

## ORIGINAL ARTICLE

# Utilization of Waste Sunflower Oil Biomass as Biodiesel: Its physical and chemical properties Identification and Implementation

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### ABSTRACT

The study was carried out to investigate the biodiesel production from waste sunflower oil, its characteristics and exhausted emission quality. Biodiesel was prepared to come after transesterification bioprocess from waste cooking sunflower oil using methanol and base-catalyst (NaOH). Several parameters were evaluated for the optimum formation of biodiesel like alcohol:oil molar ratios, different catalyst concentrations, temperatures and stirring speed. The fatty acid methyl esters found in the biodiesel were methyl palmitate, methyl linoleate, methyl oleate and methyl stearate. The viscosity of the biodiesel produced was within the range of international ASTM standards. A multi-element analysis of the biodiesel showed that it's met the specifications of the ASTM standard. The emission tests of biodiesel gave the carbon monoxide and hydrocarbon emissions and showed lower than that of petrodiesel. However, the nitrogenous oxides emission and specific fuel consumption were higher than that of conventional diesel fuel. It can be concluded that biodiesel made from waste sunflower oil can be considered as a great potential source of commercial biodiesel provided some pre-treatment steps are carried out.

Keywords: Biodiesel; sunflower oil; viscosity; emission; fuel consumption

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### INTRODUCTION

Waste cooking vegetable oils can be utilized for the production of biodiesel such as sunflower, palm, soybean, coconut, rapeseed, safflower and cottonseed oils [1-6]. These all sources are considered to have the potential resources as raw materials for the production of biodiesel. Diesel derived from rapeseed oil is the most common biodiesel available in Europe, while biodiesel from soybean and palm oil are dominant in the United States and Malaysia, respectively [2, 7, 8]. It can be also derived from renewable biomass feedstock, such as algae and animal fats, biodegradable (approximately 4-5 times as fast as petroleum diesel [9]). There are no restrictions on the use of other types of vegetable oils.

Within several methods to produce biodiesel, transesterification is one of the most used ways to produce biodiesel. Transesterification is the process of reacting a triglyceride molecule with an excess of alcohol in the presence or absence of a catalyst to produce fatty acid methyl esters and glycerol [10]. The purpose of the transesterification of vegetable oils to their methyl esters process is to get the lower viscosity of the oil. Since the viscosity is the most vital characteristics to assess the biodiesel quality and its effects on the operational injecting equipment. Consequently, fuel consumptions and gases emission rate are important as well. The increase of viscosity makes incomplete combustion and poorer atomization of the fuel spray due to the low fluidity of the fuel [11]. The lower viscosity biofuel can be generated by using transesterification reaction of triacylglycerides oil with an alcohol in the presence of an alkali or acid catalyst [12].

Biodiesel fuel is an environmentally friendly fuel that can be used in diesel engines with little or without any significant modifications [13]. It also releases fewer exhaust gases compared to petrol diesel fuel or petrodiesel [13]. Biodiesel is also non-flammable, non-toxic and non-explosive. It has a high flash point (with a flash point of 423 K as compared to 337 K for petro diesel), has a higher heat content has excellent lubricity [10]. Studies have shown that biodiesel reduces the emission of carbon monoxide, sulphur, polyaromatics, unburned hydrocarbon, smoke, noise and particulate emissions compared to regular diesel fuel [10]. Therefore, the objectives of this study were to produce biodiesel from waste cooking sunflower oil, to evaluate the different variables affecting the alkaline methanolysis of waste oil and to determine the characteristics of the biodiesel, for example, to identify methyl esters present in the biodiesel, the exhaust emissions and fuel consumption by engines.

## MATERIALS AND METHODS

### Preparation of waste sunflower oil

Sunflower oil was purchased from Midvalley and sent to the food stall for cooking purposes. The waste sunflower cooking oil was then collected and filtered to remove food residues and solid precipitates. This process was done by pouring the Waste Vegetable Oil (WVO) into a filter funnel covered with a layer of filter paper and the filtered oil was left to drip into a conical flask. For the transesterification process, it is important that the oil contained minimal amounts of water because water will react with the catalyst and cause problems with soap formation and separation of the glycerin layer.

