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Contribution to the preparation of biofuels of vegetable origin (sunflower oil), case of the region of Ouargla

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Dedication

To the person who taught me perseverance, patience, and generosity; my father may Allah protect him.

To the person who taught me, and went through a lot all the way long; the one who her praying is the secret of my success, my mother may Allah heals her.

To my brother Salah and sister Karima and Hafida; my support through the journey. To all the members of both families of Tekha and Ben Lattet.

To my friends Khaoula, Lalla, Razika and Rabia.

To all my classmates.

To my friend Ibrahim Bekkicha, for helping me throughout the journey.

To every person who had convinced in idea and called for it, and Worked on implementing it, only for the sake of Allah and for the greater good: I dedicate this humble work for you.

TEKHA Nesrine



Dedication

To the person who taught me perseverance and patience, to the person who raised me to be generous; my father may Allah have mercy him.

To the person who told me and encouraged me to reach the person who I am right now; the person who her praying are the secrets of my success and her kindness is the cure of my wounds; to my mother may Allah heal her as soon possible.

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To those who believed in an idea and stood for it and worked hard to implement it only for the sake of Allah and for the greater: I dedicate this humble work for you.

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List of Abbreviations

AN	Acid number
C.A	Codex Alimentarius
CaO	Calcium oxide
CH₃ONa	Sodium methoxide
CO₂	Carbon dioxide
CN	Cetane number
CV	Conventional
DG	Diglycerides
E .S	European Standard
EU	European Union
FAME	Fatty acid methyl ester
FFA	Free fatty acid
GL	Glycerol
GC-MS	Gas chromatography coupled with a mass spectrometer
HCl	Hydrochloric acid
IN	Iodine number
KOH	Potassium hydroxide
MG	Monoglycerides
MgO	Magnesium oxide
NaOH	Sodium hydroxide
NO₂	Nitrogen dioxide
Mo₃	Molybdenum trioxide
SN	Saponification number
SO₂	Sulfur dioxide
t	Time
T	Temperature
TG	Triglycerides
USA	United States of America
WCO	Waste cooking oil
Y	Yield
ZnO	Zinc oxide
ZrO₂	Zirconium dioxide

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**GENERAL
INTRODUCTION**

Introduction

Recently, the production of clean natural energy sources has gained a significant increase. The interest is due to the increase in energy consumption, the volatility of environmental conditions, and the ever-increasing decrease in supplies of available fuel generation. Depletion of non-renewable resources is at an all-time high, and they will soon be exhausted. Fossil fuel combustion also releases harmful pollutants into the environment, which leads to a wide variety of other problems.

To deal with these issues, scientists around the world are focusing their attention on this Issue. Biodiesel, a first-generation biofuel that is popular as an alternative to petroleum-based diesel, is growing in popularity around the world. In addition to biodiesel's biodegradability, technological feasibility, reduction of greenhouse gas emissions, non-toxicity, and carbon neutrality, biodiesel has many benefits **(Ayesha Hameed., and all.2022)**, as well as its ability to replace fossil fuels and meet growing energy demand. Many countries such as the USA, Canada, and France, they put in place many policies to reduce the use of energy based on fossil fuels and emissions of greenhouse gases and promote the production of biofuels from renewable sources such as biomass, energy crops, as well as domestic and industrial waste. **(Rahul Saini., and all.2021)**

According to the biodiesel production estimate, approximately 80% of the total cost is shared by the biodiesel feedstock, a blend of fatty acid methyl esters (FAME) consisting of oil raw materials such as vegetable oils, inedible oils, waste cooking oil and animal fats, waxes can be used without much change in diesel engines. Biodiesel development typically requires homogeneous catalysts such as (NaOH, KOH) on a commercial scale, resulting In a high rate of reactions and reasonably mild conditions; However, these catalysts cannot be recovered or separated and dealt with by a costly series of filtration, recycling, and separation procedures that produce large amounts of wastewater, degradation of facilities, operating costs, and high energy use. Advantages and efficiencies of heterogeneous catalysts over homogeneous catalysts in esterification reaction have been documented to solve commonly such problems because they are less toxic, more stable, easier to isolate, and environmentally friendly in nature. **(Ayesha Hameed., and all.2022)**

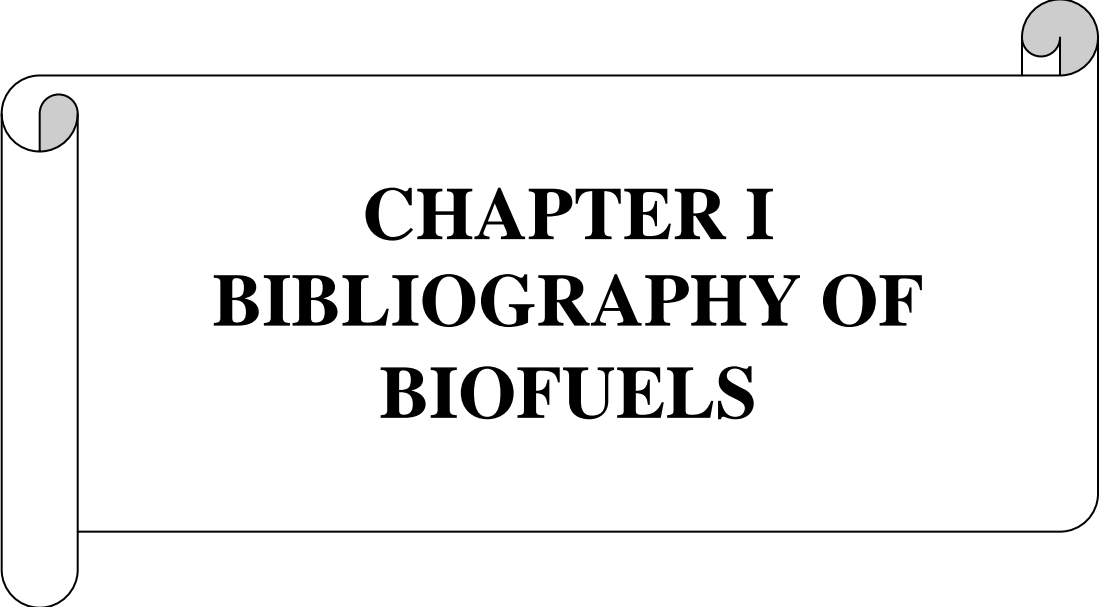
In this work, we aim to evaluate some low-value or harmful wastes and materials to convert the environment into green (bio) fuels of high quality by the modern method (we depend transesterification method). For this purpose, we use Waste Cooking Oil (WCO) to produce

INTRODUCTION GENERAL

biodiesel because it is depleted in nature...and so forth. Putting these worthless materials into consumption cycle by producing green fuel finally, we checked the quality of the product by analyzing and comparing physical and chemical properties by international standards.

This thesis is subdivided into following chapters:

- **Introduction**
- **The First Chapter:** Bibliography of biofuels
- **The Second Chapter:** Material and method
- **The third Chapter:** Result and discussion
- **Conclusion**



**CHAPTER I
BIBLIOGRAPHY OF
BIOFUELS**

I.1. Introduction

Renewable energies contribute effectively to achieving sustainable development the human side does not have any negative impact on the environment, and this is what contributes to preserving it.

Renewable energy is represented by solar energy, wind energy, hydropower, energy biomass, and other, which is an energy that does not run out from the planet earth, unlike the traditional energy, such as: oil, gas and other.

Biofuels can make a significant contribution in the short to medium term range, contribute to energy independence.

I.2. Biofuel

It can be defined as liquid fuels produced from biomass for either transport or burning purposes. They can be produced from agricultural and forest products and the biodegradable portion of industrial and municipal waste. **(Fatma Zohra Ben chikh, 2022)**

I.2.1. Biofuel generation

Biofuels for transport are commonly addressed according to their current or future availability as first, second, third or fourth generation biofuels. Second and third generation biofuels are also called "advanced" biofuels. **(Kaushlesh Kumar Yadav, 2018)**

I.2.1.1. First-generation biofuels

First generation biofuels are produced commercially from food crops using traditional technology. Provided by crops such as sugar cane, rapeseed, sunflower seed or oil palm, these plants were originally selected as food or fodder, and most are still used mainly to feed people. The most common first-generation biofuel is bioethanol (currently over 80% of liquid biofuel production by energy content), followed by biodiesel, and then bioga.

I.2.1.2. Second-generation biofuels

Second-generation biofuels can be produced from a variety of non-food and food sources such as seeds, grains, or whole plants from crops such as maize.

The starchy or cellulosic materials are converted into reducing sugars which are used instead as carbon. Second generation biofuels are produced through the use of liquid technology, by thermochemical conversion of biomass. It is mainly used to produce biodiesel.

I.2.1.3. Third-generation biofuels

Algae fuel is a kind of biofuel from green algae and cyanobacteria. Microalgae are feedstocks from hydroponics and are rich sources of triglycerides (from algal oil) for biodiesel production. The process technology is basically the same as that of biodiesel from second generation feedstock.

I.2.1.4. Fourth generation biofuels

It includes genetically manipulated micro – organisms and algae that can produce higher amounts of biofuel feedstocks, or can generate commodity chemicals of biofuel interests through introducing genes for whole metabolic pathways in their genomes or by introducing plasmids harboring the respective genes. A lot of genetically manipulated cyanobacteria, bacteria and algae can produce enhanced amounts of lipids and carbohydrates, or can produce ethanol, butanol etc. These correspond to fourth generation biofuels.

