



## Studying of Characterisations of biodiesel obtained from traditional seed oils

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### Abstract

In recent years, the acceptance of biodiesel as an alternative fuel has grown all over the world. The raw materials used to produce biodiesel include traditional seed oils (sunflower and olive oil) and used frying oils. In this study, the properties of these three different raw materials (sunflower, olive oil and used frying oil) were studied by developing a suitable analytical method; then these three different raw materials were converted to biodiesel by transesterification reactions where triglyceride bonds are broken down by reacting with methanol in the presence of potassium hydroxide as a catalyst to produce fatty acid methyl ester (FAME). The properties of the produced biodiesel were studied and compared with ideal diesel. Vegetable oil has different types, with varying properties. The most important properties are degree of unsaturation and free fatty acid content, as the chemical composition of any vegetable oil depends on these two properties.

**Key Words:** Traditional seed oils, Biodiesel, diesel fuel, Characterisation of vegetable oil.

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## دراسة خواص وقود الديزل المستمد من زيوت البذور التقليدية

### الملخص

في السنوات الأخيرة، ازداد قبول الديزل الحيوي كوقود بديل في جميع أنحاء العالم. والمواد الخام المستخدمة لإنتاج وقود الديزل الحيوي تشمل زيوت البذور التقليدية (عباد الشمس وزيت الزيتون) وزيوت القلي المستخدمة، في هذه الورقة تمت دراسة خصائص هذه المواد الخام الثلاثة المختلفة (عباد الشمس وزيت الزيتون وزيت القلي المستخدم) من خلال تطوير طريقة تحليلية مناسبة. كما تم تحويل هذه المواد الخام الثلاثة إلى وقود الديزل الحيوي من خلال تفاعلات الاستبدال حيث يتم تكسير الروابط الثلاثية الجلسيريدات عن طريق التفاعل مع الميثانول في وجود هيدروكسيد البوتاسيوم كمحفز لإنتاج استر الميثيل.

الحمض الدهني. وتوصلت نتائج الدراسة إلى أن الزيوت النباتية لها أنواع مختلفة، مع خصائص متفاوتة وأن أهم الخصائص هي درجة عدم التشبع ومحتوى الأحماض الدهنية الحرة، لأن التركيب الكيميائي لأي زيت نباتي يعتمد على هاتين الخاصيتين. كما تمكنت الدراسة من معرفة خصائص وقود الديزل الحيوي المنتج ومقارنته مع الديزل المثالي.

**الكلمات المفتاحية:** الزيوت النباتية، وقود الديزل الحيوي، وقود الديزل، توصيف الزيوت النباتية

### Introduction

Vegetable oil molecules consist of glycerol with three long carbon chains, which are called 'fatty acid' chain. The composition of different combinations of different fatty acids on each vegetable oil gives it unique properties (Marchetti et al., 2008).

We would like to state that, because oleic acid contains one double bond in its molecular formula, it is stable to thermal oxidation. Therefore, it is preferable for the production of biodiesel but, although most sunflower oils contain about 20% oleic acid, hybrid varieties may reach an 80%



content. Therefore, researchers have a special interest in the synthesis of biodiesel from sunflower oils (Dossat, 2002)& (Knothe,2001).

Biodiesel is a fuel produced from natural oils such as vegetable oils, animal fats or waste cooking oils. The idea of producing such fuel has a rather long history. The first demonstration of plant oil was in 1900 at the International Exhibition in Paris where R. Diesel demonstrated the first test of an engine working on plant oil (peanut oil) (Dayrit et al., 2008). Subsequently, interest in biofuel passed through a number of significant stages when the demand for it was very high, such as in the Second World War and during the energy crises of the 1970s and 1980s(Knothe,2001). The production of biodiesel has started to increase in recent decades; there was a 33-fold increase in production in some European countries during the period 1998–2010 (Feofilova, 2010).

However, some used oil has also been used for the production of biodiesel and some of it consists of free fatty acid due to the reaction between the water content in fried food and the triglyceride which causes it to split up into its component parts.

Although some waste oils also contain varying amounts of fish and animal oils which may be solid at room temperature, waste cooking oils can be recycled, thus avoiding a significant environmental problem (Fukuda and Kondo, 2001).

### **The Aim of Study**

The general aim of this study was to characterise two properties of different kinds of vegetable oil as a basis for biodiesel. One is the degree



of unsaturation and the other is the free fatty acid content. These could be determined by developing analytically method.

This study was motivated by a need to study the influence of raw material properties (degree of unsaturation and free fatty acid content) on biodiesel properties where biodiesel was compared with ideal diesel.

