

# Production and characterization of biodiesel from locally sourced sesame seed oil, used cooking oil and other commercial vegetable oils in Erbil-Iraqi Kurdistan

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**Abstract:** Production of biodiesel from locally produced sesame oil and commercially available sunflower oil, corn oil, and hazelnut oil and used cooking oil was carried out by transesterification method. Effect of different parameters was studied; including effect of temperature, oil to alcohol ratio, time of transesterification, and type and amount of alkaline catalyst in order to achieve the optimum condition to obtain the highest conversion. The results indicate that 60°C, 1:6 oil to methanol ratio, 2hrs reaction time and 1.5 wt % KOH were optimum conditions for transesterification to achieve the highest conversion of the vegetable oils to the corresponding biodiesel. The obtained biodiesels were analyzed using ASTM methods to determine the characteristic fuel properties; kinematic viscosity, specific gravity, flash point, pour and cloud point, water content, total sulfur content, acid number and cetane number. For comparison, two commercially available petro-diesels were analyzed using the same tests used for the produced biodiesels.

**Keywords:** Biodiesel, Sesame Seed Oil, Used Cooking Oil, Transesterification

## 1. Introduction

The major part of all energy consumed worldwide comes from fossil sources (petroleum, coal and natural gas). However, these sources are limited, and will be exhausted by the near future. Thus, looking for alternative sources of new and renewable energy such as hydro, biomass, wind, solar, geothermal, hydrogen and nuclear is of vital importance. Vegetable oils have become more attractive recently because of their environmental benefits and the fact that they are made from renewable resources [1-4].

In recent years there has been increasing interest in developing biodiesel as an alternative to fossil fuels, mainly in the automotive sector, because of its compatibility with current available engines. Its energy content and the physical and chemical properties are similar to conventional diesel fuel, allowing its use either on its own or mixed with conventional diesel in any diesel engine without requiring any modifications either to the ignition system or the fuel injector [5].

Biodiesel fuel (BDF) is defined as the mono-alkyl esters of long-chain fatty acids synthesized by transesterification of triglyceride in vegetable oils or animal fats with alcohol, and is therefore a renewable energy resource [6].

BDF is superior to conventional diesel in terms of its sulfur content, aromatic content and flash point. It is essentially sulfur free and non-aromatic while conventional diesel can contain up to 500 ppm SO<sub>2</sub> and 20–40 wt% aromatic compounds. These advantages could be a key solution to reduce the problem of urban pollution since transport sector is an important contributor of the total gas emissions. Amongst vehicle fuels, diesel is dominant for black smoke particulate together with SO<sub>2</sub> emissions and contributes to a one third of the total transport generated greenhouse gas emissions [7].

BDF has higher oxygen content than petro-diesel and its use in diesel engines have shown great reduction in emission of particulate matter, carbon monoxide, sulfur, polyaromatics, hydrocarbons, smoke and noise. In addition, burning of

vegetable oil based fuel does not contribute to net atmospheric CO<sub>2</sub> level because such fuel is made from agricultural materials which are produced via photosynthetic carbon fixation. The substitution of conventional diesel fuels with fatty acid methyl esters already comprises a commercial activity in many countries of the world. The use of biodiesel has not expanded into developing countries, due to the higher prices than conventional diesel. The higher cost of biodiesel is due to its production mostly from expensive high quality virgin oil. Use of low cost feedstock such as waste frying oils and non-edible oils in an acid-catalyzed process should help to make biodiesel competitive in price with petroleum diesel [8, 9].

The annual worldwide demand for diesel fuel approaches 1.0 billion tons. This far exceeds the current and future biodiesel production capabilities of the vegetable oil and animal fat industries. Worldwide, vegetable oil feedstocks are estimated to be 100 million tons, which would supply only 10% of the demand for diesel fuel even if none were diverted to other uses [10].

Because BDF is oxygenated, it is a better lubricant than diesel fuel, increasing the life of engines, and is combusted more completely. Indeed, many countries are introducing biodiesel blends to replace the lubricating effect of sulfur compounds in low-sulfur diesel fuels [11].