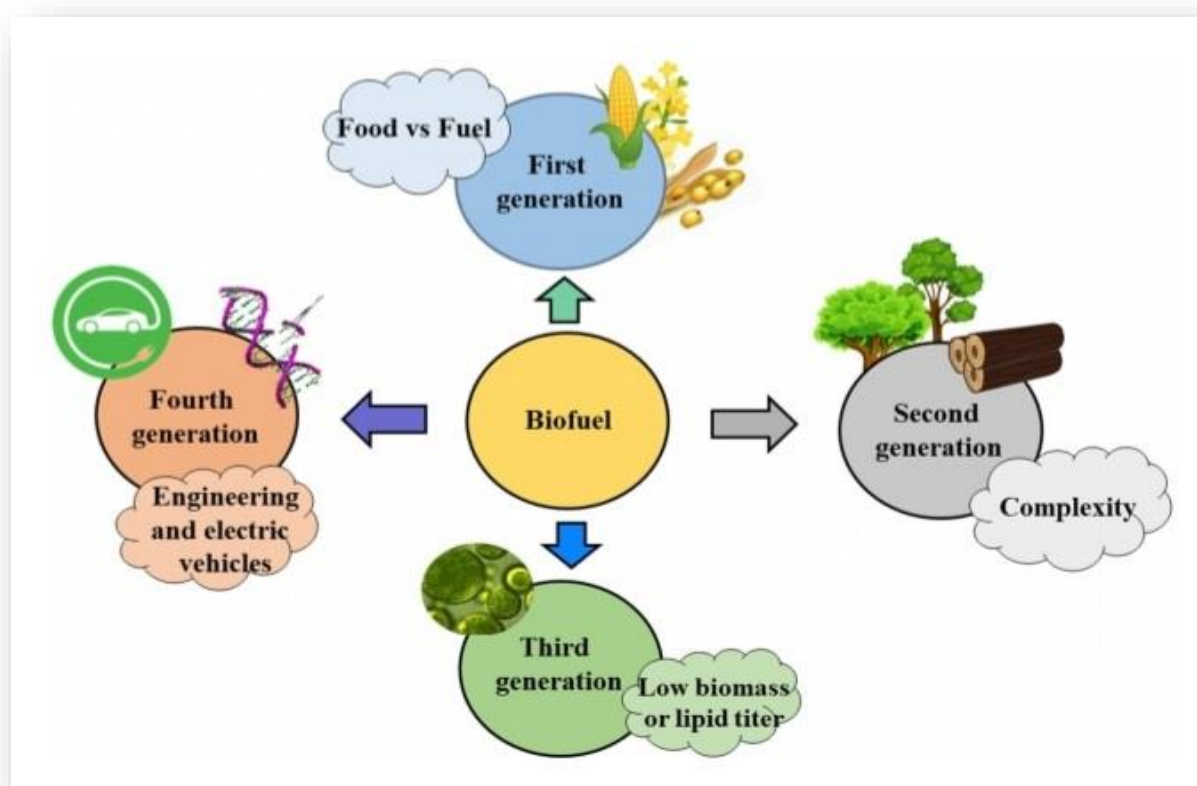


Figure I.1: Different generation of biofuels (Rahul Saini., and all.2021)

I.2.2. The advantages and disadvantages of biofuels

I.2.2.1. Advantages of biofuels

There are several benefits that contribute to the biofuel sector's growing importance to energy markets. Looking at the pros and cons of biofuels, the following are five primary benefits **(Electronic Reference 12)**.

Maintenance Costs:

Many biofuels are similar in cost to gasoline.

However, from an environmental perspective, biofuels are cleaner and produce lower carbon emissions. Biofuel also aids in reducing greenhouse gases (GHG emissions) and combating global warming.

Sourcing:

Global energy demand is always on the rise. Current oil and gas reservoirs will be of value for many more years, but the world's fossil fuel supply is finite and our dependence on fossil fuels must come to an end.

Renewability:

Biofuel is derived from living organisms like plants and animals. They can also exist in dead or recently living plants, crops, shrubs, grass, and even animal waste .More biodiesel products can be made available from different plant oil materials and waste oil.

Carbon Neutrality:

Another advantage of biomass energy is its use of the planet's natural carbon cycle.

Plants photosynthesize carbon from the atmosphere. When biofuels are burned, they release carbon back into the atmosphere. Hence, plants absorb an equal amount of carbon as to what biofuels can emit.

The Economic Factor:

Hydrocarbon energy sources are not available in every country.

Thus many locations around the world are reliant on importing oil and gas products. The importation cost can be offset by the adoption of biofuels. This aids economic independence, increasing employment opportunities and reduces reliance on foreign oil and gas.

I.2.2.2. Disadvantages of biofuels

Many view biofuels as a viable option for a cleaner and more cost-effective form of energy production. Despite the many advantages of biofuels, there are also several disadvantages to consider.

These include: **(Electronic Reference 12)**

Long-Term Costs:

While maintenance costs of biofuel can be lower, the production costs remain high.

B Biofuel Monoculture:

Monoculture is when the same crop is repeatedly grown in the same location. The major problem of biofuel mass utilization is the lack of plant genetic diversity.

Fertilizer Use and Biofuels:

Fertilizers are used for crops to grow bigger and faster.

Fertilizers can be very harmful to the land and surrounding environment, animals, and people. Fertilizers have also been proven to contribute to water pollution. This is especially true of fertilizers that contain high levels of phosphorus and nitrogen.

Food vs. Fuel Sources:

There is an ongoing debate about which takes priority, food or fuel sources? As the global population grows, so does the demand for available resources. For instance, not all lands are used for agricultural and livestock production. Some agricultural lands are converted for energy crop production.

This raises some major concerns about its potential impact on the food supply chain. Increased use of biofuels may result in increased food prices.

Pollution and Water Use:

Biofuels have a smaller carbon footprint than fossil fuels during consumption. Still, biofuel crop production requires large amounts of both oil and water resources.

I.2.3. Effects of biofuels on the environment

A study indicates that biofuels have a positive impact on the environment in contrast to fossil fuels, whose combustion leads to the production of a high percentage (72%) of carbon dioxide and greenhouse gases (CO₂, NO₂, SO₂), unlike biofuel, which reduces emissions the greenhouse gases by (65%- 40%) Because the combustion of both types of fuel does not have

the same effect as shown in **figure (I.2)**, where fossil fuels produce toxic carbon dioxide in the atmosphere, which leads to global warming, unlike biofuels, which burn better because it contains the oxygen atom in a chemical formula, where the carbon emitted from it acts as a source of energy for plants (they use it in the photosynthesis process). As a result, biofuels are more environmentally friendly (**Fatma Zohra Ben chikh, 2022**).

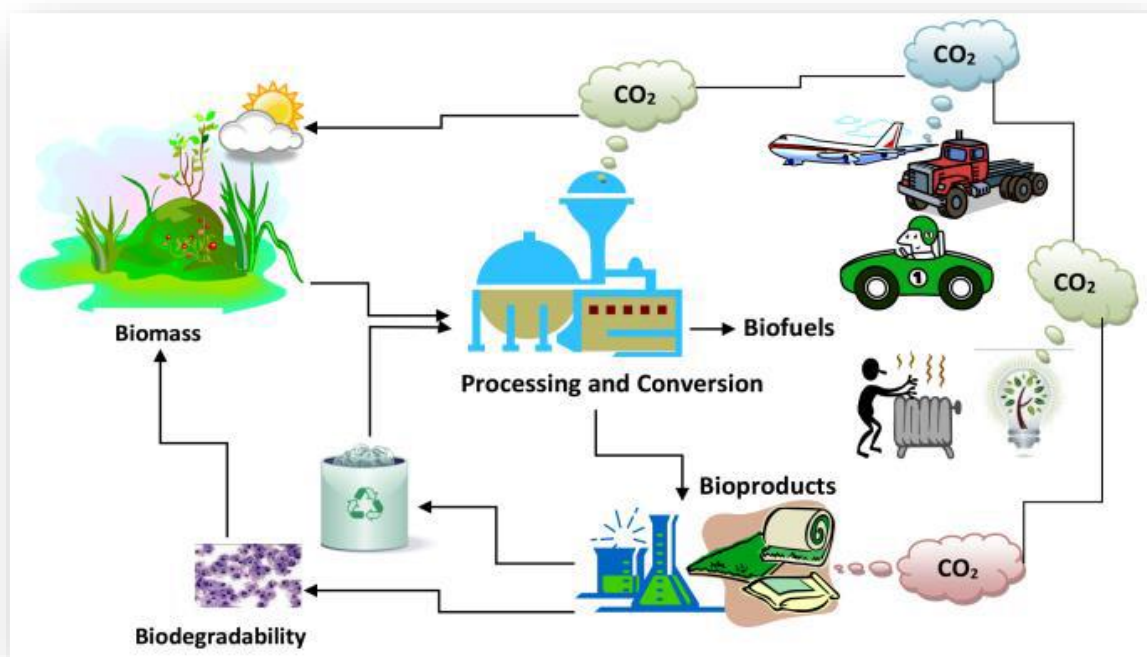


Figure I.2: Biomass life cycle for biofuels.

