, to use alkaline catalysts, free fatty acid (FFA) level should be below a desired limit (ranging from less than 0.5% to less than 3%). Beyond this limit the reaction will not take place and the product formed will be soap and water instead of esters and the yield of esters will be too less. In addition to, this reaction has several drawbacks; it is energy intensive; recovery of glycerol is difficult; the catalyst has to be removed from the product; alkaline wastewater requires treatment and the level of free fatty acids and water greatly interfere with the reaction (Khare, 2007). Acid catalysts include sulfuric, hydrochloric, ferric sulfate, phos-phoric and organic sulfonic acid. Some researchers have claimed that acid catalysts are more tolerant than alkaline catalysts for veg-etable oils having high free fatty acids and water. Therefore, acid catalyst is used to reduce the free fatty acids contents to a level safe enough for alkaline transesterification which is preferred over the acid catalyst after the acid value is reduced to the desired limit. It has been reported that acid-catalyzed reaction gives very high yield in esters. It has been reported that the homogeneous transesterification consumes large amount of water for wet washing to remove the salt produced from the neutralization process, and the residual acid or base catalyst. Nevertheless, there are many companies around the world commercializing this technology because of its relatively lower energyuse, high conversion efficiency and cost effective reactants and cat-alyst Lipase catalysts such as Diazomethane CH₂N₂ have shown good tolerance for the free fatty acid level of the feedstock. Moreover, they are known to have a propensity to act on long-chain fatty alcohols better than on short-chain ones. Therefore, the efficiency of the transesterification of triglycerides with ethanol is higher com-pared to that with methanol in systems with or without a solvent. In this reaction, there is no need for complex operations for the recovery of glycerol and the elimination of catalyst and soap. However, the reaction yields and the reaction times are still unfavorable compared to the alkaline catalysts. Moreover, lipases are very expensive for large scale industrial production and they are unable to provide the degree of reaction completion required to meet ASTM fuel specifications. Recently, it has been found that the use of solvent-tolerant lipases, multiple enzymes and immo-bilized lipases-making catalysts can be developed as costeffective enzymes (Kamel,etal,1991). In general, chemically catalyzed processes, including alkali catalyzed and acid catalyzed ones have been proved to be more practical than the enzyme catalyzed process (Speroni,etal,2005). The catalytic transesterification has some problems such as: high time consumption, lag of reaction time caused by the extremely low solubility of the alcohol in the triglyceride phase and the need for separation of the catalyst and saponified impurities from Biodiesel. These problems are not faced in the non-catalytic transesterification methods. For instance, supercritical methanol method uses lower energy and completes in a very short time (2-4 min) compared to catalytic transesterification. Further, since no catalyst is used, the purification of biodiesel and the recovery of glycerol are much easier, trouble free and environment friendly (Koko,etal,2000). However, the method has a high cost in reactor and operation (due to high pressures and high temperatures), and high methanol consumption (e.g., high methanol/crude-oil molar ratio of 40/1) (Hardman,etal,1970). There are a variety of studies have been conducted for trans-esterification reaction for different catalysts, alcohols and molar ratios at different temperatures.

1.8Balanites aegyptiaca:

*Balanitesaegyptiaca*fig 1:1, classified either as a member of the Zygophyllaceae or the *Balanitaceae*.(Hall, 1991) This tree is native to much of Africa and parts of the Middle East.(Hall etal, 1992).

There are many common names for this plant.(*Balanitesaegyptiacus* (*L*) *Delile*] In English the fruit has been called desert date, soap berry tree or bush, Thron tree, Egyptian myrobalan, Egyptian balsam or Zachum oil tree;(Ndoye etal.2004) in Arabic it is known as lalob, hidjihi, inteishit, and heglig (hijlij). In Hausa it is called aduwa, in Swahili mchunju and in Amharicbedena.[The wealth of india] *balanitesaegyptiaca* Del, a member of the family Zygophyllaceae, is one of the most common but neglected wild plant species of the dry land areas of Africa and South Asia. In India, it is particularly found in Rajasthan, Gujarat, Madhya Pradesh, and Deccan .it is one of the most common trees in Senegal.



Fig1.1 Balanitesaegyptiacatree:

(Sulaiman and Jackson,1959). It can be found in many kinds of habitat, tolerating a wide variety of soil types, from sand to heavy clay, and climatic moisture levels. In Sudan the *balanitesaegyptiaca* trees are found in multi areas like southern kordofan state, Abu jibeiha province and Sinnar state also found in Elgadarf (alhwata) East of Sudan (Usher, 1984).

1.8.1 Scientific Classification of Balanitesaegyptiaca Tree:

Kingdom: Plantae

(unranked): Angiosperms

(unranked): Eudicots

(unranked): Rosids

Order: <u>Zygophyllales</u>

Family: <u>Zygophyllaceae</u>

Genus: <u>Balanites</u>

Species: Balanitesaegyptiaca

1.8.2Description:

The *Balanitesaegyptiaca* treeFig 1:1 reaches 10 m (33 ft) in height with a generally narrow form. The branches have long, straight green spines arranged in spirals. The dark green compound leaves grow out of the base of the spines and are made up of two leaflets which are variable in size and shape.(National plant data center) The fluted trunk has grayish-brown, ragged bark with yellow-green patches where it is shed. The tree produces several forms of <u>inflorescence</u> bearing yellow-green <u>bisexual flowers</u> with five long greenish petals. In Senegal, they are <u>pollinated</u> by <u>halictid bees</u>, <u>Halictus gibber</u>, and <u>flies</u>, <u>Rhiniaapicalis</u> and <u>Chrysomiachloropiza</u>. The carpenter and <u>Camponotussericeus</u> feeds on the nectar exuded by the flowers. The larva of the cabbage tree emperor moth <u>Bunaeaalcinoe</u> causes <u>defoliation</u> of the tree.

1.8.3Agro Forestry:

The tree is managed through <u>agroforestry</u>. It is planted along irrigation canals and it is used to attract insects for trapping. The pale to brownish yellow wood is used to make furniture and durable items such as tools, and it is a low-smoke <u>firewood</u> and good <u>charcoal</u>. The smaller trees and branches are used as living or cut fences because they are resilient and thorny. The tree <u>fixes nitrogen</u>. It is grown for its fruit in <u>plantations</u> in several areas. The bark yields <u>fibers</u>, the <u>natural gums</u> from the branches are used as <u>glue</u>, and the seeds have been used to make <u>jewelry</u> and <u>beads</u>.(National plant data center).

1.8.4Distribution:

Balanitesaegyptiaca is found in the Sahel-Savannah region across Africa. It is one of the most common trees in <u>Senegal</u> and Mauritania, one of its local names is teishit.(Kirtikaretal,1823). It can be found in many kinds of habitat, tolerating a wide variety of <u>soil</u> types, from <u>sand</u> to heavy <u>clay</u>, and climatic moisture levels, from <u>arid</u> to subhumid. It is relatively tolerant of flooding, livestock activity, and <u>wildfire</u>.(Pandeyetal,2005).

1.9Uses OfBalanitesaeqyptiaca:

1.9.1Traditional Uses:

Aqueous extract of fruits showed spermicidal activity without local vaginal irritation in (human beings), up to 4% sperms becoming sluggish on contact with the plant extract and immobilis within 30 seconds. the effect was concentrationrelated. Protracted administration of the fruit pulp extract produced hyperglycemiainduced testicular dysfunction in dogs. Seeds are used as expectorant, antibacterial, and antifungal. Fruit is used in whooping cough, also in leucoderma and other skin diseases. the bark is used as spasmolytic. (Khare and India medicinal plants, 2007). The seed is used as a febrifuge.(Creach etal,1951) Root extracts have proved 'slightly effective' against malaria. In Kenya, a root infusion is used as an emetic.(Beentje, 1994) In asthma, about 10 gm of seed powder is taken with glass of water in the morning for 10 days.(Jagtap,etal,2009) Tablets are prepared from roots mixed with 'Hing' powder (Ferula asafoetida); by adding Piper bettle leaf, juices are taken once with water for 9 days, soon after menstruation to avoid unwanted In Egyptian folk medicine, the fruits are used as an oral hypoglycemic(Kamel ,1998) and an anti diabetic; an aqueous extract of the fruit Sudanese folk medicine in mesocarp is used in the treatment jaundice.(Sarkeretal, 2000) Used in food preparations and herbal medicine, especially in Africa and some developing Countries. (Obidah etal, 2009). The fresh leaf of the plant Acalypha is pounded with small amount of root of Balanitesaegyptiaca and Cissusquadrangularis, and then soaked in water for an hour or two. It is decanted and administered intranasally and orally. Latex of the plant is used in epilepsy, administered through intranasal route. (Araya, 2007) Used as tooth paste. (Khalid etal,2010). Fruits are used to treat dysentery and constipation. The seed oil is used to treat tumors and wounds.(Ojoetal, 2006). Used as laxative, also used in treatment of hemorrhoid, stomach aches, jaundice, yellow fever, syphilis, and

epilepsy.(Barley etal,1962). The fruit is used to treat liver disease and as a purgative, and sucked by school children as a confectionary in some countries the bark is used in the treatment of syphilis, round worm infections, and as a fish poison. The aqueous leaf extract and saponins isolated from its kernel cakes have antibacterial activity. Seeds are used as anthelmintic and purgative. Ground seeds are given to camels to cure impaction and colic. (Khan,2009).

In Chifra District which country, the root of plant is used for the treatment of render pest and anthrax. In East Africa, it is widely used as anthelmintic. Root is used in various folk medicines for the treatment of abdominal pain and as purgative, while the bark is employed as a fish poison and also as a remedy for malaria and syphilis. The root, bark, kernel, and fruit have been shown to be lethal to mollusks.(Kameleal,1991). In Sudanese folk medicine, it is used to treat jaundice.(Ndabanezeetal,1994) Its anti malarial and molluscidal activity is well studied.(Kwuosaetal,1993). In vitro anti plasmodial test of the dichloromethane and methanol extract of stem bark of the plant showed anti malarial activity.