In addition, this biofuel is completely miscible with petroleum diesel, allowing the blending of these two fuels in any proportion. BDF can be used neat or blended in existing diesel engines. However, differences in the chemical nature of biodiesel (mixture of mono-alkyl ester of saturated and unsaturated long chain fatty acids) and conventional diesel fuel (mixture of paraffinic, naphthenic and aromatic hydrocarbons) result in differences in their basic properties, affecting engine performance and pollutant emissions. BDF, produced from any vegetable oil or animal fat, generally has higher density, viscosity, cloud point and cetane number, and lower volatility and heating value compared to commercial grades of diesel fuel [12].

BDF is generally made of methyl esters of fatty acids produced by the transesterification reaction of triglycerides

with methanol in the presence alkali as a catalyst [13, 14]. The purpose of the transesterification process is to lower the viscosity of the oil. Among the alcohols that can be used in the transesterification reaction are methanol, ethanol, propanol, butanol and amyl alcohol. Methanol and ethanol are used most frequently, ethanol is a preferred alcohol in the transesterification process compared to methanol because it is derived from agricultural products and is renewable and biologically less objectionable in the environment despite the low cost and physical and chemical advantages of methanol. The transesterification reaction can be catalyzed by alkalis, acids, or enzymes [15, 16].

Generally, two types of raw materials have been used in the biodiesel processes as vegetable oils and animal fatty acids. Some vegetable oils such as sunflower oil, olive oil, soybean oil, cotton oil, hazelnut oil, rubber seed oil, mahua oil, jojoba oil, tobacco seed oil, rapeseed oil, etc. have usually been used for producing biodiesel fuel. Also, tall oil distilled was esterified with alcohol and tested in a direct injection diesel engine at full load condition [17]. This work aims firstly to obtain biodiesel from locally produced sesame seed oil and commercially available corn, hazelnut, sunflower, and used cooking oils. The second, to determine the physical and chemical properties of the produced biodiesels according to ASTM methods. The third, to compare the properties of the produced biodiesels with the commercially available petrodiesels No.1 and No.2.

## 2. Methods and Materials

### 2.1. Materials

Different vegetable oils were used as feedstocks; locally produced sesame seed oil, commercial corn oil, sunflower oil, hazelnut oil and used cooking oil.

The chemical and physical properties of these vegetable oils were characterized as shown in Table (1). Methanol, KOH and NaOH were obtained from Aldrich Company Ltd and used as alcohol and catalyst for transesterification.

**Table 1.** Physicochemical properties of commercial vegetable oils.

No	Characteristics	Unit	Test Method	Sesame seed oil	Used oil	Corn oil	Sunflower oil	Hazelnut oil
1	Kinematic Viscosity @40°C	mm <sup>2</sup> s <sup>-1</sup>	ASTM D445	33.61	37.61	33.54	32.9	36.17
2	Density @15.5°C	g cm <sup>-3</sup>	ASTM D1298	0.936	0.931	0.924	0.915	0.945
3	Specific gravity	----	ASTM D1217	0.937	0.9399	0.92499	0.91597	0.94600
4	Total Sulfur Content	mass%	ASTM D4294	0.0167	0.0387	0.0290	0.0279	0.0309
5	Flash point	°C	ASTM D92	312	330	309	301	325
6	Pour point	°C	ASTM D97	-3.0	+3.0	-6.0	-6.0	-3.0
7	Cloud point	°C	ASTM D2500	+3.0	+9.0	+1.0	+2.0	+1.0
8	Water content	vol%	ASTM D6304	0.00	0.00	0.00	0.00	0.00
9	Acid number	mg KOH g <sup>-1</sup>	ASTM D664	0.443	0.458	0.410	0.402	0.395

Diesel was taken from commercial diesel (No.1 and No.2) with their characteristics listed in Table (2). Batch stirred reactor of 1.0 L volume is used for preparation of biodiesel from oil seeds using transesterification. Batch stirred reactor consists of three necks for stirrer, condenser and inlet of

reactant as well as for placing the thermocouple to observe the reaction temperature. Process parameters such as reaction temperature, reaction duration, amount of catalyst and volume of methanol were optimized in 1.0 L batch stirred reactor.