I.2.4. International production of biofuels

Interest in biofuels has increased further in the past decade with the development of climate change mitigation policies and strategies to reduce greenhouse gas emissions from the transportation sector. Global production of biofuels is dominated by the United States and Brazil – producing 69% of all biofuels in 2018 – followed by Europe (EU-28) with 9%. Biodiesel production has more than tripled, from 12 to 41 billion liters. Currently, biofuels account for about 3.4% of total transportation fuels worldwide. Argentina, Brazil, and the United States also produce significant amounts of biodiesel, mostly from soybean and cooking oil used, while Malaysia and Indonesia produce biodiesel from palm oil. (**Electronic Reference 11**)

I.3. Biodiesel

Biodiesel is a promising nontoxic and biodegradable renewable Fuel comprised of mono-alkyl esters of long chain fatty acids, which is produced by a catalytic transesterification reaction of vegetable oils with short-chain alcohols. (M.C.G. Albuquerque., and all.2009)

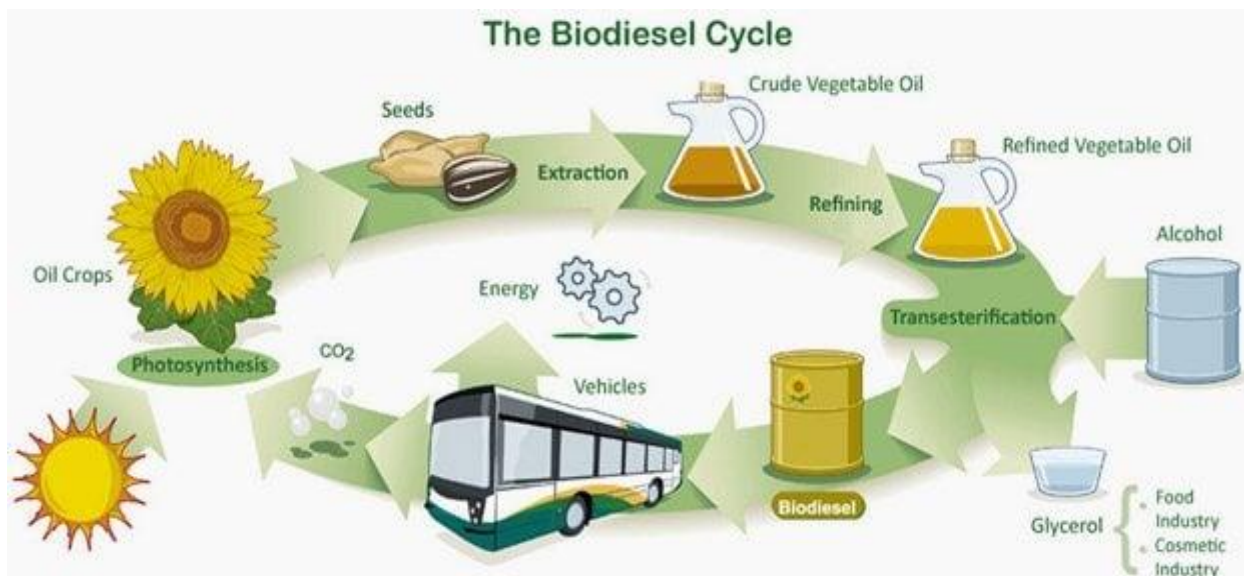


Figure I.3: Biodiesel production cycle from renewable bio-oils. (Electronic Reference 13)

I.3.1. Biomass of biodiesel

The raw materials used in the production of biodiesel vary among countries, climates, and policies. But in general, we can classify these raw materials in three categories: (Rafael Luque., and all.2021)

- ✓ **Edible vegetable oils:** Is produced from vegetable resources; it is used mainly for direct human consumption as food intake. It is more expensive than non-edible due to the limited supply and high demand. The most common edible oil feedstocks that used for biodiesel production are palm, soybean, sunflower, rapeseed and peanut oil.
- ✓ **Inedible vegetable oils:** is the oil that is not valid as a food for human beings as it is not healthy; it is used mainly in industrial applications such as biofuel, soap. As, jojoba, Jatropha, Karanja.
- ✓ **Animal fat:** Animal tallow oil is a promising alternative for producing biodiesel because it is a cheap raw material. Since animal tallow is a valueless product to commercial food industries, it avoids the food vs fuel de bateaux.

- ✓ **Waste Oil (Used cooking oil):** Used cooking oil is a promising alternative for biodiesel production because of its many advantages. Compared with the other oils, used cooking oil characterized by low price, availability, ease of assemblage from houses and restaurants and recevabilité.

I.3.2. Advantages and disadvantages of biodiesel

I.3.2.1. Advantages

- Biodiesel is a renewable energy source as opposed to oil, the reserves of which are finite as the reserves of other fossil fuels.
- Biodiesel can decompose easily under natural conditions, and over 90% pure biodiesel can be degrading in a few weeks.
- Compared with common diesel and petrol, biodiesel has higher combustible value that makes it relatively safe to be stored and transport.
- Biodiesel contains much less sulphur which not only provides lower share of toxic substances in the exhaust but also enables to provide the lubrication of movable parts during the work of the engine. **(Khoudrane Lamia, 2020)**

I.3.2.2. Disadvantages

- High viscosity and surface stress would lead to bigger drops which may cause problems with the system of fuel injection.
- Vegetable oil contains much more unsaturated compounds than diesel, so biodiesel from it is much easier subjected to oxidation. This parameter correlates with the iodine number.
- More expensive due to the raw material. Nowadays, the raw material of biodiesel usually soybean oil in US and peanuts oil in EU. **(Khoudrane Lamia, 2020)**

I.3.3. Biodiesel production methods

To produce derivatives of vegetable oils having properties and performances quite similar to

those of diesel. There are several ways to produce and use biodiesel, including: (Oyetola Ogunkunle., Noor A. Ahmed. 2019)

I.3.3.1. Dilution

Dilution involves the addition of diesel fuel to triglycerides to make it thinner (reduce the viscosity) for better engine performance. No chemical process is needed for this method. It is established that complete replacement of some vegetable oils for diesel fuel is not realistic experimentally due to variation in the viscosity and pour points. Consequently, mixing 20%–25% blends of vegetable oil with diesel has been examined to produce quality performance results for diesel engines.

With varieties of non-edible seed oil such as turpentine, cotton seed, linseed, rubber seed, *Jatropha curcas* and are reported in some literatures. Diluted fuels such as, preheated palm oil and palm oil/diesel oil blends, palm oil/waste cooking oil mixtures, were prepared and successfully applied as fuels in an IC engine.

I.3.3.2. Micro-emulsification

Micro-emulsification, which is also known as co-solvent blending, is the thermodynamic equilibrium dispersion of microstructures with average diameter, d , less than 0.25 of the wavelength of visible light. Much agitation is needed for the mixture to remain in a single state. A micro-emulsification be made by using a co-solvent to dissolve immiscible substances such as vegetable oils and an ester, or vegetable oils and alcohols, such as methanol, ethanol, butanol, hexanol, with the addition of a surfactant and a cetane improver. Addition of diesel fuel is optional depending on the type of engine. Micro-emulsification is a stable technique for reducing high viscosity of triglycerides.

I.3.3.3. Pyrolysis

Pyrolysis refers to anaerobic thermal disintegration of organic materials in the presence of a catalyst. The fragmented matters can be animal fats, vegetable oils, natural triglycerides or FAME. Common products of pyrolysis of triglycerides are alkanes, alkenes, alkadines, aromatics and carboxylic acids. The liquid components of the disintegrated fats are similar to diesel fuels. The Pyrolysis products have CN, viscosity, flash point, and pour point properties lower than diesel fuel but similar heating values.

I.3.3.4. Transesterification

Transesterification has been extensively used in reducing the viscosity of vegetable oil and influencing their conversion to biodiesel. It is a process by which alkyl esters are produced from chemical reactions between alcohol and vegetable oils in the presence of catalyst. The most affordable and available alcohols in this reaction include methanol and ethanol. The type of alcohol used does not entirely determine the biodiesel yield; the selection is determined by cost and performance. Transesterification is a reversible reaction involving a sequence of three reaction stages in which the triglyceride in oil is converted into diglyceride, and diglyceride is converted into monoglyceride (methyl or ethyl ester). Three moles of alcohol are required in this reaction for each mole of triglyceride. But usually, more molar ratio is often employed to shift the reaction forward for maximum biodiesel production. Three ester molecules are produced in the overall chemical reactions; one ester molecule from each step Glycerol, which has commercial value, is derived as a by-product of this reaction. (Oyetola Ogunkunle., Noor A. Ahmed. 2019)

I.3.4. The mechanism of the transesterification reaction

The reaction mechanism of the esterification reaction is summarized in three consecutive and reversible steps: The first is the conversion of triglycerides (TG) to diglycerides. Then, diglyceride seach step, a (DG) convert to monoglycerides (MG) which in turn convert to glycerol. (GL) At fatty acid ester molecule is formed (Fatma Zohra Ben chikh, 2022).

I.3.4.1. basic catalysts mechanism

Base-catalyzed reaction is another conventional, popular transesterification. This reaction had been known since 1880s but it was not until 1920 and 1921 that systematic studies appeared in a comprehensive manner.

There are various alkali-based catalysts have been utilized as homogeneous and heterogeneous transesterification. Base catalyzed transesterification is substantially less time consuming with respect to acid catalyzed transesterification and it is regularly used technique for commercial purpose (Khoudrane Lamia, 2020).

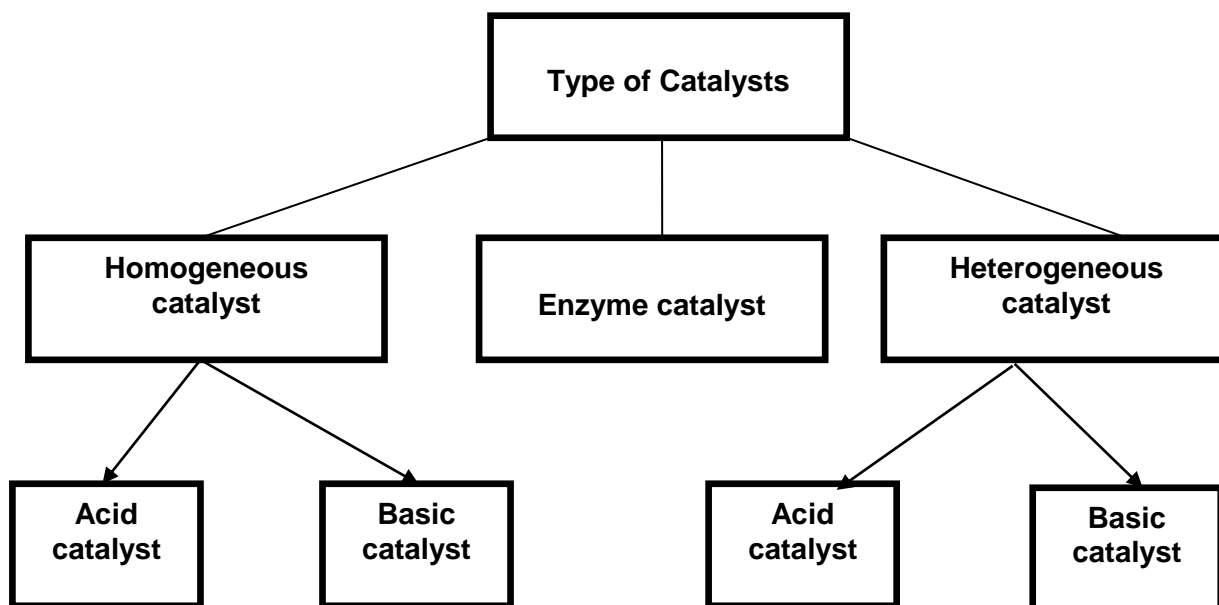
I.3.4.2. Acid catalyst mechanism

Transesterification has been carried out traditionally and most frequently by the use of acid catalysts such as sulfuric, sulfonic, phosphoric, and hydrochloric acids. This method is employable in various cases unless acid sensitive components are involved (Khoudrane Lamia, 2020).