In Senegal, Nigeria, Morocco, and Ethiopia, *Balanitesaegyptiaca* is taken a purgative for colic and stomach ache. In Chad, fresh twigs are burnt to keep insects away. For intestinal worm, the fruits are dried and mashed in millet porridge and eaten. (Breyeretal,1982). In Libya and Eritrea, the leaves are used for cleaning infected wounds. In Sudan and Chad the bar, *Balanites.aegyptiaca* is component of soap.(Oliver-Bever ,1986). The use of the kernel oil for treatment of wounds has been reported from Nigeria For contraception, in Nigeria, a mixture of dried leaves powder of *Balanitesaegyptiaca* and *Ricinuscommunis* in water and in Somalia, the bark of root is crushed and mixed with two glasses of water, which is then filtered. This preparation is repeated for three days and one glass is drunk three times daily for three days. (Neuwinger,1944).

1.9.2Food:

Balanitesaegyptiaca has been cultivated in Egypt for more than 4000 years, and stones placed in the tombs as votive offerings have been found as far back as the Twelfth Dynasty. The tree was figured and described in 1592 by Prosper Alpinus under the name 'agihalid'. Linnaeus regarded it as a species of Ximenia, but Adanson proposed the new genus of Agialid. The genus Balanites was founded in 1813 by Delile. (Schmidt etal, 2001).

The yellow, single-seededfruit is edible, but bitter. (Pandeyetal, 2005) Many parts of the plant are used as famine foods in Africa; leaves and flowers are eaten raw or cooked, the oily seed is boiled to make it less bitter and eaten mixed with sorghum, The tree is considered valuable in arid regions because it produces fruit even in dry times. The fruit can be fermented for alcoholic beverages. The seed cake remaining after the oil is extracted is commonly used as animal feed in Africa. The seeds of Balanitesaegyptiaca have molluscicide effect on Biomphalariapfeifferi. (The wealth Of India). Where the species coexist, African elephants consume the desert date. (Khare and India medicinal plants, 2007).

1.10Botancial Description:

It is multibranched, spiny shrub or tree up to 10 m tall. Crown spherical, in one or several distinct masses. Trunk short and often branching from near the base. Bark dark brown to grey, deeply fissured. Branches armed with stout yellow or green thorns up to 8 cm long. Leaves with two separate leaflets; leaflets obovate, asymmetric, 2.5 to 6 cm long, bright green, leathery, with fine hairs when young. Flowers in fascicles in the leaf axils, and are fragrant, yellowish-green.

1.11Distribution and Habitat:

Natural distribution is obscured by cultivation and naturalization. It is believed indigenous to all dry lands south of the Sahara, extending southward to Malawi in the Rift Valley, and to the Arabian Peninsula, introduced into cultivation in Latin America and India. It has wide ecological distribution, but is mainly found on level alluvial sites with deep sandy loam and free access to water. After the seedling stage, it is intolerant to shade and prefers open woodland or savannah for natural regeneration. It is a lowland species, growing up to 1000 m altitude in areas with mean annual temperature of 20 to 30°C and mean annual rainfall of 250 to 400 mm.(Khare and India medicinal plants,2007).Fruit is a rather long, narrow drupe, 2.5 to 7 cm long, 1.5 to 4 cm in diameter. Young fruits are green and tormentose, turning yellow and glabrous when mature. Pulp is bitter-sweet and edible. Seed is the pyrene (stone), 1.5 to 3 cm long, light brown, fibrous, and extremely hard. It makes up 50 to 60% of the fruit. There are 500 to 1 500 dry, clean seeds per kg.

Flowers are small, inconspicuous, hermaphroditic, and pollinated by insects. Seeds are dispersed by ingestion by birds and animals. The tree begins to flower and fruit

at 5 to 7 years of age and maximum seed production is when the trees are 15 to 25 years old.

1.11.1Medicinal Uses:

Desert date fruit is mixed into porridge and eaten by nursing mothers, and the oil is consumed for headache and to improve lactation. Oil from the fruit is used to dress Bark extracts and the fruit repel (Creach etal, 1951) or destroy freshwater snails and copepods, organisms that act as intermediary hosts host the parasites Schistosoma, including Bilharzia, and guinea worm, respectively. Existing worm infections are likewise treated with desert date, as are liver and spleen disorders. A decoction of the bark is also used as an Abortifacient and an antidote for arrow-poison in West African traditional medicine. (Nodyeetal, 2004). The seed contains 30-48% fixed (non-volatile) oil, like the leaves, fruit pulp, bark and roots, and contains the sapogeninsdiosgenin and yamogenin (Creachetal, 1951). Saponins likewise occur in the roots, bark wood and fruit. (Nodye,etal,2004). Diosgenin can be used to produce hormones such as those in combined oral contraceptive pills and corticoids.(Kirtikar Detal, 1823). The oil exhibited anticancer activity against lung, liver, and brain human carcinoma cell lines. It also had antimutagenic activity against Fasciolagigantica-induced mutagenicity besides anthelmintic activity against hepatic worms (Schistosomamansoni and Fasciolagigantica). Preliminary screening showed that the oil had antiviral activity against Herpes simplex virus. It also had antimicrobial activity against selected strains of Gram-positive bacteria, Gram-negative bacteria, and Candida.(Dawidaretal, 1969).

1.12Phytochememical Constituents:

1.12.1Leaves:

It contains saponin, furanocoumarin, and flavonoid namely quercetin 3-glucoside, quercetin-3-rutinoside; 3-glucoside, 3-rutinoside, 3-7-diglucoside and 3-rhamnogalactoside of isorhamnetin. (Salwa and Elhadidi,1988).

1.12.2Fruit:

Mesocarp of fruit contains 1.2 to 1.5% protein and 35 to 37% sugars, 15% organic acids, other constituents like 3-rutinoside and 3-rhamnogalactoside, diosgenin; (Hosny, etal, 1992) it also contain a mixture of 22R and 22S epimers of 26-(O- β -D-glucopyranosyl)-3- β -(4-O-(β -D-glucopyranosyl)-2-O-(α -L-rhamnopyranosyl)- β -D-glucopyranosyloxy)-22,26-dihydroxyfurost-5-ene. However, kernel contains a xylopyranosyl derivative of above saponin present in mesocarpBalanitoside, (furostanol glycoside) and 6-methyldiosgenin, (

Charlemagne, et al, 2008) balanitin-3 (spirostanol glycoside) have been reported from fruits (mesocarp) of balanitesaegyptiaca] Balanitin-6 and -7: Diosgenylsaponins two pregnane glycosides namely pregn-5-ene-3β,16β,20(R)-triol 3-O-(2,6-di-O-α-(balagyptin), 1-rhamnopyranosyl)-β-d-glucopyranoside and pregn-5-ene-3β,16β,20(R)-triol 3-O-β-d-glucopyranoside, long chain hydrocarbon. The kernels contained 45.0 to 46.1% oil and protein (32.4%), oil contains mainly palmitic, stearic, oleic, and linoleic acids which were the main fatty acids (Chapagain and Wiesman Z,2007)] Nine saponin have been reported from kernel cake of Balanitesaegyptiaca, from the nine components, six saponins with molecular masses of 1196, 1064, 1210, 1224, 1078, and 1046 Da were identified, with the compound of mass 1210 Da being the main saponin (ca. 36%). [The leaves and fruit kernels of Balanitesaegyptiaca L. were found to contain six diosgeninglucosides including di-, tri-, and tetraglucosides. Hydrolysis of the saponins gave 25D-spirosta-3, 5-diene and 3β-chloro-25D-spirost-5-ene balanitin-1, -2, and -3. (Sahran,etal,1970).

1.12.3Root:

the root is reported to contain steroidal saponin about 1% glycosides and the major sapogenin is yamogenin (Hardamanetal,1970) other glycosides; $(3\beta,12\alpha,14\beta,16\beta)$ -12-hydroxycholest-5-ene-3,16-diylbis(β -D-glucopyranoside), $(3\beta,20s,22R,25R)$ -, and $(3\beta,20s,22R,25s)$ -26- $(\beta$ -D-glucopyranosyloxy)-22-methoxyfurost-5-en-3-yl- β -D-xylopyranosyl- $(1\rightarrow 3)$ - β -D-glucopyranosyl- $(1\rightarrow 4)$ [α -Lrhamnopyranosyl- $(1\rightarrow 2)$]- β -D-glucopyranosyl- $(1\rightarrow 3)$ - β -D-glucopyranosyl- $(1\rightarrow 4)$ [α -Lrhamnopyranosyl- $(1\rightarrow 4)$] β -D-xylopyranosyl- $(1\rightarrow 3)$ - β -D-glucopyranosyl- $(1\rightarrow 4)$ [α -Lrhamnopyranosyl- $(1\rightarrow 2)$]- β -D-glucopyranoside, *balanitins* 1 to 7 have been reported from root and bark of *Balanitesaegyptiaca*.(Sarkeretal,2000).

1.12.4Bark:

reported contain furanocoumarinbergapten the bark is to and dihydrofuranocumarin D- marmesin, (Ansare and Ahmed, 2006) two alkaloid namely, N-trans-feruloyltyramine and N-cis-feruloyltyramine, and three common syringic acid; and 3-hydroxy-1-(4-hydroxy-3metabolites, vanillic acid, methoxyphenyl)-1-propanone, (Kapse, et al, 1997). long-chain aliphatic compound, 10-methyl-n-heptacosane, and a new sugar, diglucosyldirhamnoside, have also been reported from the stem-barks, It also contains beta-sitosterol, bergapten, marmesin, and beta-sitosterol glucoside, balanitin-1,-2, and -3; balanitin-1 for example possesses a yamogeninaglycone with a branched glucose and rhamnose side chain (Elmastry,etal,210).