### Transesterification reaction

The transesterification reactions were performed using waste sunflower cooking oil. The cooking oil was first heated to 60 °C in an incubator to liquify the oil and to remove water from the waste sunflower oil. The oil was not heated above 65 °C to prevent methanol evaporation<sup>14</sup>. The methanol and dissolved catalyst were then added to 56 ml of waste sunflower oil and the mixture was stirred vigorously. The reaction mixture was stirred for particular reaction time in an incubator shaker at room temperature. At this stage, the waste sunflower oil is converted to its corresponding esters.



### Purification of the methyl esters

Once the reaction is complete, two major products exist glycerin and biodiesel. Each has a substantial amount of excess methanol that was used in the reaction. Excess methanol was used in the biodiesel formation to ensure the total conversion of the oil to its esters. The reaction mixture was poured into a beaker and left under a fan for about 20 minutes to dry. Excess methanol in the mixture is then removed via evaporation. Phase separation occurs at this stage. The reaction mixture was then poured into a separating funnel. The glycerin layer is much denser than biodiesel layer. As a result, the two layers will be separated by gravity forces, where glycerin is at the bottom. Glycerin can be drawn off the bottom of the separating funnel, while the methyl esters (biodiesel) form the upper layer (Fig. 1).

Once the glycerin and biodiesel layers have been separated, the glycerin layer was removed and the mixture was neutralized with an acid. An emulsion will occur if water is present. Water should be removed as far as possible in the catalyst ingredients. If water is present, large clumps of caustic soda or sodium hydroxide can form and these are hard to break up. Once separated from the glycerin, the biodiesel is purified by washing gently with warm distilled water to remove residual catalyst or soaps, this procedure is continued until the methyl ester layer becomes clear. It is then dried by using anhydrous sodium sulphate to remove all the water and sent to storage. Then the finished biodiesel will be analyzed to assess the ASTM specifications.

### Conditions for biodiesel production

Methanol was used for the transesterification reaction to produce biodiesel from waste sunflower oil. Five different molar ratios of oil to alcohol, 4:1, 3:1, 1:3 1:4 and 1:6 were used in this reaction. Sodium hydroxide was used in the experiments as a catalyst. Four different percentages of catalysts by weight of oil were used to produce biodiesel. These were 0.05 g, 1.00 g, 1.50 g and 2.00 g of NaOH. The temperature

range was between 25 °C - 62.5 °C and the reaction time range was between 0.5 - 8.5 hours. Lastly, the stirring speed was varied between 50 - 300 rpm.

### **Biodiesel analysis**

#### **Gas chromatography-mass spectrometry (GC-MS) analysis**

The specific component of methyl esters in the biodiesel was determined using an Agilent 6890 Gas Chromatography installed with a mass spectrometry detector. A capillary column, (length: 30 m, film thickness: 0.25µm and ID: 0.25 mm) was used. Helium was used as the carrier gas. One micro-litre of the biodiesel sample was injected manually. Samples were introduced to the column via an inlet. Once in the inlet, the heated chamber vaporizes the biodiesel and a constant flow of helium moves through the inlet. A portion of the helium flow acts to transport the sample into the column. Another portion of the helium flow gets directed to purge the inlet of the sample following injection. Yet another portion of the flow is directed through the split vent in a split ratio.

#### **Multi-element concentration**

##### **Engine exhaust emission test**

The exhaust emission test was also conducted to evaluate the exhaust emission characteristics of the biodiesel. For these, the 2.50 litres of biodiesel was required to perform the engine emission test. The BOSCH gas analyzer model EET 008.36 was used to measure carbon monoxide (CO) and unburned hydrocarbon (HC). Whereas, the Bacharach model CA300NSX analyzer was used to measure NO<sub>x</sub> concentration. The measurement of CO and NO<sub>x</sub> were according to SAE J117 June'95 standards and HC to SAE J215 March'95 standards. Fuel consumption of the biodiesel was also measured and compared with the fuel consumption of normal petrodiesel. The model of the engine was YANMAR TF120-M. This engine is a horizontal, water-cooled and single cylinder. The accumulation of deposit was carried out for 8 hours at 2000 rpm constant engine speed and 15 Nm load for each test fuel.

## **RESULTS AND DISCUSSION**

With regard to catalyst concentration, the optimum value for alkaline transesterification is 2% (Fig. 3).<sup>15</sup> It was reported that as the catalyst concentration increases, the biodiesel yield increases as well as a maximum concentration of 1%. When it exceeded 1%, a decrease was observed. But from above Figure, it can be seen that maximum biodiesel has been achieved by 2% NaOH. When the catalyst concentration was below 2%, emulsion or soap (due to saponification reaction with sodium hydroxide) has been formed and hence increase the viscosity of the whole solution and finally results in the formation of a gel. These explanations referred that a catalyst concentration of 2% is optimum for transesterification. Fig. 4 showed the maximum biodiesel yield was achieved using a molar ratio of 1:6 for oil:methanol. Theoretically, an oil/methanol ratio of 1:3 was enough to form methyl esters from the reaction of methanol with triglycerides. According to many studies, a ratio of 1:6 was sufficient for the high yield (almost complete) of methyl esters [16].