I.3.5. Effect of condition on the transesterification

I.3.5.1. Effect of rate and type of catalyst

Biodiesel is synthesized by transesterification with lower alcohols or by esterification of fatty acids in the presence or absence of a catalyst. There is a different type of catalyst used in the process of transesterification of oils for the production of biodiesel. (Fatma Zohra Ben chikh, 2022).



FigureI.4: Classification of the catalyst used in biodiesel production

a. Homogeneous catalyst

The homogeneous catalysts are usually employed to catalyze transesterification reactions for converting triglycerides to biodiesel fuel. As a result, homogeneous:

- **Alkaline homogeneous catalysts:** such as NaOH, KOH, CH₃ONa
- **Acidic homogeneous catalysts:** such as HCl, H₂SO₄

b. Heterogeneous catalysis

In recent years, heterogeneous catalyst has attracted immense attention for biodiesel production. Heterogeneous catalysts mostly appear in a solid form; thus, the reaction mixture and the catalyst are in a different phase as they can be tailored to match specific requirements

and be easily recovered and reused. Heterogeneous or solid catalysts can be grouped into two categories:

- **Basic heterogeneous catalysts:** CaO, MgO, ZnO.
- **Acidic heterogeneous catalysts:** ZrO₂, MoO₃.

c. Enzymatic catalysis

Enzymatic catalysts are biological catalysts, where the enzyme catalyst has attracted great interest in biodiesel production because of its rapid decomposition in the environment because it has a protein structure, and it requires a low temperature to give high productivity however it but it requires a longer time to obtain a good yield (95%). There are several types of enzyme catalysts, but differ according to the type of biomass and the interaction of the user.

d. The advantages and disadvantages of catalyst

Each different type of catalyst used in the process of transesterification has the advantages and disadvantages as shown in **Table I.1. (Rafael Luque., and all.2021)**

Table I.1: The advantages and disadvantages of catalyst.

Type of Catalysts	Advantages	Disadvantages
Homogeneous catalyst	Homogeneous chemical catalysts contain several Advantages: -Including high selectivity. -Spin frequency. -Reaction rate. - Easy optimization of activity.	-Difficult to separate the catalysts from product . -Technically difficult to recycle the catalyst . -Catalyst needs to be removed or washed by a large amount of hot water . -Produce large amount of industrial wastewater . -Homogeneous acid catalysts are corrosive to equipment.

<p>Heterogeneous catalyst</p>	<ul style="list-style-type: none"> -Easily separated for recycling -Minimize the product separation and Purification costs . -Economically viable to compete with Commercial petro-base diesel fuel. -No formation of soap by the end of the reaction . -Noncorrosive and nontoxic catalysts. 	<ul style="list-style-type: none"> -Long reaction time and slow reaction rate. -High requirements for alcohol/oil, reaction temperature, and pressure -The cost of producing the catalyst is high. -Soap formation as a side reaction. -The cost of producing the catalyst is high.
<p>Enzyme catalyst</p>	<ul style="list-style-type: none"> -Noticed biodiesel can be excellently produced by enzymes. -Avoid soap making. -Non-polluting recyclable. 	<ul style="list-style-type: none"> -High cost. -Hypersensitive to alcohol.

I.3.5.2. Influence of water and free fatty acids

Water and FFA in oils and fats can pose a great problem during transesterification. When water is present, especially at elevated temperatures, it can hydrolyze the triglycerides to diglycerides and form an FFA. However, the presence of water at average temperatures leads to formation of excessive soap formation. When an alkali catalyst such as sodium or potassium hydroxides is present, the FFA will react to form saponified product (**Khoudrane Lamia, 2020**).

I.3.5.3. Effect of rate and type of alcohol

Alcohol is one of the most important raw materials used in the manufacture of alcoholic beverages Biodiesel production. The most widely used acyl acceptor is methanol and to some extent, ethanol. Other alcohols used in pro Biodiesel is short-chain alcohols such as propanol, butanol. Methanol and ethanol are the two most widely used alcohols in biodiesel production. Methanol is particularly preferred, because of its physical and chemical advantages. Besides, the reaction with triglycerides is fast and can be easily dissolved in sodium hydroxide. In

addition, triglycerides can interact with different types of alcohol. But alcohols are short chain **(Idris Atadashi Musa. 2016)**

I.3.5.4. Effect of reaction temperature

The reaction rate and the efficiency of the transesterification process are strongly affected by the reaction temperature. The viscosity of the oil decreases with increasing temperature which accelerates the reaction rate due to the energy supplied for the reaction. The transesterification process is usually carried out at the boiling temperatures of methanol and ethanol are respectively 65°C and 78°C. **(Fatma Zohra Ben chikh, 2022)**

I.3.5.5. Effect of reaction time

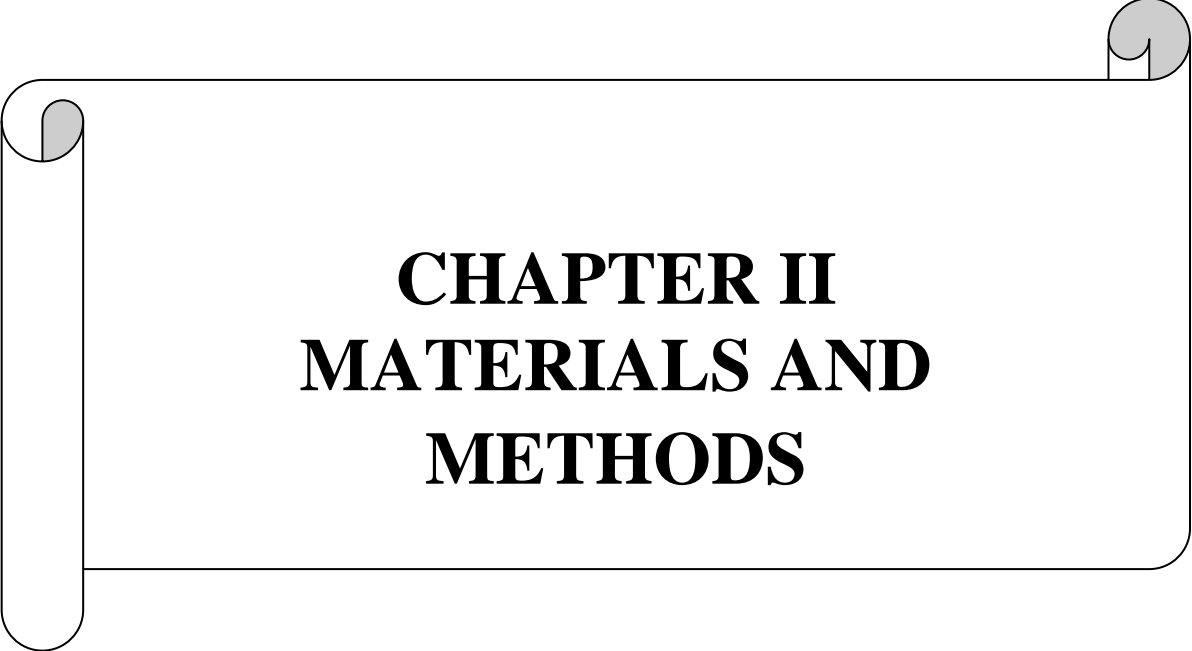
Temperature and reaction time are closely related parameters. As we have just seen in the previous paragraph, a temperature rise accelerates the reaction, which also means that the reaction time is shortened. In this research, biodiesel generated from waste cooking oil is discussed to explore the effect of time using conventional process. **(Fatma Zohra Ben chikh, 2022)**

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**CHAPTER II
MATERIALS AND
METHODS**

CHAPTER .II: MATERIALS AND METHODS

II.1.Stages of manufacturing vegetable oils

Technical transactions of vegetable oils are carried out to obtain oil or its derivatives, according to the following stages: (بن قسوم الخنساء, لبوز فاطمة الزهراء, 2018)

1/ Receipt and storage of seeds: Seeds are received in the factory according to preconditions, such as the minimum percentage of oil in the seeds ,And the percentage of impurities, such as: buds, then the storage process takes place in large quantities that suit the production capacity of the factory so that the factory can operate throughout the year.

2/ Seed preparation: After receiving the seeds, the seeds are subjected to a number of treatments aimed at improving the quality of the oil. These treatments include the following:

-Cleaning: It aims to remove foreign materials associated with oilseeds such as: dirt.

-Removing the husks: This Is done in the case of seeds that have a thick husk or are attached to the seed.

-Grinding (mashing): It works to liberate the oil from inside the oil cells in the seeds.

-Steam heat treatment: It is done for some oil seeds that you need before the oil extraction process.

3/ Oil extraction:

Among the most important methods of extracting oil are the following:

-Extraction by mechanical pressure: It is suitable for oily fruits, as the oil is extracted from cooked slides by mechanical pressure (compression), by means of screw machines or by hydraulic pressure.