1.13Pharmaclogical Activity:

1.13.1Cardioprotective Antioxidant Activity:

The plant acts as antioxidant against 0 adriamycin-induced cardiotoxicity in experimental mice. Adriamycin when administered intraperitoneally, it cause elevation of serum lactate dehydrogenase, creatine phosphokinase, glutamate oxaloacetate transaminase, glutamate pyruvate transaminase, lipid peroxide, total nitric oxide, erythrocyte lysate superoxide dismutase (SOD), glutathione peroxidase (GPx), and plasma catalase (CAT) in mice heart tissue. Adriamycin drug reduced the activities of SOD, GPx, and CAT. Pretreatment with *Balanitesaegyptiaca* extract significantly (*P*<0.05) prevented these alterations and restored the enzyme activities to near normal levels. (Dwivedietal,2009).

1.13.2Anthelmintic activity:

The crude aqueous extract of root bark of Balanitesaegyptiaca was showed a dosedependent inhibition of spontaneous motility (paralysis) in adult earthworms. And also possesses vermicidal activity. (Koko,etal,2000) It is reported that stem bark water extract (9 g/kg body weight) of Albiziaanthelmintica and fruit mesocarp water extract (9 g/kg body weight) of Balanitesaegyptiaca shows significant anthelmintic activity compared with albendazole (20 mg/kg body weight) Fasciolagigantica adult worm. (Kokoetal, 2005) And a single dose of 200 mg/kg body weight of Balanitesaegyptiaca fruit mesocarp also showed activity against Schistosomamansoni in infected mice when compared with praziquantel.(Shalaby,etal,2010). Balanitin-7 is isolated from aqueous extract Balanitesaegyptiaca seeds and reported as anthelmintic agent when tested by in vitro means of an original anthelmintic assay, using Caenorhabditiselegans as a biological model.(Antoetal, 2005). The methanolic extract of Balanitesaegyptiaca fruits is reported to have anthelmintic action against different stages of Trichinellaspiralis anthelmintic drug compared with albendazole.(Doughari Pukuma, 2007). The aqueous extract of *Balanitesaegyptiaca* also has molluscicidal agent iuvenile and adult **Bulinus** globosus and to Bulinustruncatus.(Karuppusamyetal, 2002).

1.13.3Antibacterial Effects:

Aqueous and organic leaves extracts of *Balanitesaegyptiaca* and *Moringaoleifera* were reported to have antibacterial effect against *Salmonella typhi* isolated from blood clot culture using the disc diffusion method. The extracts of *Balanitesaegyptiaca* plants demonstrated the highest activity than *Moringaoleifera*. The ethanolic extracts of both plants demonstrated highest activity whereas the aqueous extracts of both plants showed the least activity at 100 mg/ml as compared with ethanolic extracts. The activities of these plant extracts were comparable with those of antibiotics, ciprofloxacin, cotrimoxazole, and chloramphenicol, commonly used for treating typhoid fever. The antibacterial activity appears to increase when extracts of the two plants were used in combination at 100 mg/ml each. Preliminary phytochemical screening showed that plant extracts contain saponins, tannins, and phenols, and *Balanitesaegyptiaca* possesses anthraquinones. The antibacterial activities of the extracts on *S. typhi* were reasonably stable when treated at 4, 30, 60, and 100°C for 1 hour. However, it reduces significantly when the pH was altered toward alkalinity. (Wufenetal, 2007).

The aqueous and ethanolic extracts of leaves of six plants viz., *Balanitesaegyptiaca* (L.) Del, *Hyptissuaveolens*Poit, *Lawsoniainermis* L., *Leucasaspera* L., *Lobelia nicotianifolia* Roth, and *Phyllanthusmaderaspatana*L.were reported as antibacterial when tested individually and in combinations against five different diarrheagenic *bacteria, Bacillus cereus, Staphylococcus aureus, Escherichia coli, Salmonella enteritidis, and Listeria monocytogenes*. Ciprofloxacin (20 µg) was used as antimicrobial standard. The highest antimicrobial activity was in both crude aqueous leaf extract and crude ethanolic leaf extract of *Lobelia nicotianifolia*, when all extracts were tested individually. However, in combination, the highest activity was observed in crude ethanolic leaf extract *Lobelia nicotianifolia* + *Balanitesaegyptiaca* against *S. aureus*.(Wufenetal, 2007).

1.13.4Antivenin Activity:

The acetone and methanolic extracts of stem bark of plant has reported an antivenin activity against saw-scaled (*Echiscarinatus*) viper venom concentration at lethal dose (0.194 mg/ml), when administered intramuscularly to Wistar albino rats. Both extracts were found to be effective at 75 and 100 mg/ml concentrations. (Pettitetal,1991).

1.13.5Anticancer Aactivity:

A mixture of steroidal saponins: balanitin-6 (28%) and balanitin-7 (72%), isolated from *Balanitesaegyptiaca* kernels, demonstrated appreciable anticancer effects in human cancer cell lines *in vitro* by using against A549 non–small-cell lung cancer (IC₅₀, 0.3 μM) and U373 glioblastoma (IC₅₀, 0.5 μM) cell lines.Bal6/7 displayed higher antiproliferative activity than etoposide and oxaliplatin, markedly less active than taxol. It indicated that balanitin 6/7 mixture is more a cytotoxic compound than a cytostatic one. *In vitro* anticancer activities are due to partly depletion of (ATP)i, leading in turn to major disorganization of actin and it does not induce an increase in intracellular reactive oxygen species. In vivo, bal6/7 increased the survival time of mice bearing murine L1210 leukemia grafts to the same extent reported for vincristine. (Medaetal, 2010).

1.13.6Anti-inflammatory and Analgesic Activity:

Ethanol and petroleum ether extracts of aerial parts of Balanitesaegyptiaca have been reported to have significant anti-inflammatory action on a carrageenan-induced hind paw odema in rats, the paw volume was measured, plethy smometrically, at 0 and 3 hours after injection and analgesic activity by using Eddy's hot plate method and tail-flick method in albino rats. The ethanol and petroleum ether extracts showed a greater anti-inflammatory and analgesic effects comparative with the standard drugs, indomethacin and diclofenac sodium, respectively. It also indicated that the ethanolic extract of *Balanitesaegyptiaca* exhibited more significant activity extract the of than petroleum in treatment pain and inflammation.(Speronietal, 2005). In vitro antioxidant, xanthine oxidize acetylcholinesterase inhibitory activities It is reported that the galls and leaf extracts and fractions of Balanitesaegyptiaca showed a significant antioxidant, xanthine oxidize, and acetylcholinesterase inhibitory activities. The total phenolics and flavonoids were measured using Folin-Ciocalteu and AlCl₃ reagents, respectively. Two methods, that is, FRAP (Iron (III) to Iron (II) reduction activity) and ABTS (2, 2-azinobis-3-ethylbnzothiazoline-6-sulphonate) assay were used to estimate the total antioxidant capacity of the plant materials. Dichloromethane fraction of the Gall and ethyl acetate fractions of the leaves were reported to have highest antioxidant activity. The antioxidant activities were correlated significantly with the total phenolic and flavonoid contents. The study also showed that Balanitesaegyptiaca galls and leaves fractions exhibited a moderate xanthine oxidize

inhibitory activity compared with the acetylcholinesterase which was weakly inhibited by the tested extracts and fractions. (Zarrougetal, 1990).

1.13.7Anti-inflammatory, Antinociceptive and Antioxidant ctivities:

Methanolic and butanol extracts and of two new saponins isolated from *Balanitesaegyptiaca* showed significant anti-inflammatory, antinociceptive activity in the carrageenin-induced odema in the rat, and acetic acid-induced writhing test in mice and antioxidant action using *in vitro*, a method based on the Briggs–Rauscher oscillating reaction. Samples, extracts and pure substances, were intragastrically administered to animals. (Wiesman and Chapagain, 2006).

A saponin extract and water extract from fruit kernel of *Balanitesaegyptiaca* was investigated as a mosquito larvicide. Both extracts were tested against second and fourth instar larvae of the three mosquito species namely *Anopheles arabiensis*, *Culexquinquefasciatus*, and *A. aegypti*, and LC₅₀ and LC₉₀ values were determined. Second instar larvae were more susceptible than fourth instar larvae in all cases. The larvae of *Anopheles arabiensis* were more susceptible than *Culexquinquefasciatus* and *A. aegypti* to its larvicidal effects. The saponin was more active than the water extract. (Wiesman and Chapagain, 2003).

1.13.8Hepatoprotective Activity:

Administration of the aqueous extract to biliary duct-ligated rats showed a dose-dependent significant decrease in serum bilirubin level. For three days, the animals were given different concentration of the extract intraperitoneally. Bilirubin concentration was reduced by 22.2% in the animal that received 1.2 g bark extract each day, by 31.6% in those given 2.4 g, and by 45.9% in those given 4.8 g.

1.13.9Antidiabetic Activity:

The pure saponin, extracted from the balanite fruit mesocarp, and water extract have been reported as hypoglycemic agent when tested on albino rats in different concentrations dose and Daonil (as a standard medication). It was also reported to inhibit *Escherichia coli* growth in rats. The aqueous extract of the mesocarp of fruits of *Balanitesaegyptiaca* was reported to have antidiabetic effect in streptozotocininduced diabetic mice. (Annan and Dickson, 2008).