### **Biodiesel analysis**

#### **Gas chromatography-mass spectrometry (GC-MS) analysis for chemical composition of waste cooking oil**

Gas chromatography-mass spectrometry (GC-MS) was used. The gas chromatograph utilized a capillary column which depended on the column's dimensions (length, diameter, film thickness) as well as the phase properties. The variation in the chemical characteristics of various compounds in a solution has divided the compounds as the sample moved to the length of the column. Different molecules took different periods to wash out with a solvent from the gas chromatograph which was known as the retention time, and consequently, this led the mass spectrometer to find out the ionized molecules separately [17]. This is generally happened by mass spectrometer where each molecule has been broken into ionized fragments. The way a compound splits into fragments was characteristic of its structure. The fatty acid methyl ester (FAMES) profile in the produced biodiesel is shown in Fig. 5. It can be seen that there are a total 4 compounds that are present in the biodiesel sample produced from waste sunflower cooking oil. Details of these compounds are shown in the library search report (Table 1) containing the retention time, area (%), possible identities, and the qualities of the compounds. Each of the peaks of the solution has its unique retention time which is important in determining the chemical compounds in the biodiesel sample [18].

The FAMES are separated according to carbon number (the number of carbon atoms in the fatty acid chain, not including the methyl ester carbon) and according to the degree of unsaturation. The position of the double bond(s) and their configuration (cis or trans) are also important parameters and adds additional information to the characterization of the biodiesel. Better separation was done (Fig. 6), the

yields of isolated FAMES were methyl palmitate, methyl linoleate, methyl oleate and methyl stearate (Table 1). As shown in Table 1, the biodiesel consists mainly of 9-octadecenoic acid (z)-, methyl ester (methyl oleate) (i.e. C18:1) and 9, 12-octadecadienoic acid (z, z)-, methyl ester (methyl linoleate) (i.e. C18:2). The biodiesel produced composed mainly 18 carbon fatty acids. Besides linear saturated fatty acids, di-unsaturated and polyunsaturated fatty acids were seen [19]. As can be seen the mass spectrum (Fig. 5) of the highest peak indicated that the compound present was linoleic acid methyl esters.

### **Characterization of the biodiesel**

#### **Kinematic viscosity**

From Table 2, the viscosity of the biodiesel produced from transesterification of waste sunflower oil was 4.74 cSt. This kinematic viscosity value at 40 °C is limited between 1.9 to 6.0 CST according to the American Society for Testing and Materials (ASTM D445). Therefore, the waste sunflower oil-based biodiesel met the specification of the ASTM standards. The viscosity value of waste cooking oil is decreased after transesterification and prevented operational problems such as engine deposits [8, 10]. The viscosity values of vegetable oils vary between 23.2 and 53.0 cSt, 9 to 17 times greater than petroleum diesel fuel [10]. The kinematic viscosity is a basis design specification for the fuel injectors used in diesel engines [20]. The viscosity of biodiesel depends on each component of the solution also and its chain length of either the fatty acid or alcohol moiety in a fatty acid methyl [21]. Therefore, these chemical compositions of sunflower biodiesel are suitable for maintaining the desired viscosity as far ASTM.

#### **Total acid number (TAN) and total base number (TBN)**

The total acid number (TAN) is expressed as milligrams potassium hydroxide per gram of sample, which is required to titrate a sample to a specified endpoint. The total acid number is a direct measure of free fatty acids present in biodiesel sample. If the free fatty acid (especially unsaturated fatty acids) content of the biodiesel is higher, then the oxidation stability of biodiesel will be less [14]. The total base number (TBN) was a measure of biodiesel's reserve alkalinity. It was measured in milligrams of potassium hydroxide per gram (mg KOH/g), which assists in the control of acids produced during the explosion process, reduces the affinity of sludge buildup and improves lubricity characteristics of the oil. TBN determined how effective the control of acids was formed during the combustion process. The ASTM standard approved a maximum acid value for biodiesel of less than 0.8 mg KOH/g and 10-15 mg KOH/g of total base number for diesel engine operations. TAN was 1.4 and base was 11 (Table 4). This might be due to the sample being less reactive or there was excessive base value. Free fatty acids are broken down from the triglycerides during cooking stage and this can affect the process of transesterification [23].