FigureII.1: Vegetable oil extraction machine by mechanical pressing.(Electronic Reference 3)

CHAPTER .II: MATERIALS AND METHODS

-Solvent extraction: organic solvents such as hexane and petroleum ether are used, which have high efficiency in oil extraction.

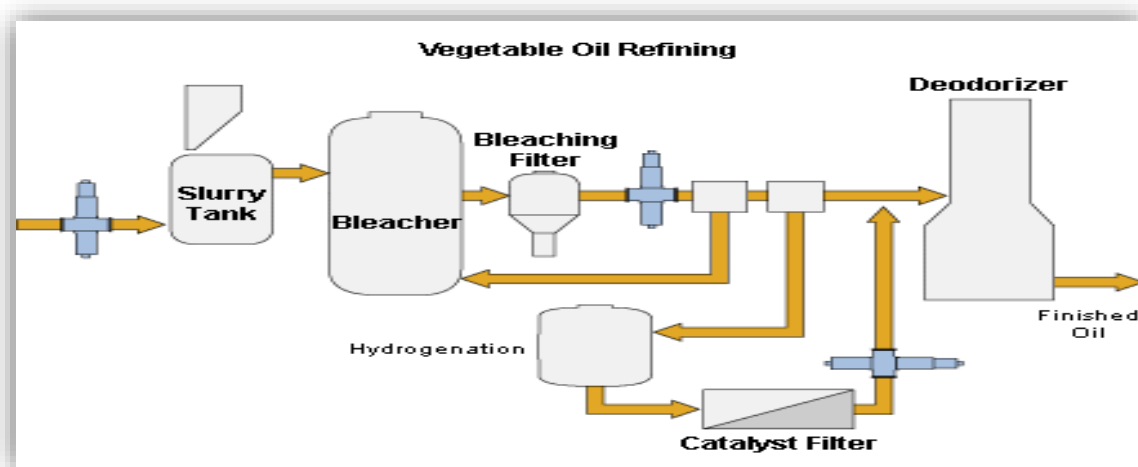
The oil obtained after the extraction process is called crude oil, and it contains 95% of triglycerides, and the remaining 5% are natural compounds.



FigureII.2: Solvent oil extraction device. (Electronic Reference 4)

4/ Refining:

Refining operations include a set of technical operations that are carried out with the aim of converting crude oil into oil suitable for consumption, by removing impurities and unwanted materials, through some of the operations mentioned in the **FigureII.3**.



FigureII.3: Scheme representing the extraction of vegetable oil by refining.(Electronic Reference 5)

CHAPTER .II: MATERIALS AND METHODS

II.2 Take samples: The samples were taken from restaurants in wilaya of Ouargla in the south of Algeria.

II.3. Transesterification

Transesterification is the traditional technique most used for the production of biodiesel. During this reaction, vegetable oils react with methanol molecules to form methyl monoesters and Glycerol. In this present study, we applied this method to waste cooking oil obtain biodiesel.

-The effect of changing the reaction conditions on the esterification reaction and studying the yield by the normal (traditional) method. Among the conditions that the study will be conducted on are as follows:

- ✓ The effect of the amount of oil on the reaction.
- ✓ The effect of the amount of methanol on the reaction.
- ✓ The effect of temperature on the reaction.
- ✓ The effect of time on the reaction.
- ✓ The effect of the amount of catalyst on the reaction.

II.3 .1. Biodiesel manufacturing protocol:

The biodiesel manufacturing protocol consists of 3 steps:

1-Reaction: We used 250 ml of oil heated at 70 ° C with 50 ml of methanol mixed with 2 g of sodium hydroxide (NaOH) catalyst, mixing for 10 minutes, 800 rpm.

2-Separation stage: We leave the mixture for the biodiesel to separate from the glycerin for 12 hours. Separate the biodiesel from the glycerin with a separating funnel or manually in a bottle.

3-Washing: We add an amount of distilled water to get rid of the residual alcohol and catalyst and repeat the process several times to obtain almost pure biodiesel.

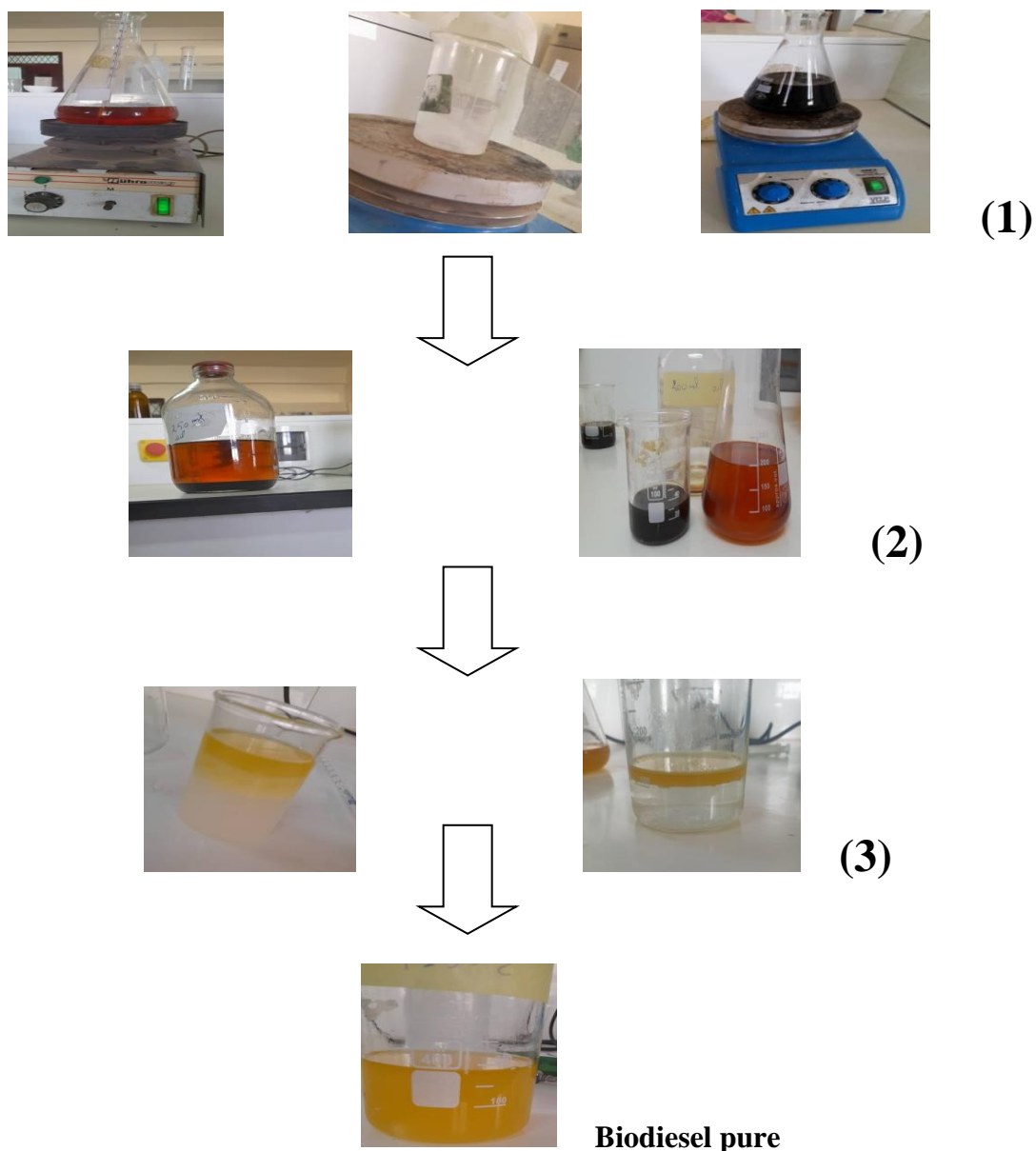


Figure II.4: Biodiesel preparation method: (1) Reaction, (2) Separation stage and (3)Washing

II.3 .2.Analysis

II.3 .2.1.GC-MS analysis of fatty acid

Gas chromatography coupled with a mass spectrometer (GC-MS) is the most widely used analytical method to determine the many fats and volatile oil compounds. In addition, the potential of GC coupled to MS This technique is reserved for the analysis of easily vaporizable and thermally stable molecules is well known for the determination of volatile compounds contained in complex samples of perfumes, allowing the identification of complex sample compositions.(Fatma Zohra Ben chikh,2022)

CHAPTER .II: MATERIALS AND METHODS

Table II.1: Operating conditions for GC/MS analysis.

GC Program	
Capillary column	30 m, ID: 0.25 mm, film :0.25 mm
Injection Temperature (°C)	250 °C
Ion Source Temperature (°C)	200 °C
Carrier gas (rate of flow)	1.21 ml min ⁻¹
Oven Program	60 to 300°C ,10°C/min
Injected quantity	0.5µL
Injection Mode	Split
MS Program	
Mass spectra	45 –500 m/z
	30–35 min

II.3 .2.2.Characterisation of biodiesel and vegetable oil

II.3 .2.2.1.Physical Characterisation

- Density

It is the ratio of the mass of a given volume of oil at 20°C, and the mass of a equal volume of distilled water at the same temperature. (Bourachouhe Karim, Boudei Amir.2017)

- Viscosity

Viscosity is an important characteristic of fuels; it directly influences combustion and affects engine performance and pollutant emissions, and the ease of starting the engine. High viscosity leads to poor spraying which is related to poor volatility causing incomplete combustion in the engine. This poor combustion results in degraded engine performance and higher pollutant emissions. (Bourachouhe Karim, Boudei Amir.2017)

-Melting Point and Freezing Point

Allows to estimate the degree of purity of the fatty substance.