1.13.10Antiviral Activity:

It is reported that bark aqueous extract of *Balanitesaegyptiaca* used in treatment of both AIDS and Leukemia. An oral administration of the aqueous extract (30% w/v

given at 100 ml every 8 hours for 30 days) for the treatment of HIV patients have shown excellent results. The same was given to patients with leukemia and a good increase in platelets and a normal blood differential reading after one month was noted. (Abdel-rahimetal,1986).

1.13.11Wound Healing Activity:

It is reported that *Balanitesaegyptiaca* have potent wound-healing activity, as evident from the wound contraction. The results also indicated that plant possess potent antioxidant activity by inhibiting lipid peroxidation, bleaching DPPH (2, 2-diphenyl-1-picrylhydrazyl) radical, and protecting against oxidant injury to fibroblast cells. (Wanietal, 2010).

1.13.12Hypocholesterolemic Aactivity:

It is reported that whole and extracted pulp of *Balanitesaegyptiaca* fruits reported a hypocholesterolemic effect when tested on adult albino rats. (Jenaetal, 2010).

1.13.13Diuretic Activity:

Ethanol and methanolic extracts of *Balanitesaegyptiaca* werereported to have a diuretic effect when tested on Wistar albino rats with (150 and 300 mg/kg) oral doses. Frusemide was used as standard. The results indicate that ethanol and methanol extracts shows a significant (*P*<0.05) increase in the urine volume and electrolyte

1.14Advantages OfBiodiesel:

Biodiesel has 10–11% of oxygen; this makes biodiesel a fuel with high combustion characteristics Biodiesel reduces net carbon-dioxide emissions by 78% on a lifecycle basis when compared to conventional diesel fuel and reduces smoke due to free soot (Linetal,2011) Biodiesel is renewable, non-toxic, non-flammable, portable, readily available, biodegradable, sustainable, eco-friendly and free from sulfur and aromatic content, this makes it an ideal fuel for heavily polluted cities. Biodiesel also reduces particular matter content in the ambient air and hence reduces air toxicity. It provides a 90% reduction in cancer risks and neonatal defects due to its less polluting combustionbiodiesel helps rural development to restore degraded lands over a period. Moreover, it has good potential for rural employment generation (Pittitetal,1991) Biodiesel serves as climatic neutral in view of the climatic change that is presently an important element of energy use and development Biodiesel has higher cetane number

(about 60–65 depending on the vegetable oil) than petroleum diesel which reduces the ignition delay Production can be raised easily and is less time consuming No need for drilling, transportation, or refining like petroleum diesel. Therefore, each country has the ability to produce Biodiesel as a locally produced fuel. Moreover, there is no need to pay tariffs or similar taxes to the countries from which oil and petroleum diesel is imported (United State Environmental Protection Agency, 2014), Biodiesel has superior better lubricity properties. This improves lubrication in fuel pumps and injector units, which decreases engine wear, tear and increases engine efficiency Biodiesel is safe for transportation, handling, distribution, utilization and storage due to its higher flash point (above 100–170c) than petroleum diesel (60–80c) biodiesel reduces the environmental effect of a waste product and can be made out of used cooking oils and lards (United State Environmental Protection Agency, 2014), Biodiesel may not require engine modification up to B20. higher However, minor modification blends may need some (Karuppusamyetal, 2008).

1.14.1 Disadvantages OfBiodiesel:

Biodiesel has 12% lower energy content than diesel, this leads to an increase in fuel consumption of about 2–10%. Moreover, Biodiesel has higher cloud point and pour point, higher nitrogen oxide emissions than diesel. It has lower volatilities that cause the formation of deposits in engines due to incomplete combustion characteristicsbiodieselcauses excessive carbon deposition and gum formation (polymerization) in engines and the oil gets contaminated and suffers from flow problem. It has relatively higher viscosity (11–18 times diesel) and lower volatility than diesel and thus needs higher injector pressure (United State Environmental Protection Agency, 2014), Oxidation stability of Biodiesel is lower than that of diesel. It can be oxidized into fatty acids in the presence of air and causes corrosion of fuel tank, pipe and injector (United State Environmental Protection Agency, 2014), Due to the high oxygen content in Biodiesel, advance in fuel injection and timing and earlier start of combustion, biodiesel produces relatively higher NOxlevels than diesel in the range of 10–14% during combustion *Biodiesel* can cause corrosion in vehicle material (cooper and brass) such as fuel system blockage, seal failures, filter clogging and deposits at injection pumps (Ansari,etal,2006) Use of biodiesel in internal combustion engine may lead to engine durability problems including injector cocking, filter plugging and piston ring sticking, etc. (Beentje, 1994) As more than 95% of biodiesel is made from

edible oil, there have been many claims that this may give rise to further economic problems. By converting edible oils into biodiesel, food resources are being used as automotive fuels. It is believed that large-scale production ofbiodiesel from edible oils may bring about a global imbalance in the food supply-and-demand market (Charlemagne, et al, 2008) Lower engine speed and power, high price, high engine wear, engine compatibility (Charlemagne, et al, 2008), Transesterification process is expensive (cost of fuel increases), these oils require expensive fatty acid separation of less effective (or expensive acid (Chauhanetal, 2010), Transesterification has some environmental effects such as waste disposal and water requirement for washing, soap formation, etc. (Achten,etal,2008), standard EN 14214 has a Karl Fischer moisture specification of 0.050 wt% maximum tried (Canakci and Sanli,2010).

1.15Blends Biofuels:

Blends of biodiesel and conventional hydrocarbon-based diesel are products most commonly distributed for use in the retail diesel fuel marketplace. Much of the world uses a system known as the "B" factor to state the amount of biodiesel in any fuel mix:(Mahanta and Shrivastava A,2001)

- 100% biodiesel is referred to as B100.
- 20% biodiesel, 80% petrodiesel is labeled B20.
- 5% biodiesel, 95% petrodiesel is labeled B5.
- 2% biodiesel, 98% petrodiesel is labeled B2

Blends of 20% Biodiesel and lower can be used in diesel equipment with no, or only minor modification(Beerens,2007) although certain manufacturers do not extend warranty coverage if equipment is damaged by these blends. The B6 to B20 blends are covered by the ASTM D7467 specification.(Forsonetal,2004).

Mixing in tanks at manufacturing point prior to delivery to tanker truck Splash mixing in the tanker truck (adding specific percentages of biodiesel and petroleum diesel).

In-line mixing, two components arrive at tanker truck simultaneously. Metered pump mixing, petroleum diesel and biodiesel meters are set to X total volume, transfer pump pulls from two points and mix is complete on leaving pump.

1.16ApplicationsOf Bbiofuels:

Biodiesel can be used in pure form (B100) or may be blended with petroleum diesel at any concentration in most injection pump diesel engines. New extreme high-pressure (29,000 psi) common rail engines have strict factory limits of B5 or B20, depending on manufacturer. Biodiesel has different solvent properties than petrodiesel, and will degrade natural rubber gaskets and hoses in vehicles (mostly vehicles manufactured before 1992), although these tend to wear out naturally and most likely will have already been replaced. Which is nonreactive to biodiesel? Biodiesel has been known to break down deposits of residue in the fuel lines where petrodiesel has been used. As a result, fuel filters may become clogged with particulates if a quick transition to pure biodiesel is made. Therefore, it is recommended to change the fuel filters on engines and heaters shortly after first switching to a biodiesel blend(Demirbas AH and Demirbas, 2007).

1.17Fuel Efficiency:

The power output of biodiesel depends on its: blend, quality, and load conditions under which the fuel is burnt. The thermal efficiency for example of B100(biodiesel) as compared to B20 will vary due to the content of the various blends. Thermal efficiency of a fuel is based in part on fuel characteristics such as: viscosity, specific density, and flash point; these characteristics will change as the blends as well as the quality of biodiesel varies. The American Society for Testing and Materials(ASTM) has set standards in order to judge the quality of a given fuel sample.(Chauhanetal,2010).

1.18Combustion:

Fuel systems on modern, diesel engine were not designed to accommodate biodiesel. Traditional direct injection fuel systems operate at roughly 3,000 psi at the injector tip while the modern common rail fuel system operates at high pressure more than 30,000 PSI at the injector tip. Components are designed to operate at a wide temperature range, from below freezing to over 1,000 degrees Fahrenheit. Diesel fuel is expected to burn, efficiently, and produce few emissions as possible. As emission standards are being introduced to diesel engines the need to control harmful emissions is being designed into the parameters of diesel engine fuel systems. The traditional inline injection system is more forgiving to poorer quality fuels as opposed to the common rail fuel system. Higher pressures and tighter

tolerances of the common rail system allowfor greater control over atomization and injection timing. Control of atomization, combustion allow for greater efficiency of modern diesel engines. Components within a diesel fuel system interact with the fuel in a way to ensure efficient operation of the fuel system and so the engine. If a fuel is introduced to a system-that has specific parameters of operation-and you vary those parameters by an out of specification fuel you may compromise the integrity of the overall fuel system. Some of these parameters such as spray pattern and atomization are directly related to injection timing.(Agarwal,2007).

During atomization biodieseland its blends produced droplets that were greater in diameter than the droplets produced by traditional petrodiesel. The smaller droplets were attributed to the lower viscosity and surface tension of traditional petrol. It was found that droplets at the periphery of the spray pattern were larger in diameter than the droplets at the center this was attributed to the faster pressure drop at the edge of the spray pattern; there was a proportional relationship between the droplet size and the distance from the injector tip. It was found that B100 had the greatest spray penetration, this was attributed to the greater density of B100. Having a greater droplet size can lead to; inefficiencies in the combustion, increased emissions, and decreased horse power. In another hand it was found that there is a short injection delay when injecting biodiesel. This injection delay was attributed to the greater viscosity of Biodiesel. It was noted that the higher viscosity and the greater cetane rating of biodiesel over traditional petrodiesel lead to poor atomization, as well as mixture penetration with air during the ignition delay period.(Schwab,etal,(1988)) Another noted that this ignition delay may aid in a decrease of NOxemission.(Abu-Al-Futuh,1989).