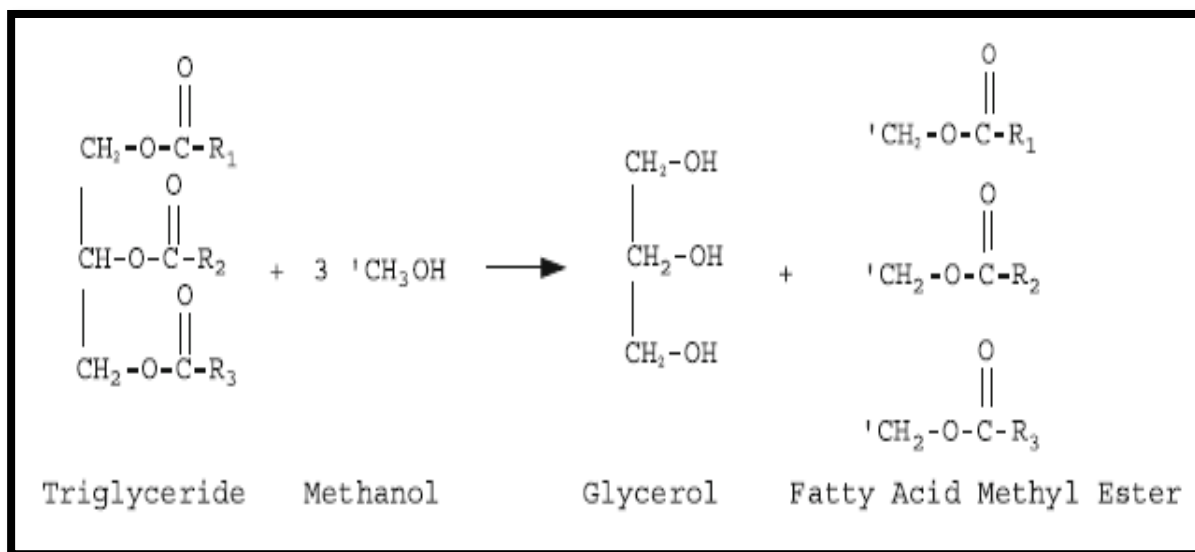
### **1-Transesterification of vegetable oils into biodiesel**

As we said earlier, vegetable oil is the main source of biodiesel; there are at least four ways to produce biodiesel from vegetable oils by reducing their viscosity and this is achieved by breaking down the triglyceride bonds (Ghadge & Raheman, 2006).

**These four ways are:-**

1. Transesterification,
2. Micro-emulsions,
3. Blending.
4. Pyrolysis.

The most common process is transesterification which is also called alcoholism as it is achieved by adding alcohol to react with triglyceride molecules in the presence of a catalyst to produce fatty acid methyl ester (FAME), which is the biodiesel and glycerine (Miguel et al., 2001).



The chemical reaction with an alcohol (methanol) is shown schematically in Fig(1).

**Fig. (1) Overall mechanism of transesterification (Fukuda and Kondo, 2001).**

In principle, glycerol removal is an easy process, but it needs vigorous stirring. Different alcohols were used for this process - ethanol, methanol, propanol, and butanol - but the most common one was methanol due to its good physiochemical composition and low cost. Pure methanol is also required as the water content can lead to a decrease in selectivity (Zhang et al.,1999). The most suitable catalyst was potassium hydroxide. Also, some research was carried out to study transesterification in supercritical media(Demibras, 2003)&( Madras et al., 2004).

However, transesterification occurred in several reversible consecutive reactions; therefore many variables should be noted in such



processes, such as reaction temperature, the amount of catalyst, mixing intensity, type and amount of catalyst and raw oil used (Schwab et al., 1998).

Many studies have examined the kinetic mechanism of this process which shows variations according to the molar ratio of oil and alcohol; for example, when the ratio is 6:1 oil/alcohol, the reaction was the second order while, when it was 30:1 oil/alcohol, the reaction was a pseudo-first-order reaction mechanism (Freedman et al., 1987).

Generally, the molar ratio of 6:1 is used to complete the reaction. Pyrolysis process is a chemical modification method using thermal energy in the presence of oxygen to decompose triglycerides, which results in different reaction products due to the wide variety of reaction pathways; however, although the produced biodiesel has the same chemical prosperities of diesel, the environmental benefits of the produced biodiesel were decreased due to oxygen removal during thermal cracking (Ma & Hanna, 1999).

The Micro-emulsions process involves different solvents such as methanol, 1-butanol and ethanol being used to lower the viscosity of biodiesel. Micro-emulsions are thermodynamically stable dispersions of water, oil and a surfactant. Although the micro-emulsions process produces fuel with lower viscosity, it has some disadvantages such as incomplete combustion of the fuel and less efficient injection into the engines (Srivastava et al., 2000).



## 1. Vegetable oil

Vegetable oil has different types, with varying properties. The most important properties are degree of unsaturation and free fatty acid content, as the chemical composition of any vegetable oil depends on these two properties

For example, the difference in the composition of the fatty acid in three different vegetable oils is given in Table (1) and the difference between their properties is given in table (2).

**Table (1): Difference composition of the fatty acid in three different vegetable oils.**

Vegetable oils	Fatty Acids						
	Lauric	Myristic	Palmitic	Stearic	Oleic	Linoleic	Linolenic
<b>Soya bean</b>	0.1	0.1	10.2	3.7	22.8	53.7	8.6
<b>Palm</b>	0.1	1	42.8	4.5	40.5	10.1	0.2
<b>Tallow</b>	0.1	2.8	23.3	19.4	42.4	2.9	0.9

**Table (2): Difference prosperities between their properties three different vegetable oils.**

	<b>Soya bean</b>	<b>Palm</b>	<b>Tallow</b>
Kinematics viscosity (mm <sup>2</sup> /s)	4.5	5.7	
Cetane number	45	62	
Cloud point (1C)	1	13	12
Pour point (1C)	-7		9
Flash point (1C)	178	164	96



Finally, there are many advantages of using vegetable oil as a fuel:

- It is environmentally friendly.
- Waste oil is cheaper.
- It provides smoother engine running - no 'knock'.
- There is less reliance on petro-chemicals.
- It offers enhanced street credibility.