(بن قسوم الخنساء, ليوز فاطمة الزهراء, 2018)

-Solubility

All fatty acids with carbon numbers greater than 8 are insoluble in water and are generally soluble in organic solvents. (بن قسوم الخنساء, ليوز فاطمة الزهراء, 2018)

CHAPTER .II: MATERIALS AND METHODS

-Normal state and appearance:

It is known that fatty substances are liquid or solid at room temperature, depending on their chemical composition. (بن قسوم الخنساء, لبوز فاطمة الزهراء, 2018)

-Refractive Index (RI)

The refractive index of oil is the ratio between the sine of the angle of incidence and Sine of the angle of refraction of a light ray of a given wavelength the air circulating in the oil is kept at a constant temperature.

It is used for identification and as a standard for the purity of oil. (Bourachouhe Karim, Boudei Amir.2017)

II.3 .2.2.2.Chemical Characterisation

-Iodine number (IN)

The iodine number is the number in grams of iodine fixed per 100g of fat. He us provides information on the degree of unsaturation of the carbon chains of the fatty acids.

- Acid number (AN)

Acidity is the percentage of free fatty acid in the fat (oil), it is expressed as a percentage of oleic acid. It tells us about the degree of hydrolysis.

- Saponification number (SN)

The saponification index corresponds to the number of milligrams of potash required to saponify the fatty acids contained in one gram of fat.

-Peroxide number (PN)

This is the quantity of product present in the sample expressed in milliequivalent grams of active oxygen per kilogram of fat oxidizing potassium iodide. This index provides information on the degree of oxidation and alteration of g.

- Ester Index (EI)

Ester index of a fatty substance is the mass of potassium hydroxide (KOH), expressed in milligrams, necessary to saponify the esterified fatty acids present in one gram of fatty substance. (Bourachouhe Karim, Boudei Amir.2017)

It also makes it possible to determine the molar mass (therefore the structure) of glycerids.

CHAPTER .II: MATERIALS AND METHODS

II.3.3. Optimisation (ideal conditions)

- **Reaction yield:**

We calculate the reaction yield by the following relationship:

$$\text{Yield} = \frac{\text{Mass of product}}{\text{Mass of reactant}} \times 100$$

a. Effect of the amount of oil

We analyzed the effect of oil quantities in the traditional way. By varying the quantities of oil from 200 to 400 ml, and the amount of catalyst 2 g of (NaOH) and by the constant temperature at 65 ° C and the reaction time 12 hours with the amount of methanol 50 ml.

Table II.2: Effect of ratio of oil on transesterification with conventional method

Voil (ml)	200	250	300	350	400
Y	50%	50%	50%	50%	75%

b. Effect of the amount of methanol

The amount of catalyst used in the esterification process has an enormous effect on conversion. This process took place with different values of the catalyst from 1 to 3 g of sodium hydroxide (NaOH) and by constant temperature at 70 ° C and reaction time 12 hours with the amount of methanol 50 ml and oil 250 ml.

Table II.3: Effect of the amount of methanol on transesterification with conventional method

Vmethanol (ml)	40	45	50	55	60
Y	80%	80%	91%	80%	80%

CHAPTER .II: MATERIALS AND METHODS

c. Effect of the reaction temperature

The reaction temperature is the most important parameter. We started the effect from the reaction temperature in the traditional way at different temperatures in 55 to 75 ° C, the catalyst ratio was 2 g, and the reaction time was 12 hours, with the amount of methanol 50 ml and oil 250 ml.

Table II.4: Effect of the temperature on transesterification with conventional method

T (°C)	55	60	65	70	75
Y	48%	50%	89%	91%	84%

d. Effect of the reaction time

We study the effect of reaction time with the conventional method. In the experiment, the different time was changed from 4 hours to 48 hours with an amount of 2g and the reaction temperature was 70°C with the amount of methanol 50ml and the oil 250ml.

Table II.5: Effect of the reaction time on transesterification with conventional method

t (h)	4	12	20	24	48
Y	80%	84%	85%	86%	91%

e. Effect of the amount of catalyst

The amount of catalyst used in the esterification process has an enormous effect on conversion. This process took place with different values of the catalyst from 1 to 3 g of sodium hydroxide (NaOH) and by constant temperature at 70 ° C and reaction time 12 hours with the amount of methanol 50 ml and oil 250 ml.

Table II.6: Effect of amount of catalyst on transesterification with conventional method

Amount Cat. (g)	1	1,5	2	2,5	3
Y	83%	84%	91%	0%	0%



CHAPTER III
RESULTS AND DISCUSSION

CHAPTER .III: RESULTS AND DISCUSSION

III.1.Results and Discussion

III.1.1. Analysis of the composition of the oil

The results of the analysis of the components of the oil using GC-MS are shown in table (II.2), from which it is noted that the percentage of fatty acids in the used oil amounted to 89.91%,with 33.25% for saturated fatty acids and 56.62% for unsaturated fatty acids. From these results, we can deduce the molar mass of this oil, which amounted to (810.35 g/mol) using Equation (1) as follows: (Fatma Zohra Ben chikh, 2022)

$$M_{oil} = (M_{acid} \times \alpha_i) + M_{gly} - M_{H_2O} \dots\dots (1)$$

Where:

M_{oil}: Molar mass of oil

M_{acid}: Molar mass of fatty acid

α_i: Percentage of fatty acid in the oil

M_{H₂O}: Molar mass of water (18g / mole)

M_{gly}: Molar mass of the glycerol molecule (136 g / mole)

Table III.1. Composition of fatty acids in waste cooking oil by GC-MS.

Fatty acid	Molecular formula	Molar mass (g/mol)	FFA%
Caprylic acid (8:0)	C ₈ H ₁₆ O ₂	144.21	0.05
Capric acid (10:0)	C ₁₀ H ₂₀ O ₂	172.26	0.13
Margaric acid (14:0)	C ₁₄ H ₂₈ O ₂	228.38	1.29
Palmitic acid (16:0)	C ₁₆ H ₃₂ O ₂	256.43	30.11
Palmitoleic acid (16:1)	C ₁₆ H ₃₀ O ₂	254.41	0.63
Stearic acid (18:0)	C ₁₈ H ₃₆ O ₂	284.48	0.5
Oleic acid (18:1)	C ₁₈ H ₃₄ O ₂	282.46	38.66
Linoleic acid (18:2)	C ₁₈ H ₃₂ O ₂	280.45	9.11
Linoelaidic acid (18:2)	C ₁₈ H ₃₂ O ₂	280.45	7.27
α-Linolenic acid (18:3)	C ₁₈ H ₃₀ O ₂	278.44	0.95
Arachidic acid (20:0)	C ₂₀ H ₄₀ O ₂	312.53	0.49
Lignoceric acid (24:0)	C ₂₄ H ₄₈ O ₂	368.63	0.72
Saturated	/	/	33.29
Unsaturated	/	/	56.62
Total			89.91

CHAPTER .III: RESULTS AND DISCUSSION

III.1.2. Characterisation of vegetable oil

Table III.2. Results of the physical characteristics of table oil.

(بن قسوم الخنساء, لبوز فاطمة الزهراء, 2018)

Physical Characterisation		C.A
Density	0,920	0,918-0,925
Refractive Index	1,461	1,4677

We note from the **Table III.2.** The result of the relative density of table oil equal to 0,920 corresponds to the standards of the Codex Alimentarius; through this value it is possible to predict the degree of purity of oil. We note that the value of the refractive index of the oil is equal to 1, 461, which confirms to the standards of the Codex Alimentarius (Codex Stan 210-1999).

Table III.3. Results of the chemical characteristics of table oil.

(بن قسوم الخنساء, لبوز فاطمة الزهراء, 2018)

Chemical Characterisation		C.A
Acid Number	0,55(mg KOH/g MG)	0,6(mg KOH/g MG)
Peroxide Number	2,77(mg KOH/g MG)	10(mg KOH/g MG)
Saponification Number	195,88(mg KOH/g MG)	187-195(mg KOH/g MG)

We note from the **Table III.3.** That the result of the acid number value is 0, 55, and this is in accordance with the standards of the Codex Alimentarius. And that the value of the peroxide number of the oil equal to 2, 77 agreed with the standards of the Codex Alimentarius 10, while the result of the saponification number of the table oil equal to 195, 88 did not comply with the standards of the Codex Alimentarius (187-195). The difference with the standards of the Codex Alimentarius can be explained by poor preservation and refining.

CHAPTER .III: RESULTS AND DISCUSSION

III.1.3. Characterisation of biodiesel

Table III.4. Results of the physical characteristics of biodiesel.

(أم السعد دباشي, رشيدة نجيمي, 2017)

Physical Characterisation		E.S
Density	0,855	0,86-0,9
Viscosity	4,019	3,5-5

Through the results in this **Table III.4.** We notice that the biodiesel fuel extracted from the frying oil in the laboratory complies with the European standard specifications.