#### **Chemical element determination**

The result shown in Fig. 6 referred that only the magnesium values belong to the limits of ASTM D6751. Sodium hydroxide was utilized as catalysts in this experiment and anhydrous sodium sulfate was used as drying agent in the biodiesel production. That is way NaOH value is little high than ASTM. In addition, the European norm as well as the American standard also limits the maximum content of phosphorus in biodiesel samples to 10 ppm. Phosphorus level was found to be very high, 20.5, in the biodiesel produced from waste cooking sunflower oil. High level of phosphorus may due to the incomplete refining of the vegetable oil and from proteins encountered in the rendering process. Phosphorus in FAME stems may also come from phospholipids (straight vegetable oil) and inorganic salts (waste cooking oil) contained in the feedstock. In waste vegetable oils, the type of oil recovery strongly influences this parameter. Phospholipids, phosphorus sources, may be impeded by phase separation method during the transesterification process due to their emulsifying properties and residual phosphorus can also be removed by the distillation process. Metal elements such as phosphorus, magnesium, and calcium are present in the biodiesel can form ash deposits in fuel injection system and poison the catalytic converters of the engines and reduce its ability to decrease the exhaust emission [24]. An ASTM D6751 standard limit for sodium, magnesium and calcium concentrations is less than 5 ppm to prevent engine damage.

#### **Engine emission test**

##### **Carbon monoxide (CO) emission**

Table 4 showed that the carbon monoxide emissions test for both the produced biodiesel and conventional diesel. The test has been carried out at the engine speed of 2000rpm with 15 Nm load condition. Carbon monoxide and unburned hydrocarbons are the products of incomplete combustion caused by low cetane number of the fuel. The tendency of carbon monoxide formation is higher when there is insufficient oxygen inside the fuel or low reactivity with oxygen during the combustion process<sup>25</sup>. As shown in Table 4, the CO emission of the produced biodiesel was much lower than the diesel, with readings of, 0.025 and 0.028 vol. % respectively. The produced biodiesel 10.71 % lower amount of carbon monoxide emission compared to the diesel. Biodiesel itself has about 11% oxygen content in it and this

facilitates complete combustion. In addition, the diesel engine used in this experiment provided more air at high speed which increases the turbulence intensity in the combustion chamber and can affect the air-fuel mixing process. This probably led to a more complete combustion and thus the lower CO emission is obtained at high speed.

#### Hydrocarbon (HC) emission

Unburned hydrocarbon emission can increase due to the injection occurring too early or more fuel to contact with the relatively cool cylinder wall. Therefore, insufficient time is responsible for completion of combustion [26]. It was found that the biodiesel produced showed lower HC emission, 22.75 ppm, compared to the diesel, 25.33 ppm (Table 4). Its emission level was 10.19% lower than the diesel fuel. In this current experiment, the significant reduction of the unburned hydrocarbons is probably due to complete combustion of the sunflower biodiesel.



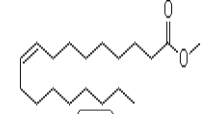
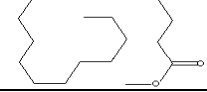
#### Nitrogenous oxide emission (NO<sub>x</sub>)

Biodiesel-fueled engines have the potential to emit more NO<sub>x</sub> as compared to that of diesel-fueled engines. The produced biodiesel showed a higher NO<sub>x</sub> emission, of 548.67 ppm, compared to the diesel (515.40 ppm) (Table 4). The NO<sub>x</sub> emission of the biodiesel was 6.46% which is slightly higher than the diesel. This increase might be due to the higher combustion temperatures and the higher oxygen content of the biodiesel, which can lead to better oxidation of the nitrogen, thus increasing the nitrogenous oxide emission [25, 26].

#### Specific fuel consumption (SFC)

Table 4 shows the variation of specific fuel consumption of the biodiesel versus the diesel fuel. It can be seen that the waste sunflower based biodiesel showed slightly higher fuel consumption (0.612 ml/sec) than that of diesel (0.588 ml/sec). At the 15 Nm load condition, the fuel consumption of biodiesel was more than 4.08 % than that of diesel. This is probably due to the oxygen content and consequently, the lower calorific value of the waste sunflower based biodiesel compared to petrodiesel [27-29].