1.19Emissions:

Emissions are inherent to the combustion of diesel fuels that are regulated by the U.S. Environmental Protection Agency (E.P.A.). As these emissions are a byproduct of the combustion process in order to ensure E.P.A. compliance a fuel system must be capable of controlling the combustion of fuels as well as the mitigation of emissions. There are a number of new technologies that are becoming phased in order to control the production of diesel emissions. The exhaust gas recirculation system, E.G.R., and the diesel particulate filter, D.P.F., are both designed to mitigate the production of harmful emissions.(AOCS Official Method,1987)

While studying the effect of biodiesel on a D.P.F. it was found that though the presence of sodium and potassium carbonates aided in the catalytic conversion of ash, as the diesel particulates are catalyzed, they may congregate inside the D.P.F. and so interfere with the clearances of the filter. This may cause the filter to clog and interfere with the regeneration process,In a study on the impact of E.G.R. rates with blends of jathropa biodiesel it was showed that there was a decrease in fuel efficiency and torque output due to the use of biodiesel on a diesel engine designed with an E.G.R. system. It was found that CO and CO₂ emissions increased with an increase in exhaust gas recirculation but NOx levels decreased. The opacity level of the jathropa blends was in an acceptable range, where traditional diesel was out of acceptable standards. It was shown that a decrease in Nox emissions could be obtained with an E.G.R. system. This study showed an advantage over traditional diesel within a certain operating range of the E.G.R. system.(Bailey, 1951).

1.20Material Compatibility:

Plastics: High density polyethylene (HDPE) is compatible but polyvinyl chloride (PVC) is slowly degraded. Polystyrene is dissolved on contact with biodiesel.

Metals: Biodiesel (like methanol) has an effect on copper-based materials (e.g. brass), and it also affects zinc, tin, lead, and cast iron. Stainless steels (316 and 304) and aluminum are unaffected.

Rubber: Biodiesel also affects types of natural rubbers found in some older engine components. Studies have also found that fluorinated elastomers cured with peroxide and base-metal oxides can be degraded when biodiesel loses its stability caused by oxidation.

1.21FT-IR:

Oil was characterized by FT-IR, using a Perkin Elmer oil analyzer equipped with the MIR TGS detector in the range 4000 e 650 cm_1 and processed with the computer software program spectrum. Fig. 1 shows the Fourier transform infrared (FT-IR) spectrum of the oil from *Banalities aegyptiaca*seeds . The resolution was 4 cm_1 and 8 scans. The characteristics peaks of oil from *Banalities aegyptiaca* seeds.

1.22GC-MS:

The qualitative and quantitative analysis of the sample was carried out by using GM/MS technique model (GC/MS-QP2010-Ultra) from japans 'Simadzu

Company, with serial number 020525101565SA and capillary column (Rtx-5ms- $30m\times0.25$ mm $\times0.25\mu$ m). The samplewas injected by using split mode, helium as the carrier gas passed with flow rate 1.61 ml/min, the temperature program was started from 60c with rate 10c/min to 300c as final temperature degree , the injection port temperature was 300c, the ion source temperature was 200c and the interface temperature was 250c. The sample was analyzed by using scan mode in the range of m/z 40-500 charges to ratio and the total run time was 26 minutes .Identification of components for the sample was achieved by comparing their retention times and mass fragmentation patents with those available in the library ,the National Institute of Standards and Technology (NIST). , results were recorded.

1.23The Objective Of This StudyAre:

- To extract oil from *Balanitesaegyptiaca*.
- To examine the physiochemical characteristics of the extracted oil.
- To Convert the extracted seeds oil into biofuel bytransesterification reaction.
- To characterize the Biofuel using varies standardTechniques.
- To compare the properties of biofeul with normal diesel.

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Chapter Two Materials And Methods

Chapter Two

Materials and Methods

2.1 Materials:

.Banalities aegyptiaca seeds were cleaned to remove dirt, sun-dried for three days and the Seeds were expressed using cold pressed solvent of extraction in College Of Agriculture Studies sham bat.

2.2 Methods:

2.2.1 Solvent Extraction:

250 ml of n-hexane are placed into a round bottom flask. 100g of the sample was inserted in the center of the extractor. The Soxhlet was heated at 60 - 70C. When the solvent was boiling the vapor rose through the vertical tube into the condenser at the top. The liquid condensate dripped into the filter paper thimble in the center which contained the solid sample to be extracted. The extract seeped through the pores of the thimble and filled the siphon tube, where it flowed back down into the round bottom flask. This was allowed to continue for 4 hours. At the end of the extraction the condenser was removed.

The flask containing the resulting mixture was connected to Liebig condenser and heated up to 65 - 700**C** during which the n-hexane evaporated off and was recovered in a conical flask; the oil extracted weight was taken. This procedure was repeated throughout the work.

2.3Characterization of The Oil:

2.3.1Determination of The Percentage of Banalities aegyptiaca Oil:

The sample to be extracted, 10 g was placed in the thimble and 200 mL of n-hexane poured into the empty round bottom flask. The solvent was heated to 70c and allowed to heat for 3 h continuously while extracting the oil by Soxhlet apparatus the yield was 53.9%.

2.3.2 Moisture Content:

The moisture content was determined according of the Association of Official Analytical Chemists (AOAC,2008). Two grams were weighed into a pre-dried and

tarred dish. Then, the sample was placed into an oven (No.03-822, FN 400, Turkey) at 105 C±1 C until a constant weight was obtained. After drying, the covered sample was transferred to a desiccator and cooled to room temperature before reweighing. Triplicate results were obtained for each sample and the mean value was reported to three decimal points according to the following formula; Calculation:

Moisture content (%) =
$$W_1$$
- W_2 / Wt_1 X 100

Where;

W1= Sample weight before drying

W2 = Sample weight after drying

Wt1=initial sample weight

2.3.3 pH:

In a dry clean 25 mL beaker, 2 g of the sample was placed followed by 13 mL of hot distilled water and the mixture was stirred slowly. The mixture was then cooled in a cold-water bath to 25°C. The pH electrode was standardized with buffer solutions (pH 4 and 7) and the electrode immersed into the sample where an average pH of three recordings per sample were recorded.

Production of Detergent from seeds Oil (Isah, 2006).

2.4Determination of Physical Parameters:

2.4.1 Specific Gravity:

Density bottle was used in determining the specific gravity of the oil. A clean and dry stoppered bottle of 25 mL capacity was weighed (W0) and then filled with the oil stoppered and reweighed to give (W1). The oil was substituted with distilled water after washing and drying the bottle and weighed to give (W2). The expression for specific gravity (Sp.gr) is:

Sp.gr =
$$\frac{W_1 - W_2}{W_2 - W_0}$$

Where W_0 = weight of dry empty density bottle; W_1 = weight of density bottle + oil; W_2 = weight of density bottle + distilled water.

2.4.2 Viscosity of Oil:

The viscometer was suspended in a constant temperature water bath and was exactly filled to the mark at the top of the lower reservoir with the oil by means of a pipette inserted in the side arm, so that the tube wall above the mark is not wetted. The instrument was then left to stand for few minutes before reading in order to equilibrate the sample temperature with that of the instrument (35C).

By means of pressure on the respective arm of the tube, the oil moved into the other arm so that the meniscus is (1cm) above the mark at the top of upper reservoir. The liquid was then allowed to flow freely through the tube and the time required for the meniscus to pass from the mark above the upper reservoir to that at the bottom of the upper reserve was recorded. (Cocks and Rede, 1966).

2.4.3Refractive Index of Oil:

Refractive index was determined by Abbe-60 refrac meter as described where a double prism was opened by means of screw head and few drops of sample were placed on the prism. The prism was closed firmly by lighting screw head. The instrument was left to stand for few minutes before reading in order to equilibrate the sample temperature with that of the instrument. The prism was cleaned between readings wiping off oil with soft cloth, then with cotton moistened with petroleum ether and left to dry. The refractive indices of all samples were determined at 35-40C.

2.5Determination of Chemical Parameters:

2.5.1 Acid Value of Oil:

The oil or melted fat was mixed thoroughly before weighing 5 gm. of cooled oil sample was weighted accurately in a 250 ml conical flask and 50 to 100 ml of freshly neutralized hot ethyl alcohol and one ml of phenolphthalein indicator solution were added. The mixture was boiled for about five minutes and titrated while hot against standard alkali solution, shacked vigorously during the titration. The weight of the oil fat taken for the estimation and the strength of the alkali used for titration shall be such that the volume of alkali required for the titration does not exceed 10 ml.

Calculation

Acid value 56.1 xVxN

W

Where:

V = Volume in ml of the standard potassium hydroxide or

Sodium hydroxide used

N = Normality of the potassium hydroxide solution or sodium hydroxide solution

W = Weight in g of the sample

2.5.2 Peroxide Value of Oil:

The peroxide value (PV) of the oils was determined according to proceduer (Wail etal, 1995). About one gm of the sample was weighed into 250 ml conical flask, 30 ml of a glacial acetic acid/chloroform solution (3:2) were added, and the flask was' swirled until the sample was dissolved. A 0.5 ml of saturated potassium iodide was added. The solution was again swirled for one minute, 30 ml of distilled water were added and 0.5 ml of 1% starch solution were added. The contents of the flask were then titrated with 0.1 N sodium thiosuphate added gradually with constant and vigorous shaking and the titration was continued until the blue colour disappeared. A blank test was carried out. The number of 0.1 N sodium thiosulphate required was recorded.