However, there are some disadvantages; for example, it may cause engine coking if misused and may destroy some injector pumps.

## 2. Biodiesel

Generally, biodiesel consists of monoalkylic esters and hydrocarbon chain in the range of 14 - 22 carbon atoms, capable of combusting properly in conventional diesel engines (Vicente Crespo et al., 2001).

The most common oils used to produce biodiesel are rapeseed, soybean, and sunflower but there are also more than 300 vegetable oils that can be used to produce biodiesel, depending on their availability in the production area; therefore, biodiesel has different names depending on the raw materials (Gerde et al., 2007):

- SME: Soybean/sunflower methyl ester
- PME: Palm Methyl Ester
- RME: Rapeseed methyl ester
- FAME: Fatty Acid Methyl Ester generally referred to as other vegetable or waste cooking oils and containing up to fourteen different types.





### 3. Advantages and disadvantages of biodiesel in comparison with diesel fuel.

In comparison with mineral diesel fuel, biodiesel fuel possesses a number of advantages(Vicente Crespo et al.,2001):-

- 1.A renewable energy source as opposed to diesel fuel.
- 2.Relatively safe due to its higher combustible value.
- 3.No significant influence on the environment because, under natural conditions, biodiesel easily decomposes; 99% of biodiesel takes only 28 days to decompose in water or soil through micro-organisms, while diesel fuels consist of more than 300 compounds which have a harmful effect on the environment.
- 4.Biodiesel contains a very small amount of sulphur (0.0001%), carbon monoxide and dioxide, aerosol and polycyclic aromatic hydrocarbons .On the other hand, biodiesel has a number of disadvantages in comparison with **diesel fuels:-**

- High surface stress and high viscosity of biodiesel results in bigger drops, which affect an engine's injection system.
- The presence of heat, air, metals and peroxides have an influence on the saturation of the hydrocarbon chain in biodiesel oil; this results in oxidation resistance which is considered the main disadvantage of biodiesel in comparison with diesel fuel.
- The lower crystallization temperature of biodiesel results in freezing, which affects the fuel pump.



## Material and Methods

### -Characterisation of vegetable oil

#### 1. Degree of unsaturation

The physical properties of vegetable oil vary widely due to their differing proportions of fatty acids. Some of those differences result from the differences in unsaturation. Therefore, a high iodine number, which means high unsaturated fatty acids, leads to an improvement in biodiesel low-temperature operability, but the oxidation stability decreases; on the other hand, the properties of the biodiesel such as cetane number and turbidity while using higher polyunsaturated fatty acids such as linolenic or linoleic acids leads to the opposite effect.

#### 2. Free fatty acid content

Different vegetable oils contain different free fatty acids content (FFA). The free fatty acid content of these vegetable oils is an important parameter. The main fatty acids in sunflower oil are linoleic and oleic acids, both with a 19-carbon atom chain, but linoleic acid has two double bonds while oleic acid has one double bond. Although some varieties have around 70 % of oleic acid, linoleic acid forms about 80 % of the free fatty acid content (Gerde et al., 2007).

Many studies have reported different ways to determine free fatty acid content, such as acid-based titration methods (Zhang et al., 2007) & (Che Man & Ma, 1999) which depend on a visual endpoint, pH metry, chromatography, GC and HPLC FTIR, <sup>31</sup>P NMR, <sup>1</sup>H NMR Spectroscopy and near IR.



### 3.Viscosity

Vegetable oils are characterised by their high viscosity in contrast to diesel fuel and that is due to their chemical structures and molecular weights. There is a significant difference between the chemical structures of vegetable oil and diesel fuel.

The viscosity of biodiesel is related to its chemical structure, as the increase in chain length leads to an increase in viscosity, while the viscosity decreases with the increase in the degree of unsaturation (double bonds). Moreover, biodiesels produced from fats and greases has high viscosity when compared with other biodiesels produced from vegetable oils, as their saturation the level is much higher (Demirbaş, 2003).

## - Experiment and techniques

### 1.Chemicals

All the chemicals were of analytical reagent grade and were used as such without any further purification. Oleic acid was procured from Fluka Chemika. Potassium hydroxide, sodium hydroxide, sodium thiosulfate iodine solution, potassium iodide, and methanol (99%) were obtained from Fisher Scientific. All the vegetable oils (sunflower oil and olive oil) were procured from Sainsbury's Supermarkets Ltd and used oil was obtained from the University of Huddersfield store. Deionised Millipore water was used to prepare solutions for titration.



## 2. Equipment

Spectrophotometer (spectral type, side port) with a silicon photodiode array with flat holographic grating as a detector from Konica Minolta Sensing Americas, Inc. was used to measure colour intensity of biodiesel.

Plain Jacket Bomb Calorimeter from the PARR INSTRUMENT COMPANY was used to obtain the heat content of biodiesel and diesel fuel.

## 3. Characterisation of vegetable oil

Characterisation of vegetable oil was achieved by measuring the degree of unsaturation and free fatty acid content.

## 4. Degree of unsaturation

The degree of unsaturation was obtained by calculating the weight in grams of iodine absorbed by 100 g of oils; this is called iodine value of oils according to the Association of Official Analytical Chemists (AOAC) Official Method 993.20.