**Table 1.** GC-MS, peak serial and chemical composition of sunflower biodiesel

Product Identification	Retention Time	Area	Quality	Formula	Chemical Structure
Hexadecanoic acid, methyl ester (Palmitic acid methyl ester)	20.453	6.33	98.3	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>14</sub> COOCH <sub>3</sub> or C <sub>16</sub> H <sub>31</sub> O <sub>2</sub> CH <sub>3</sub>	
9,12-octadecadienoic acid (z, z)-, methyl ester (Linoleic acid methyl ester)	23.654	48.12	99.1	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>4</sub> CH=CH(CH <sub>2</sub> )CH=CH(CH <sub>2</sub> ) <sub>7</sub> COOCH <sub>3</sub> or C <sub>18</sub> H <sub>31</sub> O <sub>2</sub> CH <sub>3</sub>	
9-octadecenoic acid (z)-, methyl ester (Oleic acid methyl ester)	23.753	41.12	98.5	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>7</sub> CH=CH(CH <sub>2</sub> ) <sub>7</sub> COOCH <sub>3</sub> or C <sub>18</sub> H <sub>33</sub> O <sub>2</sub> CH <sub>3</sub>	
Octadecanoic acid, methyl ester (Stearic acid methyl ester)	24.213	3.23	98.6	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>16</sub> COOCH <sub>3</sub> or C <sub>18</sub> H <sub>35</sub> O <sub>2</sub> CH <sub>3</sub>	

**Table 2.** Viscosity of the biodiesel and the ASTM standard.

Fuel	Viscosity (at Value (cSt))
Sunflower biodiesel	4.0
Diesel	1.9 - 6.0 [ASTM standard (cSt)]

**Table 3.** TAN and TBN of the biodiesel produced and their respective ASTM standard.

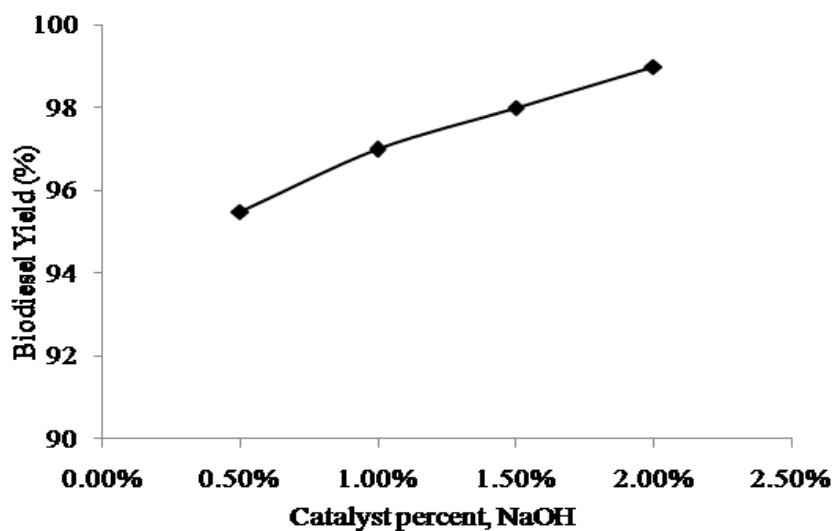
Fuel	Total Acid Number (TAN) Value, mg KOH/g	Total Base Number [ASTM standard, mg KOH/g]
Biodiesel	0.7	11.0
Diesel (TBN)	<0.8	10 - 15