Calculation:

$$PV = \underbrace{(V_{\underline{a}} - V_{\underline{b}}) \ xN}_{W}$$

Where:

Va= Volume of sodium thiosulphite solution used in titration

Vb= Volume of sodium thiosulphite solution used in blank test

W= Weight of sample in grams

N= Normality of sodium thiosulphate solution

2.5.3Saponification Number of Oil:

The determination of saponification number was carried out according to the AOAC (2008) method.

One gram of oil sample was weighed accurately in to 200 ml conical flask. 25 ml of 0.1N alcoholic KOH solution was added, and the contents of the flask were boiled under reflux for one hour with frequent rotation. one ml of phenolphthalein indicator was added, while the solution was still hot, and the excess alkali was titrated with 0.5N HCL. The numbers of ml of HCl required (a) were recorded. The same process was repeated without oil and the numbers of ml of HCl (b) were also recorded.

Calculation:

Saponification Number= $(b-a) \times 28.05 \times 100$

W

Where:

a = ml of HCl from sample.

b = ml of HCl from blank.

w = weight of oil in gram.

2.5.4 Iodine Value of Oil:

0.2 grams of oil was accurately weighed and placed in a dry and clean flask specially offered for the test .A 10 ml of chloroform was used for dissolving the oil . A 25 ml of pyridine sulphate dibromide solutions was added and finally 20 ml of KI (0.1 N) were added to the contents of the flask was then stoppered and the mixture was allowed to stand for 10 minutes in a dark place . The stopper and the side of the flask were rinsed with enough amount of distilled water , the contents of the flask was then stoppered and the mixture was allowed to stand for 10 minutes in a dark place .The stopper and the side of the flask were then shaken and titrated against 0.1N sodium thiosulphate solution using starch liquid as indicator . A blank determination was carried out simultaneously .

Calculation:

Iodine value (IV) = $(b-a) \times 0.01269 \times 100$

W

Where:

b: Volume (ml) of sodium thiosulphate in blank solution

a:Volume (ml)of sodium thiosulphate in test active solution

W: Weight (gm) of the oil sample

2.5.5Free Fatty Acids of Oil:

5 grams of the oil were weighed accurately into 250 ml conical flask. 50 ml mixture of 95% alcohol and ether solvent (1:1) were added. The solution was neutralized after addition of one ml of phenolphthalein indicator. The contents of the flask were then heated with caution until the oil was completely dissolved .The contents of the flask were then titrated with 0.01N KOH with constant shaking until a pink colour persisted for 15 seconds .The number of ml of 0.1 N KOH recorded as % .

Calculation:

Free fatty acid as (% oleic acid) = \underline{a} (ml of KOH) x N x 56.1

W

Where:

a. volume of KOH (ml).

N : Normality of KOH

W: weight of sample.

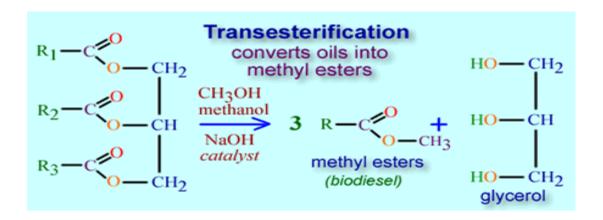
2.6 FT-IR Analysis:

Oil was characterized by FT-IR, using a Perkin Elmer oil analyzer equipped with the MIR TGS detector in the range 4000 - 650 cm_1 and processed with the computer software program spectrum. Fig. 1 shows the Fourier transform infrared (FT-IR) spectrum of the oil from *Banalities aegyptiaca* seeds. The resolution was 4 cm⁻¹ and 8 scans. The characteristics peaks of oil from *Banalities aegyptiaca* seeds.

2.7Experimental:

2.7.1 Transesterification Process:

200ml of the oil sample was put into a beaker; the methoxide was added to it. The temperature was raised to 450C on a hot plate and the mixture was stirred with magnetic striver for 25 minutes. The mixture was allowed to stand overnight in a flask. A thick brown liquid layer settled at the bottom, this was the glycerin and the lighter coloured liquid above was the biodiesel. The glycerin which is the byproduct was drawn off through the bottom tap. The biodiesel was then washed with warm water, soaps and methanol that are in the biodiesel. The water is better to dissolving the impurities than the biodiesel. A straw-yellow and a cloudy liquid layer were formed, the cloudy liquid was drained out and pH test was conducted to confirm the neutrality of the washed water/cloudy liquid. The biodiesel was heated to 550C on a hot plate to allow the remaining water to evaporate and was finally filtered using a filter paper (Mondala etal, 2009).



Scheme 2.1 Transestification Reaction

2.7.2 Determination 0f The Density:

0.7 mL of liquid sample was introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample according to (ASTM D4052).

2.7.3 Determination of the Viscosity:

The test specimen is introduced into the measuring cells, which are at a closely controlled and known temperature. The measuring cells consist of a pair of rotating concentric cylinders

and an oscillating U-tube. The dynamic viscosity is determined from the equilibrium rotational speed of the inner cylinder under the influence of the shear stress of the test specimen and an eddy current brake in conjunction with adjustment data. The density is determined by the oscillation frequency of the U-tube in conjunction with adjustment data. The kinematic viscosity is calculated by dividing the dynamic viscosity by the density according to (ASTM D7042).

2.7.4 Determination of The Cloud point:

Specimen is cooled by a Peltier device at a while continuously being illuminated by a light source. The specimen is continuously monitored by an array of optical detectors for the first appearance of a cloud of wax crystals. The detectors are sufficient in number to ensure that any solid phase hydrocarbon crystals that may form are detected. The temperature at which the appearance of a cloud of wax crystals is first detected in the specimen is recorded to 0.1°C resolution. When the recorded temperature is rounded to the next lower integer temperature, it is designated as the D 2500/IP 219 equivalent cloud point according to (ASTM D5773).

2.7.5 Determination of the Copper Corrosion:

A polished copper strip is immersed in a specific volume of the sample being tested and heated under conditions of temperature and time that are specific to the class of material being tested. At the end of the heating period, the copper strip is removed, washed and the color and tarnish level assessed against the ASTM Copper Strip Corrosion Standard according to (ASTM D130).

2.7.6 Determination of the Flash point:

A brass test cup of specified dimensions, filled to the inside mark with test specimen and fitted with a cover of specified dimensions, is heated and the specimen stirred at specified rates, An ignition source is directed into the test cup at regular intervals with simultaneous interruption of the stirring, until a flash is detected. according to (ASTM D93).

2.7.7 Determination of the Color:

Using a standard light source, a liquid sample is placed in the test container and compared with colored glass disks ranging in value from 0.5 to 8.0. When an exact match is not found and the sample color falls between two standard colors, the higher of the two colors is reported according to (ASTM D1500).

2.7.8Determination of the Cetane Index:

Two correlations in SI units have been established between the ASTM cetane number and the density and 10 %, 50 %, and 90 % distillation recovery temperatures of the fuel according to (ASTM D4737).

2.7.9 Determination of the Sulfur m/m%:

A hydrocarbon sample is either directly injected or placed in a sample boat. The sample or boat, or both, is inserted into a high temperature combustion tube where the sulfur is oxidized to sulfur dioxide (SO2) in an oxygen rich atmosphere.

Water produced during the sample combustion is removed and the sample combustion gases are next exposed to ultraviolet (UV) light. The SO_2 absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO_2 *). The fluorescence emitted from the excited SO_2 * as it returns to a stable state, SO_2 , is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

(Warning—Exposure to excessive quantities of ultraviolet (UV) light is injurious to health. The operator must avoid exposing any part of their person, especially their eyes, not only to direct UV light but also to secondary or scattered radiation that is present according to (ASTM D5453).

2.7.10 Determination of the FT-IR:

oil was characterized by FT-IR, using a Perkin Elmer oil analyzer equipped with the MIR TGS detector in the range 4000 - 650 cm_1 and processed with the computer software program spectrum. Fig. 1 shows the Fourier transform infrared (FT-IR) spectrum of the oil from *Banalities aegyptiaca* seeds . The resolution was 4 cm_1 and 8 scans. The characteristics peaks of oil from *Banalities aegyptiaca seeds*.

2.7.11Determination of the GC-MS:

The qualitative and quantitative analysis of the sample was carried out by using GM/MS technique model (GC/MS-QP2010-Ultra) from japans 'Simadzu Company, with serial number 020525101565SA and capillary column (Rtx-5ms-30m×0.25 mm×0.25 µm). The sample was injected by using split mode, helium as the carrier gas passed with flow rate 1.61 ml/min, the temperature program was started from 60C with rate 10c/min to 300C as final temperature degree , the injection port temperature was 300C, the ion source temperature was 200C and the interface temperature was 250C. The sample was analyzed by using scan mode in the range of m/z 40-500 charges to ratio and the total run time was 26 minutes . Identification of components for the sample was achieved by comparing their retention times and mass fragmentation patents with those available in the library ,the National Institute of Standards and Technology (NIST). , results were recorded.

Chapter Three Results And Discussions

Chapter Three

3.1. Results and Discussions

Table 1 as shows the physicochemical properties of *Balanites aegybtiaca* seeds oil, the results shows:

Table (1.3). Properties of Balanites aegybtiaca seeds oil

Properties	value
% Yield	53.9
Moisture content (%)0.27%	
pH 5.84	
Density (kg/m ³)	0.88(at 40 C)
Viscosity (cp)	25.87
Refractive Index	1.475
Colour Intensity	Yellow(2.6) red(1.1)
Fat content (%w/w)	54.7
Free Fatty Acid (FFA) value (%)	1.067
Peroxide value meq/kg	6.00
Acid value mgKOH/g	2.1
Saponification value mgkoH/g oil	239.35
Iodine value i ₂ /100 g oil	74.34

The phyicochemical properties of *Balanites aegyptiaca* seed oil are shown in Table (1)the saponification value of the oil is 239.35 mgKOH/g. The iodine value (IV) is relatively low, thus the oil is non-drying. The peroxide value is also very low, indicating that the oil would be stable (to a large extent), to oxidative degradation. Noticeable when the peroxide value reaches 6 meq/kg (Charles and Guy, 1991). The low %FFA reduces the tendency of the oil to undergo hydrolytic activities. In most oils, the level of free fatty acid which causes deterioration is noticed when the %FFA calculated as oleic acid falls within the range of 0.5 - 1.5% (Rossel, 1971).