## 2.2. Methodology for Analyzing Fuel Characteristics of BDF

The physical properties of the raw samples, biodiesel and its blends with petro-diesel were measured by using American Society for Testing and Materials “ASTM” standard methods, including kinematic viscosity (using U tube viscometer ” SCOTT CT52”, ASTM D 445), flash point (using Tanaka

ACO-7, ASTM D 92), pour point and cloud point (ASTM D 97/2500), acid value (ASTM D-664), density (ASTM D1298), specific gravity (ASTM D1217) , total sulfur content (using Tanaka RX-360SH, ASTM D4294), water content (ASTM D6304), cetane number ( ASTM D 613), distillation temperature@90% (ASTM D86), aniline point (ASTM D 611), diesel index and cetane index (ASTM D4737).

**Table 2.** Physicochemical properties of commercial petro-diesel fuels.

Nº	Characteristics	Unit	Test Method	Petro-diesel Nº(1)	Petro-diesel Nº(2)
1	Kinematic Viscosity @40°C	mm <sup>2</sup> sec <sup>-1</sup>	ASTM D445	2.950	2.015
2	Density @15.5°C	g cm <sup>-3</sup>	ASTM D1298	0.8444	0.8197
3	Specific gravity @ 60°F/60°F	----	ASTM D1217	0.8453	0.8205
4	Total Sulfur Content	mass%	ASTM D4294	1.0986	0.4592
5	Flash point	°C	ASTM D92	66	68
6	Pour point	°C	ASTM D97	-15.0	-18.0
7	Cloud point	°C	ASTM D2500	-6.0	0.0
8	Cetane Number	----	ASTM D613	49.7	46.4
9	Water content	vol%	ASTM D6304	0.00	0.00
10	Acid number	mg KOH g <sup>-1</sup>	ASTM D664	Nil	Nil
11	Distillation temperature@90% Recovered, vol%	°C	ASTM D86	345	353
12	Aniline point	°C	ASTM D 611	70	71
13	Diesel Index	----	----	56.73	65.43
14	Cetane Index	----	ASTM D4737	50.84	57.11

## 2.3. Transesterification

The transesterification was carried out in 1.0 L three-neck glass flask connecting with a reflux condenser using tap water to condense methanol vapor and a thermometer. The mixture was agitated by using a stainless steel stirrer. The reactor was placed in a heated water bath. KOH pellets were dissolved in methanol before being poured in the reactor containing about 200 g oil sample heated up to a desired temperature, which was 40°C, 50°C , 60°C , 70°C or 80°C. The reaction was kept at a desired temperature for 30 min, 60 min, 90 min, 120 min or 150 min. The molar ratio of methanol and oil sample varied

from 5:1 to 12:1 while the amount of KOH catalyst was ranging from 1.4 wt% to 1.8 wt% of the oil sample.

After a certain time, the mixture was poured into a separating funnel. The ester layer was separated by gravity and located in the upper layer. The glycerol, extra methanol and undesired products were in the lower layer and were decanted. The ester layer was washed several times with a small amount of hot water each until the washings were neutral. Table (3) shows the chemical and physical properties of the biodiesel obtained from these vegetable oils under the selected optimum conditions.

**Table 3.** Physicochemical properties of biodiesel from different sources of vegetable oils.

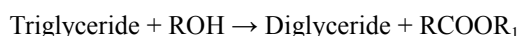
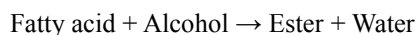
Nº	Characteristics	Unit	Test Method	Sesame seed oil	Used oil	Corn oil	Sunflower oil	Hazelnut oil
1	Kinematic Viscosity @40°C	mm <sup>2</sup> s <sup>-1</sup>	ASTM D445	4.580	5.580	4.300	5.100	5.21
2	Density @15.5°C	g cm <sup>-3</sup>	ASTM D1298	0.8972	0.8961	0.8963	0.9001	0.9012
3	Specific gravity	----	ASTM D1217	0.8981	0.8970	0.8972	0.9010	0.90216
4	Total Sulfur Content	mass%	ASTM D4294	0.0184	0.0137	0.0120	0.0144	0.0012
5	Flash point	°C	ASTM D92	155	150	158	154	165
6	Pour point	°C	ASTM D97	-5.0	-6.0	-12.0	-12.0	-6.0
7	Cloud point	°C	ASTM D2500	+1.0	+3.0	-3.0	-3.0	+1.0
8	Cetane Number	----	ASTM D613	69.3	74.6	74.8	69.2	69.2
9	Water content	vol%	ASTM D6304	0.00	0.00	0.00	0.00	0.00
10	Acid number	mg KOH g <sup>-1</sup>	ASTM D-664	0.32	0.35	0.28	0.297	0.351
11	Distillation temperature@90% Recovered, vol%	°C	ASTM D86	347	346	345	351	362

