Analysis and comparison of olive cooking oil and palm cooking oil properties as biodiesel feedstock

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Abstract. Biodiesel was utilized as an alternative source of renewable energy to counter the shortcomings of fossil fuels. In this study, the physical and chemical properties of olive cooking oils and palm cooking oil were analysed as biodiesel feedstock based on density, specific gravity, acid value, free fatty acid content (FFA), saponification value and peroxide value. The analysis results were compared according to ASTM and EN standards. Olive cooking oil A, olive cooking oil B and palm cooking oil have densities of 895 kg/m³, 897 kg/m³ and 904 kg/m³, respectively. The specific gravities of olive cooking oil A, olive cooking oil B and palm cooking oil were 0.88, 0.88 and 0.89 respectively. Olive cooking oil A, olive cooking oil B and palm cooking oil have acid value of 0.45 mg KOH/g ± 0.02 mg KOH/g with 0.23 % ± 0.01 % FFA, 0.41 mg KOH/g ± 0.02 mg KOH/g with 0.21 % ± 0.01 % FFA and 1.08 mg KOH/g ± 0.07 mg KOH/g with 0.54 % ± 0.04 % FFA, respectively. Palm cooking oil has high acid value exceeded the standard limit; thus, it requires FFA reduction before transesterification. The saponification values of olive cooking oil A, olive cooking oil B and palm cooking oil were 187.93 mg KOH/g ± 1.40 mg KOH/g, 188.40 mg KOH/g ± 0.81 mg KOH/g and 198.22 mg KOH/g ± 0.81 mg KOH/g respectively. The peroxide values obtained for olive cooking oil A, olive cooking oil B and palm cooking oil were 8.00 meq/kg ± 2.00 meq/kg, 8.00 meq/kg ± 2.00 meq/kg and 6.00 meq/kg ± 2.00 meq/kg, respectively. Fourier Transform Infrared Spectroscopy (FTIR) analysis was done and the functional groups present in all oil samples were analysed.

1. Introduction

The term of diesel refers to hydrocarbon mixture derived from petroleum via fractional distillation of crude oil between 200 °C and 350 °C under atmospheric pressure [1]. Due to recent increase in petroleum prices and uncertainties concerning availability of petroleum, the interest in biodiesel as an alternative source for non-renewable diesel has remarkably risen [2].

Biodiesel is defined as mono-alkyl esters of long chain fatty acids derived from renewable feedstock such as vegetable oil or animal fat [3]. According to Lopresto et al. (2015), biodiesel production refers to a biochemical process called transesterification that involves the reaction between triglyceride of vegetable oil or animal fat and alcohol, typically ethanol or methanol, in the presence of a base catalyst such as potassium hydroxide or sodium hydroxide. The chemical reaction of transesterification of triglyceride is shown in Figure 1. According to Ullah et al. (2014) [6], vegetable oils make up the highest rate of the current utilization as potential feedstock for production of biodiesel. In principle, any vegetable oils or seed oils consisting of triglycerides of long chain saturated and unsaturated fatty acids can be used as fuel in diesel
engines [7]. Historically, vegetable oils were used as fuel during World War II where there were serious fuel shortages [8].

![Chemical reaction of transesterification of triglyceride](source.png)

**Figure 1.** Chemical reaction of transesterification of triglyceride. (Source: Guo *et al.*, 2015) [5].

Furthermore, linseed biodiesel was also experimented by Puhan *et al.* (2009) [9] and the results shown a reduced emission of carbon monoxide, hydrocarbon and smoke as compared to diesel; however, increase in nitrogen dioxide emission was reported in comparison to diesel. Positive results were reported and shown that linseed oil was substantially lower wear for biodiesel as compared to diesel and thus it improved the life for biodiesel operated engines. The sustainable development of biodiesel had led an increase in production of various vegetable oils. Thus, the development of biodiesel from palm oil is currently more reliable to countries rich in palm oil plantations such as Malaysia [10].

1.1. Olive Cooking Oil

Olive with the botanical name of *Olea europaea* L. is an oilseed species in the family Oleaceae, found in the Mediterranean region [11]. According to Bernabei (2015) [12], olive gains a major agricultural importance as the source of olive cooking oil in the Mediterranean region.

Recent report by USDA (2016) [13] showed the olive cooking oil production in the EU was facing a significant loss because of the diminished precipitation across the Mediterranean region, as shown in Table 1. The dry weather from September through early October of 2016 impacted the yields in Spain. Besides, prolonged dryness and insect pressure have also affected the yields in Italy and Greece. The property of peroxide value is important to ensure the stability and the shelf life of olive cooking oil. According to IOC (2016), peroxide value relates to storage of olive cooking oil and measures chemical products produced via oxidation that ultimately cause rancidity. Generally, peroxide value higher than 10 meq/kg indicates the less stable olive cooking oil with a shorter shelf life. Thus, low peroxide value is more preferable to ensure the quality of olive cooking oil and its biodiesel.