The oil yield was found to be 53.9% indicating that the oil content is high, a factor that is favorable for industrial application of the oil. The moisture content of the oil is very low, therefore its stability is guaranteed. The oil has yellow colour, and it remained liquid at room temperature, thus adding to the good qualities required industrial oil raw material. The refractive index of the oil also has fallen within the same range with other vegetable oils. The pH of the *Balanities aegyptiaca* oil is (5.84) that mean the sample has acidic properties.

Table (2.3) Properties of Biodiesel and Blends:

Properties	Diese	Biodiesel	Disesl+biodiesel	Diesel+biodesel+ethanol
	1			(%)
Viscosity@40C	3.351	5.041	3.855	3.011
	1			
Viscosity@100	1.307	1.916	1.702	1.05
C				
Density @15 C	0.841	0.885	0.848	0.845
Cloud point	5.1	13.5	5.6	15.7
Copper	1a	1a	1a	1a
corrosion				
Flash point C	77.0	130	77.5	40.5
Color index	0.5	0.7	0.7	0.5
Cetane number	53.9	54.6	55.2	54.6
Sulfur m/m%	0.011	0.011	0.011	0.011

The properties of biodiesel are include; cetane number (54.6) A higher cetane namber indicates shorter time between the ignition and the initiation of fuel injection into the combustion chamber (Atabanietal, 2012). Biodiesel has higher cetane number than diesel fuel, which results in higher combustion efficiency (Atabani,tal,2013). The CN of diesel is(53.9).the viscosity of biodiesel @ 40C(5.041mm²/s), at 100C(1.916) this degrees the viscosity of Biodiesel is higher than diesel fuel that means Viscosity is the most important property of any fuel as it indicates the ability of a material to flow. The Cloud point is measured using ASTM D2500 EN ISO 23015 and D97 procedures. Generally, Biodiesel has higher CP compared to conventional diesel (Haal,1992). It used as indicator of low temperature operability of fuels and reflects their cold weather performance. Measured using a low operating temperature fuel maybe thicken and might not flow properly affecting the performance of fuel lines, fuel pumps and injectors. The flash point of biodiesel is (130C), Flash point (FP) Flash point of a fuel is the temperature at which it will ignite when exposed to a flame or a spark. Flash point varies inversely with the fuel's volatility. The flash point of biodiesel is higher than the prescribed limit of diesel fossil fuel, which is safe for transport, handling and storage purpose Balanites aegyptiaca.

The copper corrosion test measures the corrosion tendency of fuel when used with copper, brass, or bronze parts. Corrosion resulting from biodiesel might be induced by some sulfur compounds by acids; hence this parameter is correlated with acid number. The copper corrosion is 1a acceptable value.

3.2FT-IR of Oil:

The FT-IR spectra for sample of the *Balanities aegyptiaca* oil in the region from 3006.65 cm⁻¹it can be observed peaks that can be attributed to the stretching of C-H, and the stretching C=O typical of ester thus are common in FT-IR in rang 1745.56 cm⁻¹, in range 2925.35 and 2856.85 cm⁻¹ stretching of C-H from alkane CH₃ also the range 1457.26 cm⁻¹ it C-H bending for CH₂.

Table (3.3)Spectrum Peaks of Oil Seeds Ffrom Balanites aegyptiaca in FT-IR Spectra:

Absorption bands (cm ⁻¹)	Functional group	
3006.65	C-H Stretching vibration	
2925.35	C-H for CH ₃	
2856.85	C-H for CH ₃ symmetric	
1745.56	C=O for ester	
1457.26	C-H bending for CH ₂	
1162.43	C-C Stretching vibration	
721.58	(CH ₂)n	

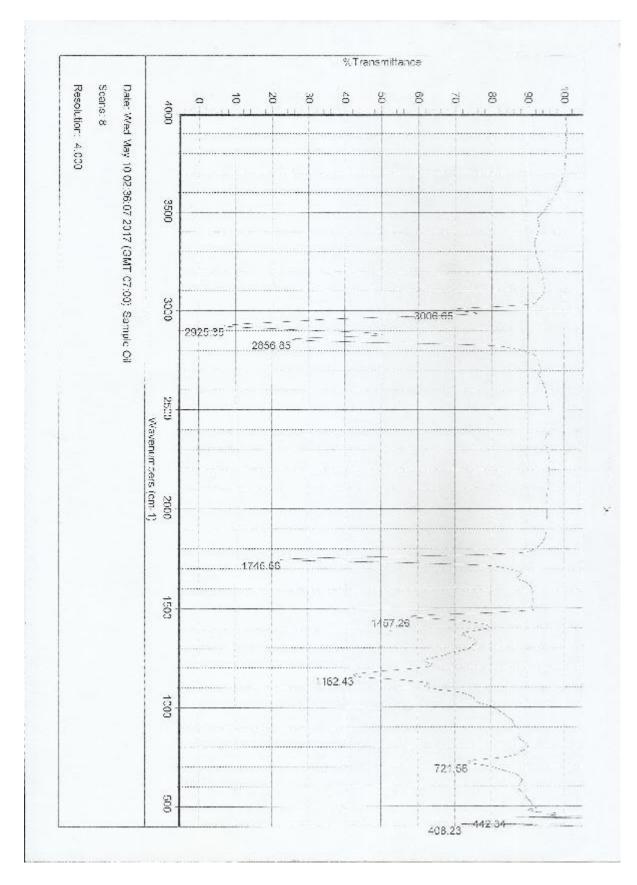


Fig (1.1) FT-IR of the oil from Balanites aegyptiaca seeds

Table(4.3). CharacterizingPeaks of Balanites aegyptiaca Biodieselin FT-IRSpectra:

Absorption bands (cm ⁻¹)	Functional group	
3006.30	C-H Stretching vibration	
2925.65	C-H for CH ₃ antisymetric	
2856.99	C-H for CH ₃ symmetric	
1743.43	C=O for ester	
1452.52	C-H bending for CH2	
1363.91	C -CH3 bending	
1171.08	C-C Stretching vibration	
1018.84	C-C Stretching vibration	
871.61	-(CH2)n- rock	
721.49	(CH2) rock	

Balanites aegyptiacabiodiesel was characterized by FT-IR, using a Perkin Elmer BiodieselFAME analyzer equipped with the MIR TGS detector in the range 4000-650 cm⁻¹ and processed with the computer software program spectrum. Fig. 2 shows the Fourier transform infrared (FT-IR) spectrum of the *biodiesel*. The resolution was 4 cm⁻¹ and 8 scans. The characteristics peaks of the *Balanities aegyptiaca* biodiesel are shown in Table 3.

The FT-IR spectra for sample of the *Balanities aegyptiaca* in the region from 3006.30 cm⁻¹it can be observed peaks that can be attributed to the stretching of C-H and the stretching C=O typical of ester thus are common in FT-IR in rang 1745.56 cm⁻¹, in range 2925.35-2856.85 cm⁻¹ stretching of C-H from alkane CH₃ also the range 1457.26 cm⁻¹ it C-H bending for CH₂, the other bands in the table3. Figer 4 characterizing peaks to *Balanites aegyptiaca* of biodiesel in FT-IR spectra

Figer 4 characterizing peaks to *Balanites aegyptiaca* of biodiesel in FT-IR spectra some of the fictional group C-H (3006.30), C=O(1743.43) and C-C(1171.08) stretching.

Figer 2 FT-IR of biodesel of Balanites aegyptiaca seeds.

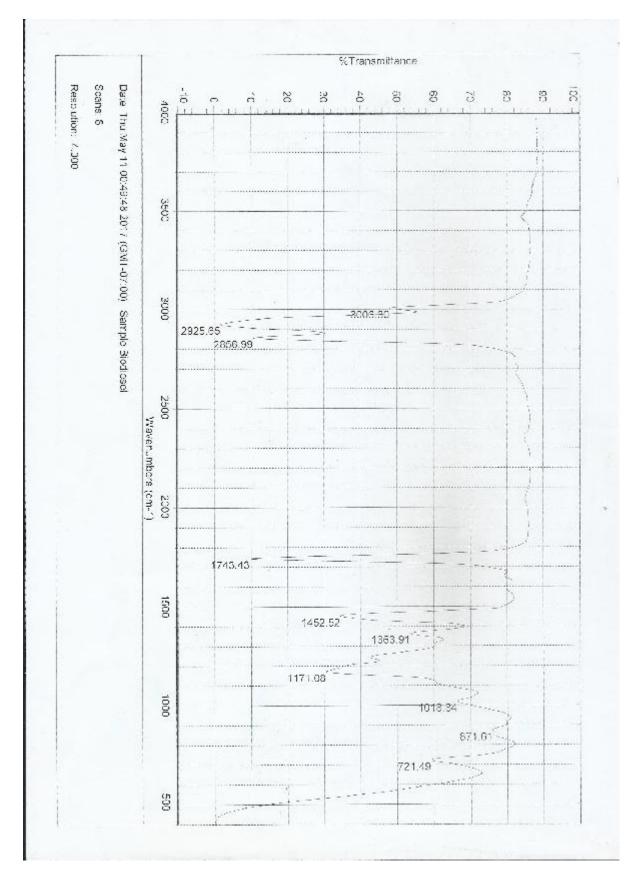


Fig (2.3) FT-IR of Biodesel from Balanites aegyptiaca seeds

3.3GC-MS AnalysisofBiodiesel:

Biodieselsample were analyzed using PerkinElmer GC –MS in total scan mode to identify the fatty acid methyl ester composition.