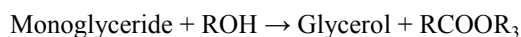
## 3. Results and Discussion

### 3.1. Production of Biodiesel under Different Procedure Conditions

First biodiesel was produced from transesterification of locally manufactured sesame seed oil by methanol as transesterification alcohol and potassium hydroxide KOH and

sodium hydroxide NaOH as alkaline catalysts by using basic procedure described in experimental section. The following scheme is the general steps of the transesterification of vegetable oil to produce methyl ester of fatty acids (biodiesel).





Several aspects, including alcohol/vegetable oil molar ratio, temperature, the type of catalyst, concentration and time of transesterification have an influence on the course of the transesterification. It was observed that the yield of produced biodiesel was influenced by these parameters and in order to fix the best and optimum reaction conditions the same transesterification procedure was followed to study the effect of each of these factors on production yield.

### 3.2. Effect of Reaction Temperature

Transesterification can occur at different temperatures, depending on the properties of oils. Transesterification of sesame oil was carried out using methanol as alcohol and KOH as alkaline catalyst at different reaction temperatures ranged between 40°C to 80°C. It was observed that at 60°C the yield of produced biodiesel was the highest value (%96) that means increasing temperature from 40°C to 60°C, the conversion increased 17.5% as shown in figure (1).

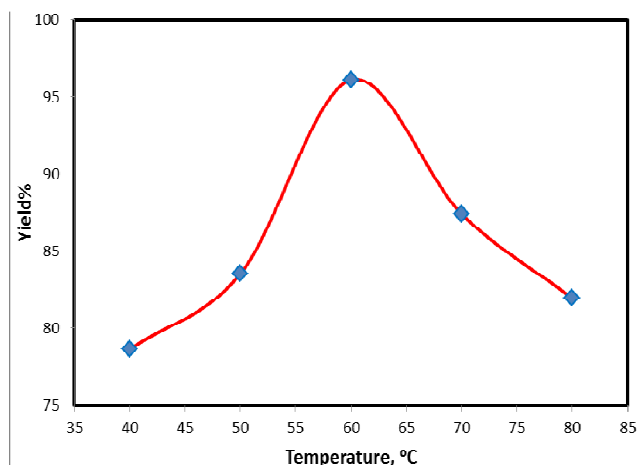


Figure 1. Effect of reaction temperature on the %BDF yield.

The effect of temperature firstly explains that the transesterification is an equilibrium process and this equilibrium is concentration dependence of alcohol; methanol and glycerol on the two sides of the reaction. Secondly at temperature higher than 60°C phase transfer may occur due to low boiling point of methanol compared to sesame seed oil and glycerol.

### 3.3. Effect of Methanol to Oil Ratio

The stoichiometric ratio for transesterification reaction requires 3 mole of alcohol and 1 mole of triglyceride to yield 3 mole of fatty acid ester and 1 mole of glycerol.

The production yield of sesame biodiesel was studied using different mole ratios of methanol to sesame oil at the previously fixed optimum temperature (60°C) using KOH as alkaline catalyst. The best yield (%96) was achieved at 6:1 mole ratio (methanol: sesame oil) and there was no increasing in production yield when higher mole ratios were used as

shown in figure (2). This means that 6:1 mole ratio provides sufficient excess of methanol to shift transesterification equilibrium toward biodiesel product.