## 5. Experiment procedure for olive oil and used oil:-

50 ml of solvent (1:1 cyclohexane: acetic acid) was added to 1.5 g of olive oil in a 50 ml volumetric flask, then 5 ml of the solution was added to 3 dark glass bottles.

10 ml of solvent (1:1 cyclohexane: acetic acid) and 25 ml WIJ's solution was added to each bottle. At the same time, a blank bottle was prepared by adding 15 ml solvent (1:1 cyclohexane: acetic acid) to 25 ml WIJ's solutions and mixed carefully.

All bottles were kept in the dark for 1 hour. Immediately after that, 20 ml of 15% aqueous potassium iodide was added to each bottle and



mixed carefully; then, 150 ml of distilled water was added to each bottle. Within 30 minutes all the bottles were titrated by 0.1 M sodium thiosulphate until the bottle contents turned a pale yellow colour. At this point 2 ml of starch solution was added to each bottle, then the titration continued until the blue colour disappeared.

The iodine value was calculated by the equation

$$\text{I.V.} = (B - S) \times [\text{Molarity of sodium thiosulphate} \times (12.69 / \text{wt of fat or oil sample})]$$

### **6. Transesterification of vegetable oil to biodiesel**

Transesterification occurs by the reaction between vegetable oils and methanol in the presence of potassium hydroxide as a catalyst to produce ester and crude glycerol which should remove.

Transesterification of sunflower oil and olive oil:-

3 g of 0.05 M Potassium hydroxide (the catalyst) was dissolved into 90 ml methanol (the alcohol). 300 ml of the oil was transferred into the reactor, which is a round-bottomed flask of 3 neck, capacity 500mL settled into a water bath at 60°C; then the catalyst/alcohol solution was transferred into the reactor and the mixture stirred for two hours. After two hours, the reaction produced two liquid phases which are crude glycerol and ester. Therefore, the mixture was transferred to a funnel to separate crude glycerol, which collected from the bottom after several minutes. Then, the remaining solution in the funnel was washed several times with deionised warm water (approximately 50°C) until the pH of the solution changed to 7. Then, the solution was heated up to 40°C to remove the alcohol ( Stavinoha & Howell,1999).( Satyarthi et al., 2009).



## 8.Characterisation of biodiesel

### -Degree of unsaturation

50 ml of solvent (1:1 cyclohexane: acetic acid) was added to 1.5 g of biodiesel in a 50 ml volumetric flask then 5 ml of the solution was added to 3 dark glass bottles.

10 ml of solvent (1:1 cyclohexane: acetic acid) and 25 ml WIJ's solution was added to each bottle. At the same time, a blank bottle was prepared by adding 15 ml solvent (1:1 cyclohexane: acetic acid) to 25 ml WIJ's solution and mixed carefully.

All bottles were kept in the dark for 1 hour. Immediately after that, 20 ml of 15% aqueous potassium iodide was added to each bottle and mixed carefully; then 150 ml of distilled water was added to each bottle.

Within 30 minutes all bottles were titrated by 0.1 M sodium thiosulphate until the bottle contents turned a pale yellow colour. At this point 2 ml of starch solution was added to each bottle, and then the titration continued until the blue colour disappeared.

The iodine value was calculated by the equation

$$I.V. = (B - S) \times [\text{Molarity of sodium thiosulphate} \times (12.69 / \text{wt of fat or oil sample})]$$

### - Free fatty acid content

Approximately 7 g of biodiesel was added to 5 different 500 volumetric flasks then 75 ml of methanol was added to each flask.

Flasks were heated up to 40°C, and then two drops of phenol phthaline were added to each then all flasks.

0.05 M sodium hydroxide was prepared and used to titrate all flasks.



The free fatty acid content was calculated by using the following equation:-

$$\text{Free fatty acid \%} = \left( \frac{\text{mL } 0.1 \text{ N NaOH} \times 28.2}{\text{Sample Wt., g}} \right)$$

## **9. Heat characterisation for vegetable oil, biodiesel and diesel**

### **a. Determination of the pour point, upper pour point and cloud point for vegetable oils and biodiesels.**

The pour point is the lowest temperature at which the liquid lost its ability to flow and the upper pour point is the lowest temperature before the pour point at which the liquid will flow very slowly. Cloud point is the temperature at which the cloud of solid appears in the liquid, where bio wax in biodiesels forms a cloud at a certain temperature. Therefore, cloud point describes the tendency of the biodiesel to plug filters in cold temperatures.

### **b. Experiment procedure**

20 ml of different vegetable oils and biodiesels were transferred into a plastic beaker and the beaker was put in the freezer.

At about 10°C, the test beaker was removed and tilted for 5 seconds to check the movement of the liquid. Pour point, upper pour point and cloud point were determined by visual observation.

### **C. Determination of calorific value**

A Plain Jacket Bomb Calorimeter with a digital thermometer was used to obtain the calorific value which is the amount of heat liberated on the complete combustion of unit weight of fuel.

### Experiment procedure

1g of this was transferred into the pellet which was weighed before and after the benzoic acid to determine the weight of benzoic acid accurately; it was then placed in the dish inside the bomb.

According to the manufacturer's instruction manual, 10 cm of ignition wire was connected to the terminals inside. The bomb was placed in the steel receptacle (Moschner et al., 2006).