**Table 4.** Fuel consumption and greenhouse gas emission analysis.

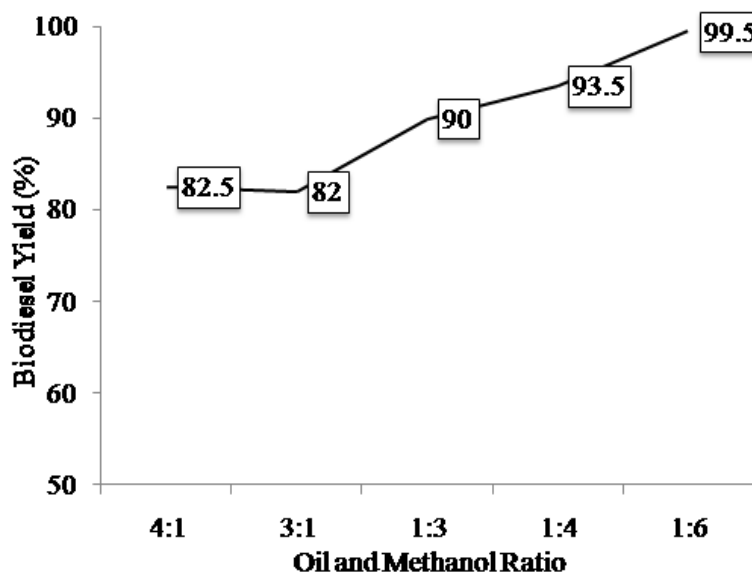
Sample	Load (Nm)	Speed (rpm)	Fuel consumption, ml/sec	HC, ppm	CO, Vol. %	NO <sub>x</sub> , ppm
Biodiesel	15	2000	0.623	21.74	0.026	549.65
Diesel	15	2000	0.591	24.53	0.029	517.41



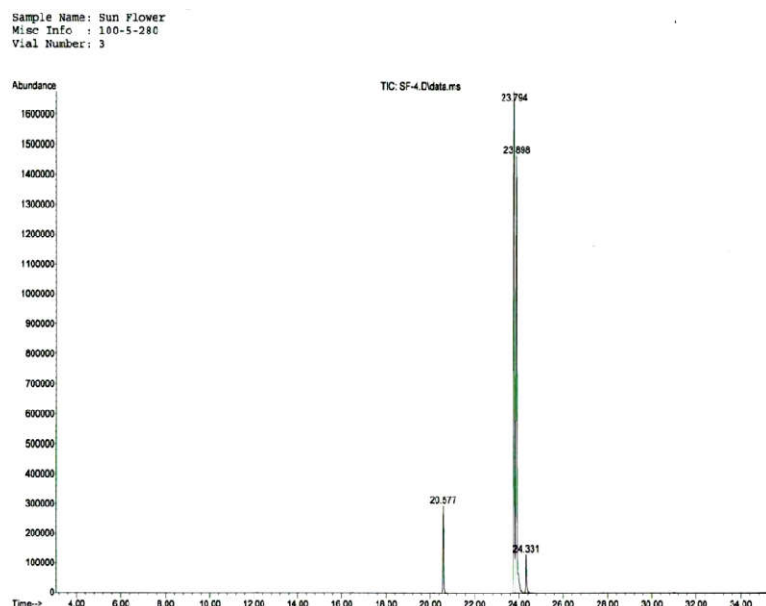
**Fig. 1.** A) Filtered waste sunflower cooking oil, B) Phase separation, upper is biodiesel and C) Formation of emulsion



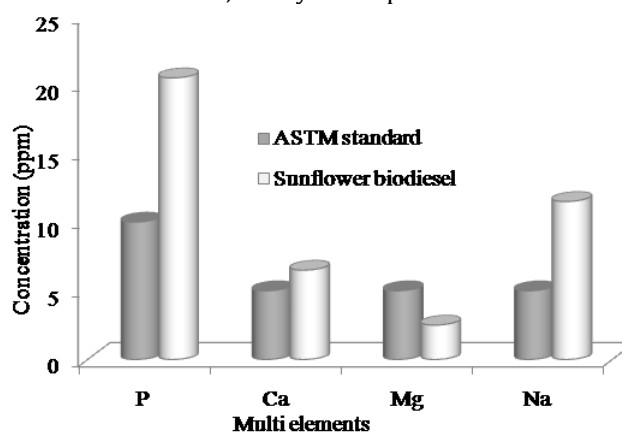
**Fig. 3.** Optimization of alkaline transesterification of waste palm oil.



**Fig. 4.** Optimization of bioethanol yield by different oil and methanol ratio.



**Fig. 5.** Mass spectrum of each peak and identification of corresponding components. Whereas, 1) Hexadecanoic acid, 2) 9,12-octadecadienoic acid (z, z)-, 3) 9-octadecenoic acid (z)-, and 4) Octadecanoic acid, methyl ester peaks.



**Fig. 6.** Chemical elements of the biodiesel produced and their respective ASTM standard.

## CONCLUSION

It can be concluded that biodiesel (99%) was successfully produced from waste sunflower oil using the alkaline transesterification bioprocess reaction. The produced biodiesel showed that lower emission of carbon monoxide and unburned hydrocarbon compared to conventional diesel. The waste cooking sunflower oil can be recommended as a potential feedstock for biodiesel production.

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