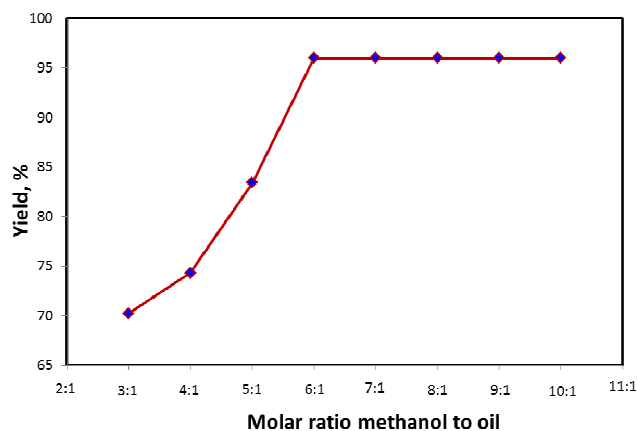


Figure 2. Effect of methanol to oil ratio on the %BDF yield.

### 3.4. Effect of (KOH) Catalyst Amount

Under the two fixed optimum values of temperature (60°C) and methanol: sesame oil mole ratio (6:1) the production yield of sesame biodiesel was examined by using different amounts of KOH. It was observed that % 1.5 KOH to sesame oil was enough to catalyze the transesterification process and gave desired alkaline medium since excess hydroxyl anion may increase the hydrolysis of ester linkage of biodiesel. Therefore the highest production yield was obtained at % 1.5 KOH as shown in figure (3).

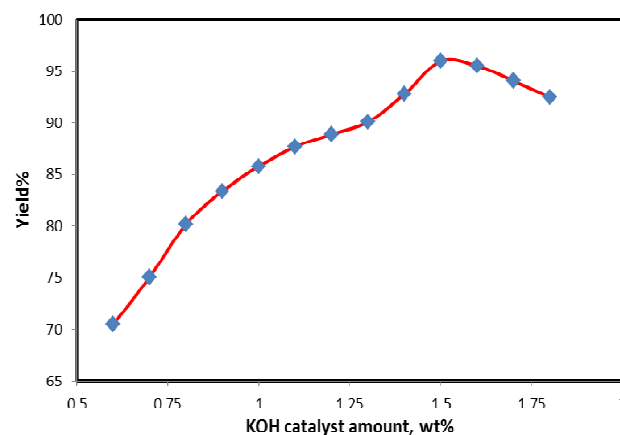


Figure 3. Effect of KOH catalyst amount on the %BDF yield.

### 3.5. Effect of Reaction Time

The previously fixed conditions as described in above sections were used to investigate the effect of reaction time on production yield and to determine the shorter enough time to obtain the higher quantity of biodiesel since for economical reasons, the best process is the one that reaches the highest conversion in the shortest period of time. Results obtained from the present experiments reveal that 120 min is sufficient for the completion of the reaction as shown in figure (4). The longer time decreases the production yield and this may be due

to increasing the probability of biodiesel hydrolysis under alkaline condition and at relatively high temperature. Therefore it is of great importance to immediate separation of the produced biodiesel from traction mixture after preceding the required reaction time.

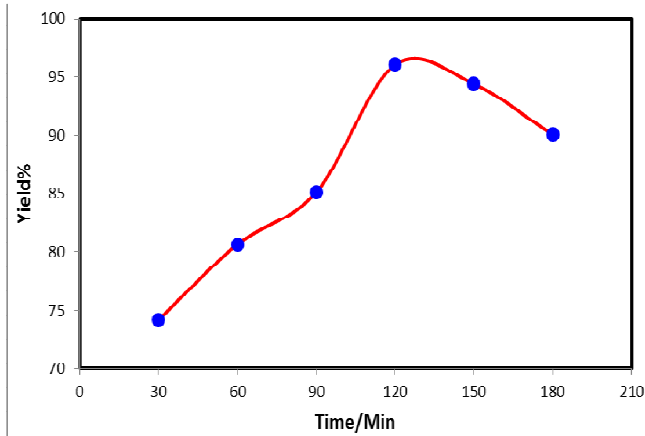


Figure 4. Effect of reaction time on the %BDF yield.