The bomb was filled with oxygen to a pressure of 20 atm then put into the bucket which was filled with 2 litres of water. The temperature was recorded for 5 minutes at one-minute intervals. Then the bomb was fired by pressing the ignition button until the indicator light went out.

The temperature was recorded at 30-second intervals until it went to steady state.







**Fig. (2) digital thermometer. Source: (Madras et al., 2004).**

## Results and Discussion

### 1.Determination of the degree of unsaturation and the free fatty acid content for sunflower oil

#### -Degree of unsaturation:

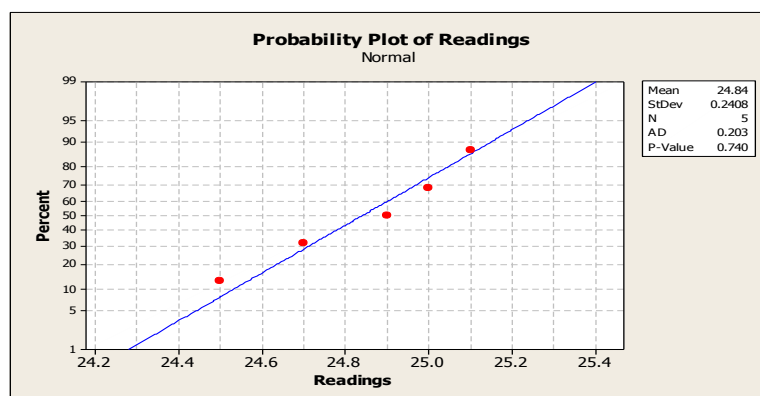
Sample(1g) was prepared by dissolving it in solvents and titrating it with

0.1 M

Blank	end
point	
45	

sodium thiosulphate

#### Results:-



Sample end point
24.9
25.1
24.7
24.5
25



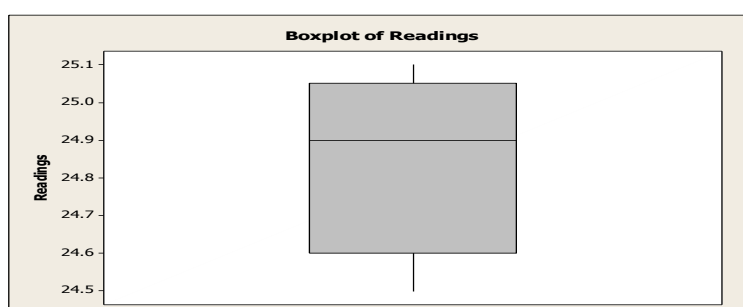
**Fig. (3) The degree of unsaturation and the free fatty acid content for sunflower oil**

**-Statistical calculation for readings:-**

**Table.(3) The values of statistical readings**

Mean	SE Mean	StDev	RSD%	Minimum	Maximum
<b>Readings</b> 25.100	24.840	0.108	0.241	0.97	24.500

We performed a normality test to confirm that the data are of the normal distribution as the portability value is higher than 0.05 at 95% confidence level, which means that there is no significant difference between the readings and the data are of the normal distribution. Therefore, we used the mean of the readings for calculations. Also, we performed a Boxplot graph to confirm the absence of outliers.





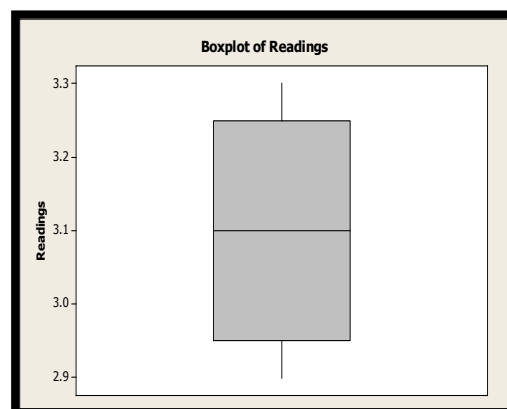
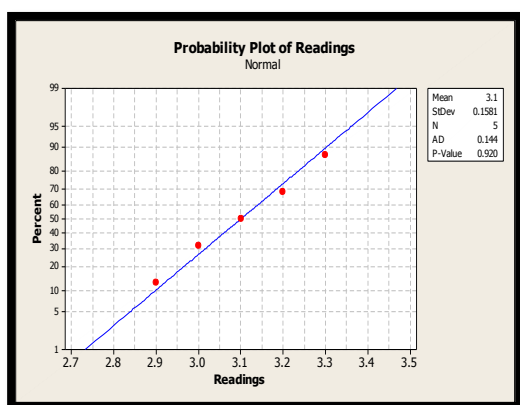
**Fig.(4) The Boxplot graph to confirm the absence of outliers.**

### - Calculation of the degree of unsaturation of sunflower oil

$$I.v. = (B - S) \times (M \text{ sodium thiosulphate}) \times \left( \frac{12.69}{\text{wt of sample oil}} \right)$$

$$I.v. = (45 - 24.84) \times (0.1) \times \left( \frac{12.69}{0.2} \right) = 126.9 \text{ g}$$