III.1.4. Effect of conditions with a conventional method

a. Effect of the amount of oil

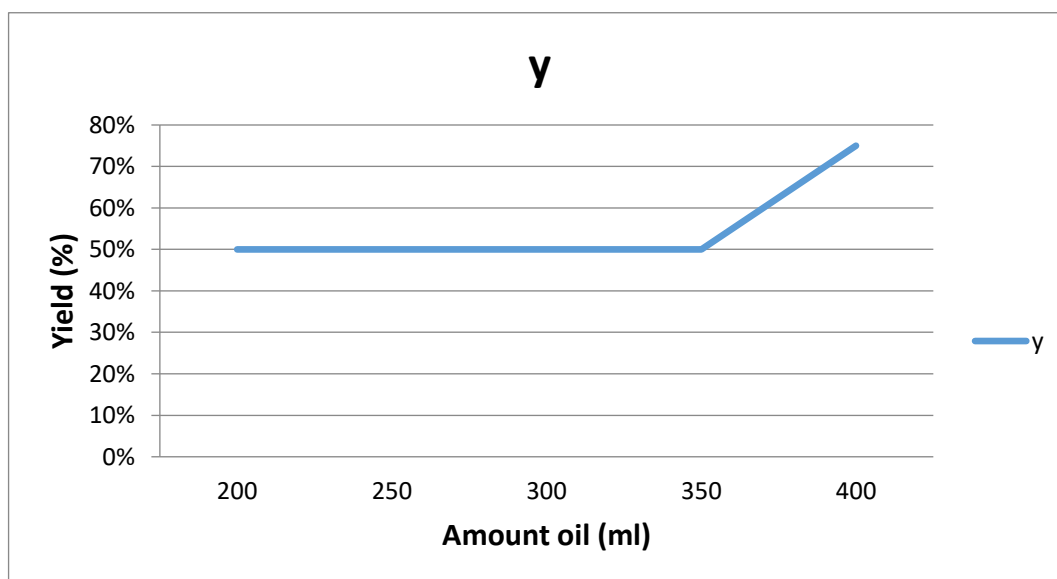
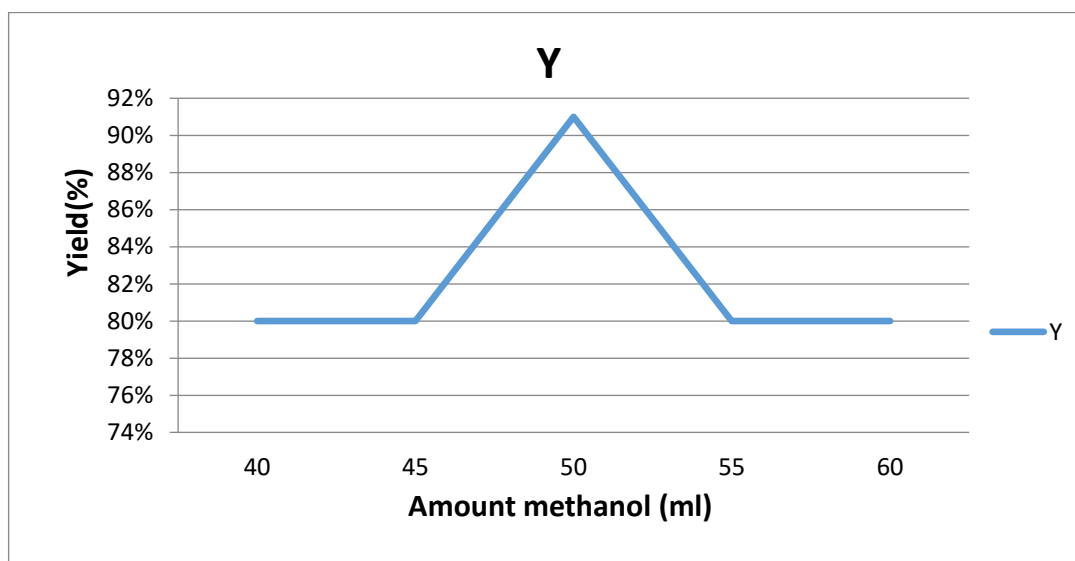


Figure III.1: A graphical curve representing effect of the amount of oil on transesterification with conventional method.

We notice in the **FigureIII.1** that the amount of oil is from 200 to 350 ml, the yield was constant at 50%, and when the amount of oil is increased to 400 ml, we notice an increase in the yield to 75 %, we explain this by the interaction of larger quantities of oil with methanol molecules to form methyl esters and glycerin, which led to higher yields.

CHAPTER .III: RESULTS AND DISCUSSION

b. Effect of the amount of methanol



FigureIII.2: A graphical curve representing effect of the amount of methanol on transesterification with conventional method.

We notice in the **FigureIII.2**, When the amount of oil is 250 ml constant during the whole experiment, that in the two fields [40-45] and [60-55] the yield was constant at 80%, and at the amount of 50 ml of methanol, we notice an increase in the yield rate to 91%. We explain this increase in productivity that 50 ml of methanol was the optimal ratio for its interaction with the amount of oil 250 ml to form fatty acid esters (biodiesel).

c. Effect of the reaction temperature

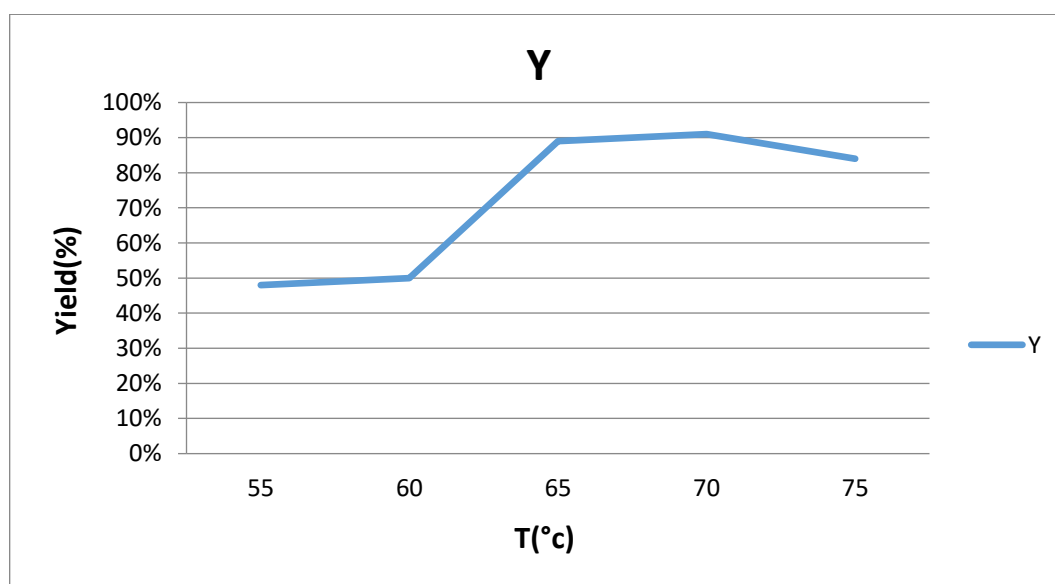
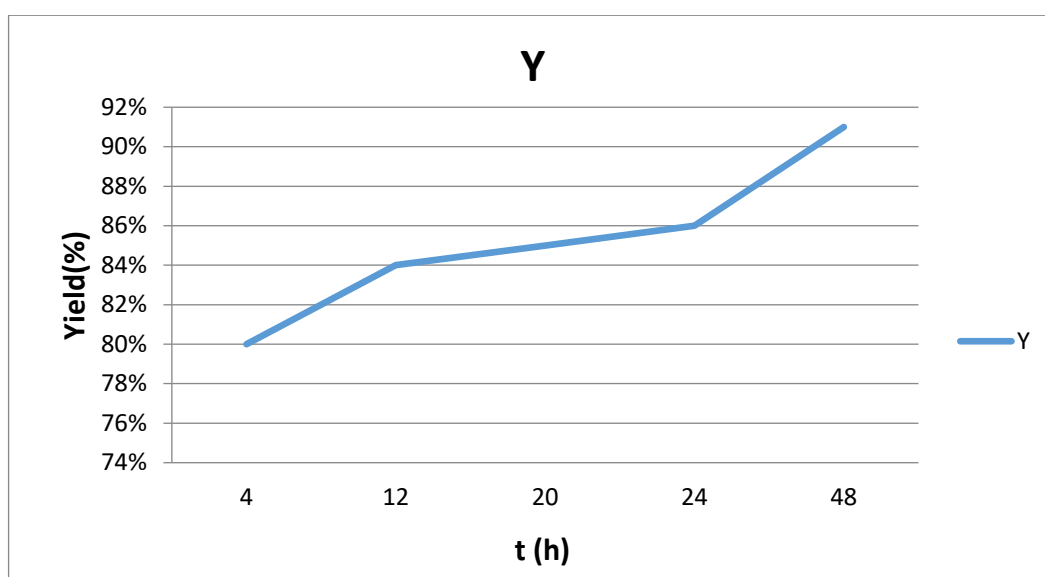


Figure III.3: A graphical curve representing effect of the temperature on transesterification with conventional method.

CHAPTER .III: RESULTS AND DISCUSSION

We note in the **Figure III.3** the temperature was raised from 55 to 75 degrees Celsius, where in the range [60-55] the yield ranged between 48% and 50%, and in the range [70-65] it increased from 89% to 91%, while at a temperature of 75°C reduced the product yield to 84%. We explain this by the fact that whenever the temperature exceeds the optimal limit (70°C), biodiesel production decreases due to the evaporation of methanol at higher temperatures.

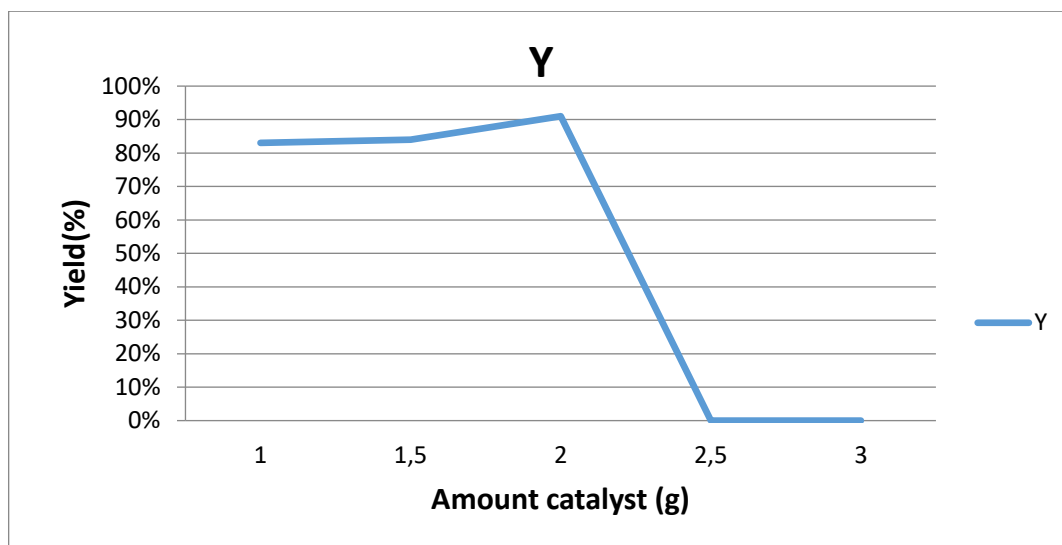
d. Effect of the reaction time



FigureIII.4: A graphical curve representing effect of the reaction time on transesterification with conventional method

We notice in the **Figure III.4** that the process of converting triglycerides into methyl esters was almost complete in about 48 hours from the reaction time and reached its maximum value of 91%. We explain this that time has an effect on the reaction process, as the longer the reaction time, the more biodiesel production yield.

e. Effect of the amount of catalyst



FigureIII.5: A graphical curve representing effect of amount of catalyst on transesterification with conventional method

In the **FigureIII.5**, we observe an increase in yield from 83% to 91% with amounts of 1 to 2 g of NaOH, and a decrease in yield with amounts of 2.5 and 3 g of (NaOH), where 2 g is most appropriate. The catalyst is a reaction accelerator, and we show that there are very high (NaOH) concentrations that do not enable us to produce biodiesel, and this can be attributed to a secondary reaction.