### 3.6. Effect of Catalyst Type

In another experiment sodium hydroxide NaOH was used instead of KOH as alkaline catalyst under the previously described optimum conditions in order to investigate the effect of alkaline catalyst on production yield of biodiesel. Figure (5) shows that %1.5 ratio of NaOH was the best amount to achieve the highest production yield( % 95) which was relatively lower than the production yield when KOH was used as catalyst (%96).

Under the optimal conditions described above and with respect to reaction temperature, methanol to oil ratio, catalyst amount, reaction time and catalyst type the transesterification of commercially available corn oil, sunflower oil, hazelnut oil and used cooking oil was carried out producing the corresponding biodiesels in order to compare the characteristic properties of sesame biodiesel with biodiesels obtained from indicated vegetable oils.

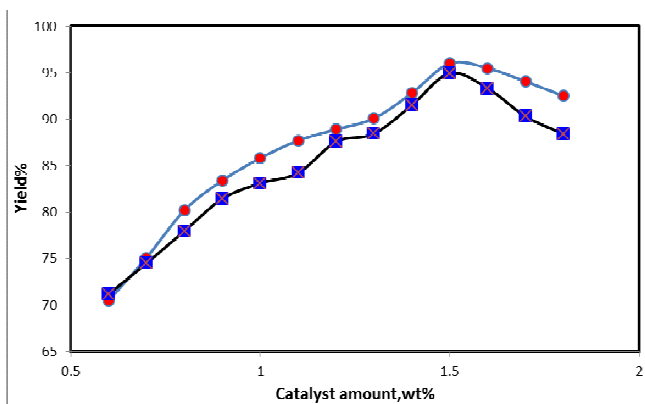


Figure 5. Effect of catalyst type on the %BDF yield.

### 3.7. Fuel Properties of the Biodiesel

The product of above transesterification was analyzed,

taking into consideration specifications for biodiesel as fuel in diesel engines. These results are shown in table (3) and compared to the property of commercial PD table (2) and the property of vegetable oils table (1).

Vegetable oils can be used as fuel for combustion engines, but its viscosity is much higher than usual petro diesel fuel. Viscosity is the most important property of biodiesel since it affects the operation of fuel injection equipment, particularly at low temperatures when the increase in viscosity affects the fluidity of the fuel. High viscosity leads to poorer atomization of the fuel spray and less accurate operation of the fuel injectors.

The viscosity values of vegetable oils were between  $32.9 \text{ mm}^2 \text{ s}^{-1}$  for corn oil and  $37.61 \text{ mm}^2 \text{ s}^{-1}$  for used cooking oil as shown in table(1), whereas those of the obtained biodiesels were between  $5.58 \text{ mm}^2 \text{ s}^{-1}$  for used cooking oil and  $4.30 \text{ mm}^2 \text{ s}^{-1}$  for corn oil as shown in Table (3). The viscosity of the vegetable oils was decreased considerably by seven to eight times after transesterification process. Compared to locally available PD No.1 (2.93) and No.2 (2.01) as shown in table (2). All of the obtained biodiesels were twice viscous than commercial PD.

The density values of vegetable oils were between  $0.945 \text{ g cm}^{-3}$  for hazelnut oil and  $0.915 \text{ g cm}^{-3}$  for sunflower oil while those of obtained BD were between  $0.901 \text{ g cm}^{-3}$  for hazelnut biodiesel and  $0,896 \text{ g cm}^{-3}$  for sesame and corn biodiesel. The density values of the BD considerably decreased via transesterification process, but still are higher than the density of the commercial PD fuel ( $0.844 \text{ g/cm}^3$  for No.1 and  $0.819 \text{ g/cm}^3$  for No.2).

The dangerous effects of sulfur in fuel for internal combustion engines is well known in damaging the diesel engines and harmful effect of emitted sulfur oxides after the combustion of fuel containing sulfur on air environment. The results from table (2) showed high levels of sulfur in commercially available PD No.1 (1.099 %mass) and for No.2 (0.460 %mass). Table (1) showed the sulfur content of vegetable oils as feedstock oils used in this work, the lowest was for sesame oil (0.0167 %mass) and the highest sulfur content was observed in used cooking oil (0.038 %mass). Table (3) showed the sulfur content of the derived biodiesels obtained from transesterification of vegetable oils, the results show considerable decrease of sulfur content in biodiesels by 2-3 factors compared to the corresponding vegetable oils except the biodiesel derived from sesame oil.