### -Free fatty acid content



C
3.1
3.2
2.9
3.0
3.3



**Fig.(5) The difference between of Free fatty acid content Boxplot graph to confirm the absence of outliers.**

**Table.(4) The Value Of Reading**

Mean	SE Mean	StDev	RSD%	Minimum	Maximum
3.1000	0.0707	0.1581	5.10	2.9000	3.3000

**Normality test:** - Normal distribution

Boxplot test: - No outliers

$$\text{Free fatty acid \%} = \left( \frac{\text{mL } 0.1 \text{ N NaOH} \times 28.2}{\text{Sample Wt., g}} \right)$$

$$\text{Free fatty acid} = \left( \frac{3.1 \times 0.05 \times 28.2}{5.66} \right) = 0.77\%$$

**-Viscosity**

**Table.(5) Result of Normality test**

Speed	Percentage %	CPS
100	22.3	71.2

**-Pour point, upper pour point and cloud point**

**Table.(6) Results of Pour point, upper pour point and cloud point**

Upper pour point	-9 °C
Pour point	-15 °C
Cloud point	-2 °C



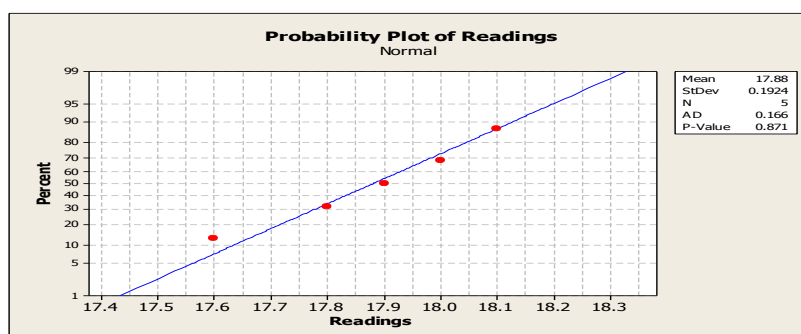
FFA
3.1
3.2
3.4
3.7
4.1

## 2.Determination of the degree of unsaturation and the free fatty acid content for biodiesel of

### sunflower oil.

#### -Degree of unsaturation

Sample (1g) was prepared by dissolving it in solvents and titrating it with 0.1M sodium thiosulphate **Results:-**





**Fig .(6) The degree of unsaturation free fatty acid content for biodiesel of sunflower oil**

**- Statistical calculation for readings:-**

**Table .(7) The values of Statistical readings of Blank end point and FFA.**

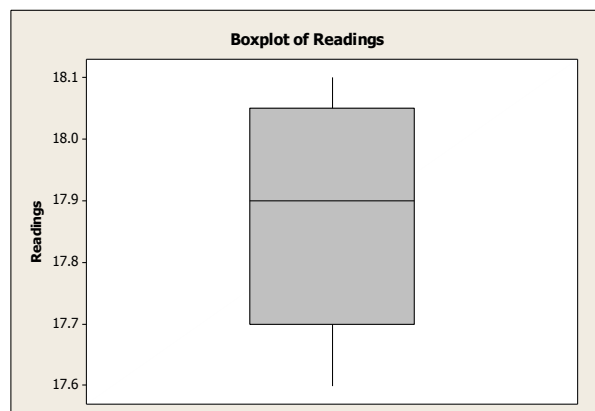
Blank end point
45

Mean	SE Mean	StDev	RSD%	Minimum	Maximum
17.880	0.0860	0.192	1.08	17.600	18.100

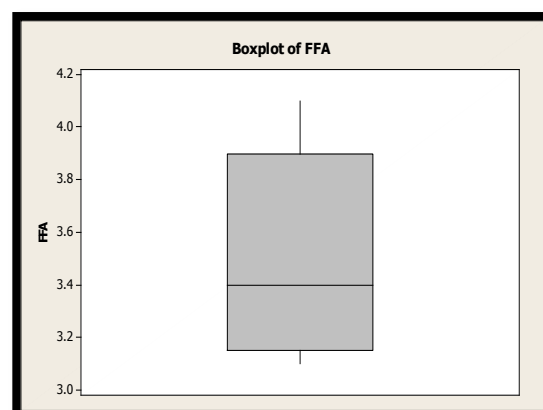
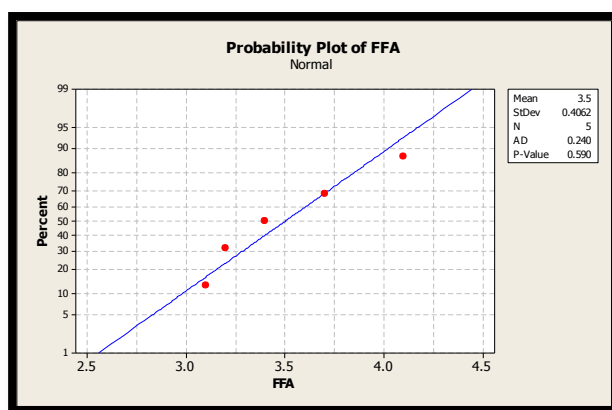
We performed a normality test to confirm that the data are of the normal distribution as the portability value is higher than 0.05 at 95% confidence level, which means that there is no significant difference between the readings and the data are of normal distribution. Also, we performed a Boxplot graph to confirm the absence of outliers. Calculation of the degree of unsaturation of sunflower oil (Madras et al., 2004).