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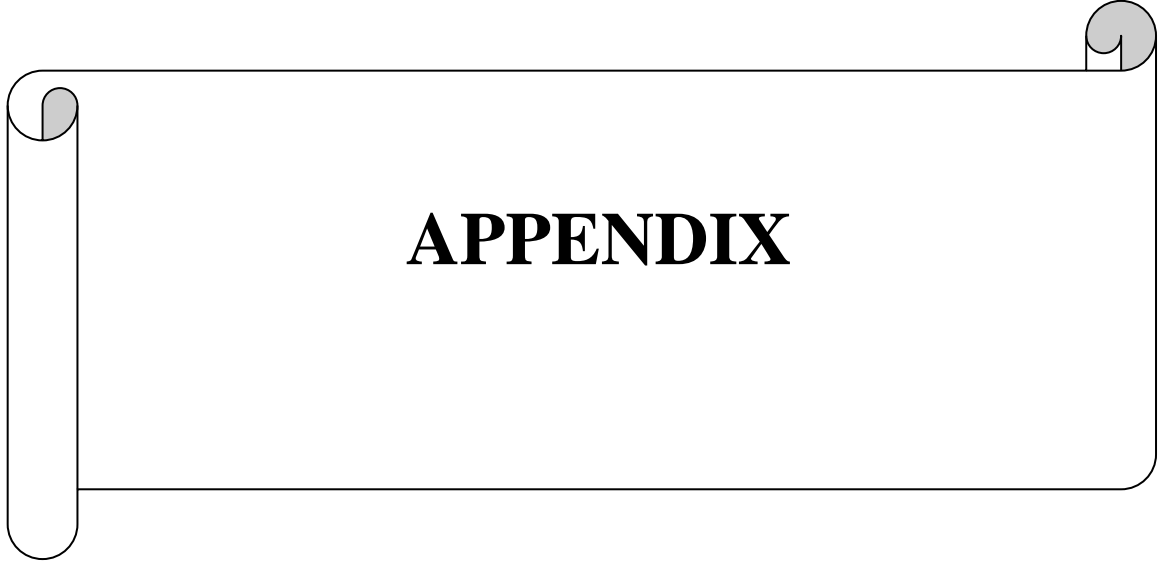
GENERAL CONCLUSION

GENERAL CONCLUSION

Biofuel is the energy derived from living organisms, whether plant or animal, unlike other natural resources such as oil and all types of fossil fuels, biodiesel is a clean fuel. Because the demand for renewable energy is increasing over time. Our goal in this study was to obtain and produce biofuel, or what is known as biodiesel, which we consider environmentally friendly due to its benefits in reducing toxic gas emissions into the atmosphere and reducing the cost of production compared to fossil diesel, which takes time and costs. Biodiesel is produced in several ways, the most important of which is the cross-esterification method, by esterification of triglycerides with alcohol in the presence of an alkaline catalyst (NaOH). For the manufacture of biodiesel, we used frying oil (250 ml), which was esterified with methanol (50 ml), using sodium hydroxide (2 g), following the cross-esterification method, we obtained the amount of biodiesel (228 ml) and the yield was 91% at a temperature of 70 ° C and a period of 48 hours.

This study also opens up future prospects:

- Sowing seeds, harvesting fuels such as the Jatropha plant, which is considered inedible, and is a source for biodiesel production.
- Effect of water quality and purity on biodiesel washing process.
- Exploiting restaurant oils In Algeria and converting them into biofuels.
- Algeria's investment in the second generation of biofuels as a good alternative to energy, based on plant residues such as wheat and corn stalks.



APPENDIX

Table 2: Chemical and physical characteristics of crude vegetable oils (see Appendix of the Standard) (continued)

	Palm olein ²	Palm stearin ³	Palm superolein ³	Pistachio oil	Rape-seed oil	Rape-seed oil (low erucic acid)	Rice bran oil	Safflower-seed oil	Safflower-seed oil (high oleic acid)	Sesame-seed oil	Soya-bean oil	Sunflower-seed oil	Sunflower-seed oil (high oleic acid)	Sunflower-seed oil (mid-oleic acid)	Walnut oil
Relative density (x° C/water at 20°C)	0.899-0.920 x=40°C	0.881-0.891 x=60°C	0.900-0.925 x=40°C	0.915-0.920 15.5°C/water 15.5°C	0.910-0.920 x=20°C	0.914-0.920 x=20°C	0.910- 0.929	0.922-0.927 x=20°C	0.913-0.919 x=20°C; 0.910-0.916 x=25°C	0.915-0.924 x=20°C	0.919-0.925 x=20°C	0.916-0.923 x=20°C	0.909-0.915 x=25°C	0.914-0.916 x=20°C	0.923-0.925 25°C/water 25°C
Apparent density (g/ml)	0.896-0.898 at 40°C	0.881-0.885 at 60°C	0.886-0.900 at 40°C						0.912-0.914 at 20°C						
Refractive index (ND 40°C)	1.458-1.460	1.447-1.452 at 60°C	1.459-1.460	1.467-1.470 at 25°C; 1.460-1.466 at 40°C	1.465-1.469	1.465-1.467	1.460- 1.473	1.467-1.470	1.460-1.464 at 40°C; 1.466-1.470 at 25°C	1.465-1.469	1.466-1.470	1.461-1.475	1.467-1.471 at 25°C	1.461-1.471 at 25°C	1.472-1.475 at 25°C; 1.469-1.471 at 40°C
Saponification value (mg KOH/g oil)	194-202	193-205	180-205	187-196	168-181	182-193	180- 199	186-198	186-194	186-195	189-195	187-194	182-194	190-191	189-198
Iodine value	≥ 56	≤ 48	≥ 60	84-98	94-120	105-126	90- 115	136-148	80-100	104-120	124-139	118-141	78-90	94-122	132-162
Unsaponifiable matter (g/kg)	≤ 13	≤ 9	≤ 13	≤ 30	≤ 20	≤ 20	≤ 65	≤ 15	≤ 10	≤ 20	≤ 15	≤ 15	≤ 15	≤ 15	≤ 20

Summary: Due to the negative impact of fossil fuels on the environment and people, and on the other hand, wastes of used frying oil, as it was noted that biofuels are environmentally friendly.

In our study, we produced biodiesel from frying oil used in the laboratory by the cross-esterification method, in which a triglyceride reacts with alcohol (methanol) in the presence of a catalyst (sodium hydroxide), and by studying influential factors, where the best yield was in the optimal conditions for these factors, namely: 250 ml of oil with 50 ml of methanol in the presence of 2 g of the catalyst (NaOH) at a temperature of 70 ° C and a time of 48 hours, where the best yield was 91%.

Key words: Used frying oil, trans-esterification, biodiesel, Environment, fossil fuels.

المساهمة في تحضير الوقود الحيوي من أصل نباتي (زيت دوار الشمس) حالة منطقة ورقلة

ملخص: بسبب التأثير السلبي للوقود الأحفوري على البيئة والناس ، ومن ناحية أخرى ، نفايات زيت القلي المستخدم ، حيث لوحظ أن الوقود الحيوي صديق للبيئة.

في دراستنا، أنتجنا وقود الديزل الحيوي من زيت القلي المستخدم في المختبر بطريقة الأسترة المتقاطعة، حيث يتفاعل ثلاثي الجليسريد مع الكحول (الميثانول) في وجود محفز (هيدروكسيد الصوديوم)، ومن خلال دراسة العوامل المؤثرة، حيث أفضل محصول كان في الظروف المثلى لهذه العوامل، وهي: 250 مل من الزيت مع 50 مل من الميثانول في وجود 2 جم من المحفز (NaOH) عند درجة حرارة 70 درجة مئوية ووقت 48 ساعة، حيث أفضل عائد كان 91%.
الكلمات المفتاحية: زيت القلي المستعمل، الأسترة التبادلية، الديزل الحيوي، البيئة، الوقود الأحفوري.

Contribution à la preparation de biocarburants d'origine végétale (huile de tournesol), cas de la région d'Ouargla

Résumé: En raison de l'impact négatif des combustibles fossiles sur l'environnement et les personnes, et d'autre part, du gaspillage d'huile de friture usée, car il a été noté que les biocarburants sont respectueux de l'environnement.

Dans notre étude, nous avons produit du biodiesel à partir d'huile de friture utilisée en laboratoire par la méthode d'estérification croisée, dans laquelle un triglycéride réagit avec un alcool (méthanol) en présence d'un catalyseur (hydroxyde de sodium), et en étudiant des facteurs influents, où le meilleur rendement était dans les conditions optimales pour ces facteurs, à savoir : 250 ml d'huile avec 50 ml de méthanol en présence de 2 g du catalyseur (NaOH) à une température de 70°C et une durée de 48 heures, où le meilleur rendement était de 91%.

Mots clés: Huile de friture usagée, trans-estérification, biodiesel, Environnement, combustibles fossiles.