The flash point values of BD are highly lower than those of vegetable oils. The flash point of the vegetable oils was between  $301^\circ\text{C}$  and  $330^\circ\text{C}$ , while the derived biodiesel had much lower flash points and were between  $150^\circ\text{C}$  to  $165^\circ\text{C}$ . This could referred to highly decreasing of the molecular weight of the biodiesels compared to the molecular weight of corresponding vegetable oils due to splitting of one molecule of triglyceride to the fatty acid esters as a result of the transesterification.

Two important parameters for low temperature applications of a fuel are cloud point (CP) and pour point (PP). The CP is the temperature at which wax first becomes visible when the

fuel is cooled. The PP is the temperature at which the amount of wax out of solution is sufficient to gel the fuel, thus it is the lowest temperature at which the fuel can flow. Generally biodiesel has higher CP and PP compared to conventional diesel.

The cloud point of vegetable oils was ranged between 1°C for corn and hazelnut oils and 9°C for used cooking oil, while the pour point was ranged between -6°C for corn and sunflower and 3°C for used cooking oil as shown in table (1). A considerable decrease in both cloud point and pour point was observed of the obtained biodiesels. The cloud point was ranged between -3°C for both corn and sunflower and 3°C for used cooking oil as shown in table (3). The lowest pour point related to sunflower (-12°C). All derived biodiesels showed lowering in pour point by 2-3 folds compared to the corresponding oils.

Cetane number (CN) is another important property of diesel fuel and it is a measure of ignition quality of diesel fuel. The higher the CN, the easier the fuel ignites when it is injected into the engine. BD has a higher CN than petro-diesel because of its higher oxygen content. This means that engines run smoother and create less noise when running on BD.

Cetane number of the obtained BD was higher than commercial PD. As shown in table (3) the value of BD was between 69 and 74, while the cetane number of PD was 49.7 for No.1 and 46.4 for No.2 and this could be referred to the absence of aromatic hydrocarbons in biodiesel since aromatics lowers the cetane number. Table (3) shows also that the biodiesel derived from used cooking oil has high CN (74.6) compared to the other, and this may be due to the presence of high content of saturated straight-chain hydrocarbons since saturated straight-chain hydrocarbons have higher CN compared to branched chain or aromatic compounds of similar molecular weight and number of carbon atoms.

The distillation temperatures at 90% recovery for biodiesel derived from sesame, used and corn oils were in the similar range (347°C to 345°C) as shown in table (3) and analogous to distillation temperatures of commercial PD No.1 (345°C). The distillation temperatures at 90% recovery of biodiesel derived from sunflower and hazelnut are relatively higher (351°C and 362°C) respectively and analogous to distillation temperatures of commercial petro diesel No.2 (353 °C).

## 4. Conclusion

The highest conversion obtained from transesterification of first locally produced sesame oil and commercial available sunflower, corn and hazelnut oils was 96%. The conversion was highly affected by reaction temperature; the optimum temperature was 60°C and reaction time; the minimum time was 120 min to achieve the highest conversion. Also oil to alcohol mole ratio was another significant parameter which must be taken into account in transesterification process beside the type and amount of alkaline catalyst. The conversion (96%) was achieved using 1.5 wt% of KOH. After performing the analysis it was concluded that the produced biodiesels have considerable low sulfur content ranged from

30% to 60% compared to PD No.1 and 14% to 28% compared to PD No.2 since low sulfur content fuels have important environmental concern. An increase of cetane number from 1.4 to 1.6 of produced biodiesels compared to commercially available PD also has significant importance in evaluation of fuel efficiency. The most disadvantages of biodiesels are firstly high viscosity which was about 10 folds more viscous compared to PD and this require some modifications in engine system and secondly high pour point of produced biodiesels which ranged between (-6 and -3) compared to PD which was -15 for PD No.1 and -18 PD No.2 and this limits the use of biodiesel in very cold weathers.

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