$$I.v. = (B - S) \times (M \text{ sodium thiosulphate}) \times \left( \frac{12.69}{wt \text{ of oil sample}} \right)$$

$$I.v. = (45 - 17.88) \times (0.1) \times \left( \frac{12.69}{0.2} \right) = 172.08$$



**Fig.(7)The degree of unsaturation of sunflower oil source( madras et al., 2004).**



**-Free fatty acid content**

**Fig .(8) Results of Free fatty acid content (FFA)**

Mean	SE Mean	StDev	RSD%	Minimum	Maximum
3.230	0.195	0.437	13.52	3.010	4.010

**Table. (8) The values of statistical reading for (FFA)**

**Normality test :-** Normal distribution

**Boxplot test:-** No outliers

$$\text{Free fatty acid \%} = \left( \frac{\text{mL } 0.1 \text{ N NaOH} \times 28.2}{\text{Sample Wt., g}} \right)$$

$$\text{Free fatty acid} = \left( \frac{3.23 \times 0.05 \times 28.2}{7.03} \right) = 0.65 \%$$

**-Pour point, upper pour point and cloud point**

**Table. (9) The values of statistical reading for Pour point, upper pour point and cloud point**

Upper pour point	-4 °C
Pour point	-5 °C
Cloud point	2 °C

$$\text{The heat capacity of calorimeter } C_p = \frac{C_{p1} + C_{p2}}{2} = \frac{20.704}{2} = 0.352 \text{ kJ/}^\circ\text{C}$$





## 2. Determination of calorific value $q$ for biodiesel made from sunflower oil

**Table. (10) The values of statistical reading for Determination of calorific value  $q$  for biodiesel made from sunflower oil**

$$q_1 = C_p \times \Delta t = 3.88 \times 10.352 = 40.16 \text{ kJ}$$

$$q_2 = C_p \times \Delta t = 3.7 \times 10.352 = 38.30 \text{ kJ}$$

$$\text{Calorific value } q = \frac{q_1 + q_2}{2} = \frac{28.19}{2} = 39.10 \text{ KJ}$$

**Table. (11) Results of Time(min) and Temperature(c)**

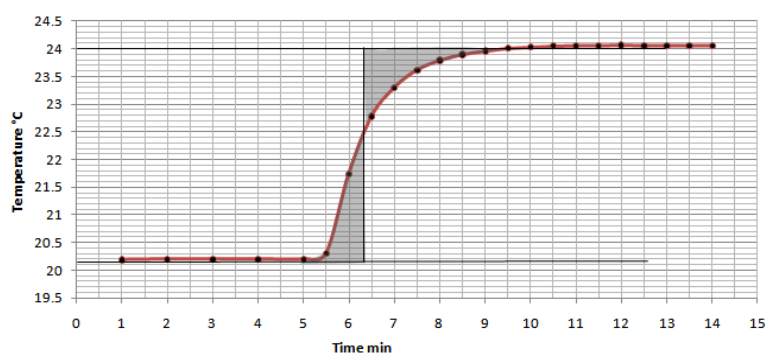
Time (min)	Temperature (°C)
1	20.19
2	20.2
3	20.2
4	20.2
5	20.2
5.5	20.3
6	21.74
6.5	22.78
7	23.3
7.5	23.62
8	23.79
8.5	23.9
9	23.96
9.5	24.01
10	24.03
10.5	24.05
11	24.06
11.5	24.06



12	24.07
12.5	24.06
13	24.06
13.5	24.06
14	24.06

$$I.v. = (B - S) \times (M \text{ sodium thiosulphate}) \times \left( \frac{12.69}{wt \text{ of oil sample}} \right)$$

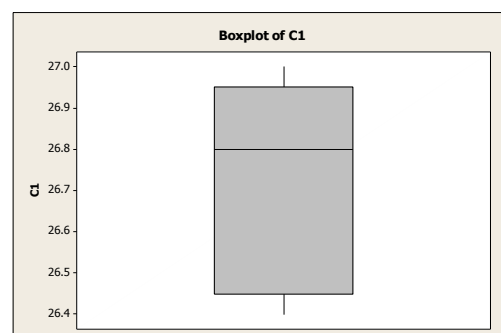
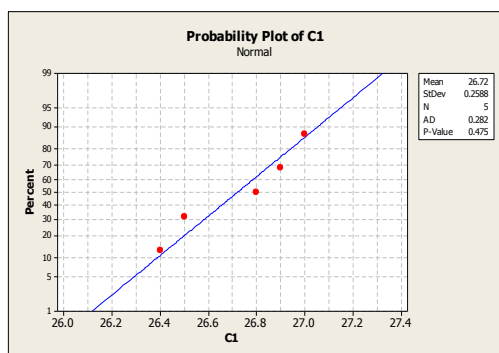
$$I.v. = (32 - 14.86) \times (0.1) \times \left( \frac{12.69}{0.3} \right) = 72.50$$