The components in the sample such as hydrocarbons —ester —phenol — alcohol and others. These components 80% is unsaturated and the 20% is saturated. The major components are 9,12- Octadecadienoic acid ,Hexadecanoic acid,methyl ester, 9-Octadecenoic acid methyl ester , methyl stearate, Cyclopropaneoctanoic acid and Eicosanoic acid methyl ester constitute about 95%.

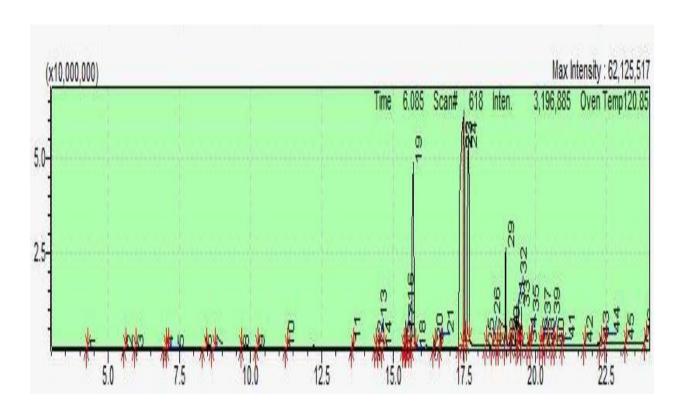


FIG (3.3) GC-MS of Biodesel

Peak Re	port TIC
A mag 0/	

			Peak Re	port TIC
Peak#	R.Time	Area	Area%	
1	4.243	120562	0.01	Undecane
2	5.579	111927		Dodecane
3	5.940	339017		Octanoic acid, methyl ester
4	6.984	282278	0.02	LalphaTerpineol
5	7.079	31699		Bicyclo[3.1.0] hexane, 6-isopropylidene-1-
6	8.352	379680	0.03	2,4-Dodecadienal, (E,E)-
7	8.676	290934	0.03	2,4-Decadienal, (E,E)-
8	9.669	51915	0.00	Tetradecane
9	10.209	83031	0.01	Nonanoic acid, 9-oxo-, methyl ester
10	11.259	89301	0.01	Dodecanoic acid, methyl ester
11	13.573	2698373	0.23	Methyl tetradecanoate
12	14.382	106307		6-Octadecenoic acid, methyl ester, (Z)-
13	14.488	86242	0.01	cis-5-Dodecenoic acid, methyl ester
14	14.647	207259	0.02	Pentadecanoic acid, methyl ester
15	15.379	194855	0.02	7,10-Hexadecadienoic acid, methyl ester
16	15.438	1187719	0.10	7-Hexadecenoic acid, methyl ester, (Z)-
17	15.483	6430990	0.55	9-Hexadecenoic acid, methyl ester, (Z)-
18	15.578	251714	0.02	trans-13-Octadecenoic acid, methyl ester
19	15.724	182714764	15.71	Hexadecanoic acid, methyl ester
20	16.447	3115125	0.27	cis-10-Heptadecenoic acid, methyl ester
21	16.657	6306153	0.54	Hexadecanoic acid, 15-methyl-, methyl es
22	17.470	451745454	38.85	9,12-Octadecadienoic acid (Z,Z)-, methyl
23	17.503	172641974	14.85	9-Octadecenoic acid (Z)-, methyl ester
24	17.664	193015411		Methyl stearate
25	18.271	778541		cis-10-Nonadecenoic acid, methyl ester
26	18.490	538164	0.05	Nonadecanoic acid, methyl ester
27	18.636	658322	0.06	Methyl 9.cis.,11.trans.t,13.transoctadeca
28	18.749	657506	0.06	.gammaLinolenic acid, methyl ester
29	18.960	48126795	4.14	Cyclopropaneoctanoic acid, 2-[[2-[(2-ethy
30	19.151	4793828	0.41	cis-11-Eicosenoic acid, methyl ester
31	19.352	18566946		Eicosanoic acid, methyl ester
32	19.407	20188920	1.74	PGH1, methyl ester
33	19.517	17226909	1.48	9,12-Octadecadienoyl chloride, (Z,Z)-
34	19.754	1180304	0.10	Methyl 2-octylcyclopropene-1-octanoate
35	19.849	6288033	0.54	Tricyclo[20.8.0.0(7,16)]triacontane, 1(22)
36	20.180	451651	0.04	Heneicosanoic acid, methyl ester
37	20.267	206263	0.02	Phenol, 2,2'-methylenebis[6-(1,1-dimethy
38	20.390	1057126	0.09	2-Octylcyclopropene-1-heptanol
39	20.594	1719956	0.15	Trilinolein
40	20.724	735485		Hexanoic acid, octadecyl ester
41	20.966	2988674	0,26	Docosanoic acid, methyl ester
42	21.730	724701	0.06	Tricosanoic acid, methyl ester
43	22.293	4441467	0.38	Propyleneglycol monoleate
44	22.469	4872697	0.42	Tetracosanoic acid, methyl ester
45	23.181	1455624	0.13	Pentacosanoic acid, methyl ester
46	23.868	2708113	0.23	Hexacosanoic acid, methyl ester
40	25.000	1162848709	100.00	

3-4.Conclusions:

From the work conducted, the produced biodiesel fuel was within the recommended standards, and therefore, it can be used for any required purpose.

- The oil yield was found to be 53.9% indicating that the oil content is high, a factor that is favorable for industrial application of the oil.
- *Balanities aegyptiaca*seeds continue high free fatty acids, when composition it can be as alternative fuel in diesel engines.
- We recommended to use biodiesel because diesel engines operated on biodiesel have lower emissions of carbon monoxide and air toxins that when operated on petroleum based diesel fuel.

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APPENDICES

Peak Re	eport TIC
Area%	Name

Peak Report TIC					
Peak#	R.Time	Area	Area%		
1	4.243	120562		Undecane	
2	5.579	111927		Dodecane	
3	5.940	339017		Octanoic acid, methyl ester	
4	6.984	282278		LalphaTerpineol	
5	7.079	31699	0.00	Bicyclo[3.1.0]hexane, 6-isopropylidene-1	
6	8.352	379680	0.03	2,4-Dodecadienal, (E,E)-	
7	8.676	290934		2,4-Decadienal, (E,E)-	
8	9.669	51915	0.00	Tetradecane	
9	10.209	83031	0.01	Nonanoic acid, 9-oxo-, methyl ester	
10	11.259	89301	0.01	Dodecanoic acid, methyl ester	
11	13.573	2698373	0.23	Methyl tetradecanoate	
12	14.382	106307	0.01	6-Octadecenoic acid, methyl ester, (Z)-	
13	14.488	86242	0.01	cis-5-Dodecenoic acid, methyl ester	
14	14.647	207259		Pentadecanoic acid, methyl ester	
15	15.379	194855		7,10-Hexadecadienoic acid, methyl ester	
16	15.438	1187719	0.10	7-Hexadecenoic acid, methyl ester, (Z)-	
17	15.483	6430990		9-Hexadecenoic acid, methyl ester, (Z)-	
18	15.578	251714		trans-13-Octadecenoic acid, methyl este	
19	15.724	182714764		Hexadecanoic acid, methyl ester	
20	16.447	3115125	0.27	cis-10-Heptadecenoic acid, methyl ester	
21	16.657	6306153	0.54	Hexadecanoic acid, 15-methyl-, methyl	
22	17.470	451745454	38.85	9,12-Octadecadienoic acid (Z,Z)-, methy	
23	17.503	172641974	14.85	9-Octadecenoic acid (Z)-, methyl ester	
24	17.664	193015411		Methyl stearate	
		778541		cis-10-Nonadecenoic acid, methyl ester	
25	18.271	538164	0.07	Nonadecanoic acid, methyl ester	
26	18.490	658322	0.03	Methyl 9.cis.,11.trans.t,13.transoctaded	
27	18.636		0.00	.gammaLinolenic acid, methyl ester	
28	18.749	657506	4.14	Cyclopropaneoctanoic acid, 2-[[2-[(2-etl	
29	18.960	48126795	0.41	cis-11-Eicosenoic acid, methyl ester	
30	19.151	4793828	1.60	Eicosanoic acid, methyl ester	
31	19.352	18566946			
32	19.407	20188920		PGH1, methyl ester	
33	19.517	17226909		9,12-Octadecadienoyl chloride, (Z,Z)-	
34	19.754	1180304	0.10	Methyl 2-octylcyclopropene-1-octanoate	
35	19.849	6288033	0.54	Tricyclo[20.8.0.0(7,16)]triacontane, 1(22	
36	20.180	451651	0.04	Heneicosanoic acid, methyl ester	
37	20.267	206263	0.02	Phenol, 2,2'-methylenebis[6-(1,1-dimeth	
38	20.390	1057126		2-Octylcyclopropene-1-heptanol	
39	20.594	1719956		Trilinolein	
40	20.724	735485	0.06	Hexanoic acid, octadecyl ester	
41	20.966	2988674	0.26	Docosanoic acid, methyl ester	
42	21.730	724701	0.06	Tricosanoic acid, methyl ester	
43	22.293	4441467	0.38	Propyleneglycol monoleate	
44	22.469	4872697	0.42	Tetracosanoic acid, methyl ester	
45	23.181	1455624	0.13	Pentacosanoic acid, methyl ester	
46	23.868	2708113	0.23	Hexacosanoic acid, methyl ester	
		1162848709	100.00		