**Fig.(9): Results of Time and Temperature**

### -Free fatty acid content with oleic acid

FFA
26.4
26.8
27
26.9
26.5





**Fig. (10) The values of statistical reading for Free fatty acid content with oleic acid**

**-Statistical calculation for readings:-**

Mean	StDev	RSD%	Minimum	Maximum
26.720	0.259	0.97	26.400	27.000

**Table.(12) Statistical calculation for readings**

Normality test :- Not normal distribution

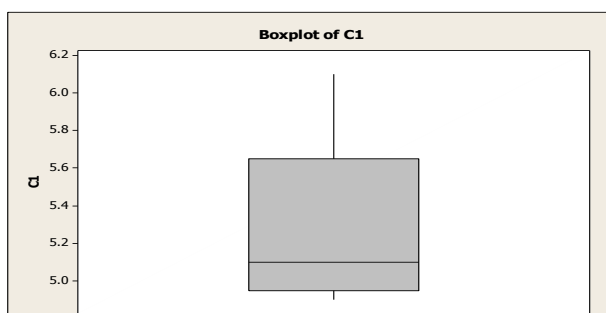
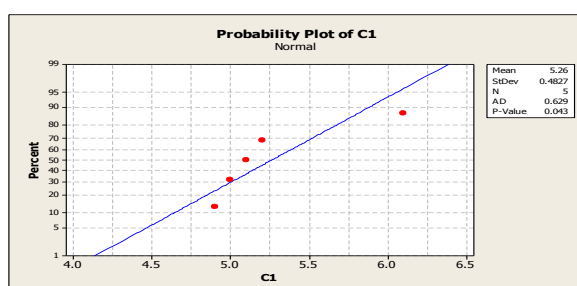
Boxplot test:- No outliers

$$\text{Free fatty acid \%} = \left( \frac{\text{mL } 0.1 \text{ N NaOH} \times 28.2}{\text{Sample Wt., g}} \right)$$

$$\text{Free fatty acid} = \left( \frac{26.72 \times 0.05 \times 28.2}{7.37} \right) = 5.1\%$$

**-Free fatty acid content without oleic acid**

FFA
4.9
5
5.1
5.2
6.1





**Fig.(11) Statistical calculation for readings Free fatty acid content without oleic acid**

**Statistical calculation for readings:-**

Mean	StDev	RSD%	Minimum	Maximum
5.260	0.483	9.18	4.900	6.100

**Table. (13) Statistical calculation for readings Free fatty acid content without oleic acid**

**Normality test :-** Not normal distribution

**Boxplot test:-** No outliers

$$\text{Free fatty acid \%} = \left( \frac{\text{mL } 0.1 \text{ N NaOH} \times 28.2}{\text{Sample Wt., g}} \right)$$

$$\text{Free fatty acid} = \left( \frac{5.26 \times 0.05 \times 28.2}{7.03} \right) = 1.0 \%$$

**-Pour point, upper pour point and cloud point**

**Table.(14) The statistical readings for Pour point, upper pour point and cloud point.**

Upper pour point	-5 °C
Pour point	-6 °C
Cloud point	9 °C



## -Dissection

**Table.(15) The Summary of the research results**

<b>C</b>	<b>Degree of unsaturation (g)</b>	<b>Free fatty acid content %</b>	<b>Viscosity CPS</b>	<b>Upper pour point °C</b>	<b>Pour point °C</b>	<b>Cloud point °C</b>
<b>Sunflower oil</b>	126.9	0.77	71.2	-9	-15	-2
<b>Olive oil</b>	48.65	0.41 without oleic acid. 4.6 with oleic acid	70	-11	-13	-1
<b>Used oil</b>	100.42	4.7	84-90	-10	-11	-3
<b>Biodiesel 1</b>	172.08	0.65	13	-4	-5	2
<b>Biodiesel 2</b>	72.50	1.0 without oleic acid. 5.1 with oleic acid	17	-5	-6	9
<b>Biodiesel 3</b>	143.06	2.1	17	-2	-9	1

The major physicochemical properties of two vegetable oils and used oil, and their biodiesels, are given in the table above; their quality parameters were indeed within the European standard EN 14214.

According to the FAME standard EN 14214, the iodine number specifying a limit for raw materials is 120. The iodine values of sunflower oil is greater than the maximum value set by the European



standard and other vegetable oils. Thus, the high iodine value of sunflower oil was expected, since it depended on the highest degree of unsaturation of sunflower oil. However, unsaturation in the fatty acid chain is considered the basic source of instability in vegetable oils (Stavinoha & Howell, 1999).

The free fatty acid content values for sunflower oil and its biodiesel were 0.77 and 0.65 respectively, which means there was a 15% decrease in the free fatty acid content after the transesterification process and this decrease due to using of alkali catalyst (saponification).

The free fatty acid content values for olive oil with oleic acid and its biodiesel were 4.6 and 5.1 respectively which means there no loss in its free fatty acid content during the transesterification processing. For use oil and its biodiesel, the free fatty acid values were 4.7 and 2.1 respectively which means there are 55% decrease in the free fatty acid content of the sunflower oil after the process.

### **Conclusion and Future research**

A complete set of characteristics for a variety of vegetable oils (sunflower oils and olive oil) with used vegetable oil and its biodiesel are presented. Characteristics tests on these vegetable oils show that they have small free fatty acid content and a high degree of unsaturation which is suitable for biodiesel, but their viscosities were very high then they were converted to biodiesel by transesterification process.

Characteristics tests and Performance tests on their biodiesels demonstrated that these biodiesels are similar to diesel fuel, as the heat of



combustion for the biodiesel fuels and diesel fuel gave the same results. In general, all produced biodiesels has almost the same heat content. Also, the viscosities of the biodiesels were smaller than the vegetable oils with a percentage of 81%. The cloud and pour points for biodiesel fuels are slightly higher than vegetable oils.

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