Abdalla *et al.* (2014) studied the characteristics of different types of olive oils in North of Morocco including acidity, iodine value and peroxide value to test their qualities. Results showed the iodine value of all olive oil samples were not exceeding the limits that gave very strong indication of saturation of molecule. Results also showed the peroxide values of all olive oil samples were not higher than limit. They concluded that olive oil has good properties as they contain low percentage of acidity and peroxide value, as well as
iodine value was within the limits for good quality olive oil. They concluded that olive cooking oil could be utilized successfully as a source for human consumption and various processes or applications such as biodiesel production.

1.2. Palm Cooking Oil
The high world production rate of palm oil in Malaysia and Indonesia created an attention towards sustainable development of palm cooking oil biodiesel to reduce the dependency on petroleum diesel. Over the past decades, several contributions have made by different research groups around the world to development of biodiesel from palm oil for petroleum-based diesel fuel replacement. Alkabbashi et al. (2009) studied the production of biodiesel from crude palm oil by transesterification process, as shown in Figure 1. The study proved that the utilization of crude palm oil as raw material was beneficial for a direct and effective conversion to biodiesel due to its availability in large scale and it minimized the cost of processing. The study also achieved the yield of 93.6% of palm oil biodiesel that meet the specifications by ASTM D6751 and EN 14214.

In this study, the physical and chemical properties of olive cooking oils and palm cooking oil were analysed as biodiesel feedstock based on density, specific gravity, acid value, free fatty acid content (FFA), saponification value and peroxide value.

2. Methodology

2.1. Sample Collection
The olive cooking oils (two samples; manufacturer A and B) and palm cooking oil were purchased from Giant Hypermarket in 1Borneo Hypermall, Sabah. All chemicals were analytical-reagent grade with no further purification.

2.2. Physical Properties Analysis of Oil

2.2.1. Density. The densities of oil samples were determined by using method proposed by Indhumathi et al. (2014) [15] with few modifications. The mass of an empty 50 mL relative density bottle was weighed. Then, each oil sample was poured into the relative density bottle. The mass of relative density bottle containing oil sample was weighed to calculate the mass of oil sample. Densities of oil samples were calculated by using Equation 1.

\[
\text{Density, } \rho = \frac{M}{V} \quad \text{(Equation 1)}
\]

Where,
- \( M \) = Mass of relative density bottle containing oil sample in gram
- \( V \) = Volume of oil sample in millilitre

2.2.2. Specific Gravity. The determination of specific gravities was performed according to method proposed by Omari et al. (2015) [16] with few modifications. The mass of an empty 50 mL relative density bottle was weighed. Then, distilled water was poured into the relative density bottle. The mass of relative density bottle containing distilled water was weighed. Same steps were carried out, by substituting the distilled water with each oil sample. Specific gravities of oil samples were calculated by using Equation 2.
Specific gravity \( \frac{W_2 - W_0}{W_2 - W_0} \) (Equation 2)

Where,
- \( W_0 \) = Weight of empty relative density bottle in gram
- \( W_1 \) = Weight of relative density bottle containing oil sample in gram
- \( W_2 \) = Weight of relative density bottle containing distilled water in gram

2.3. Chemical Properties Analysis of Oil

2.3.1. Acid Value and Free Fatty Acid Content

The acid values and free fatty acid contents of oil samples was determined by using method proposed by Canesin et al. (2014) [17] with few modifications. Each oil sample was weighed to 30.0 g in a 250 mL Erlenmeyer flask. Then, 50 mL of neutral solvent (a mixture of diethyl ether and ethanol) was added into the Erlenmeyer flask containing oil sample. The mixture was stirred vigorously for 30 minutes by using a magnetic stirrer.

Three drops of phenolphthalein indicator were added into the mixture. The mixture was titrated with 0.1 N potassium hydroxide solution to the end point. The end point at which the colour changes was recorded. All experimental procedures were performed in triplicate. Acid values of oil samples were calculated by using Equation 3.

\[
\text{Acid value} = \frac{56.1 \times V \times N}{W} \quad \text{(Equation 3)}
\]

Where,
- \( V \) = Volume of potassium hydroxide solution used in millilitre
- \( N \) = Normality of potassium hydroxide solution
- \( W \) = Weight of oil sample in gram

Acid value was expressed as percentage of free fatty acids calculated as oleic acid. Free fatty acid contents in oil samples were calculated by using Equation 4.

\[
\text{Free fatty acid content} = \frac{\text{Acid Value}}{2} \quad \text{(Equation 4)}
\]

2.3.2. Saponification Value. The method for determination of saponification value proposed by Omari et al. (2015) was performed with few modifications. Each oil sample was weighed to 2.0 g in a 250 mL Erlenmeyer flask. Then, 25 mL of 0.5 N ethanolic potassium hydroxide solution was pipetted into the Erlenmeyer flask containing oil sample. A blank solution was prepared by adding 25 mL of 0.5 N ethanolic potassium hydroxide solution into another 250 mL Erlenmeyer flask. The flasks was fitted to a reflux condenser and refluxed for 30 minutes in a water bath.

After cooling to room temperature, three drops of phenolphthalein indicator were added into the mixture. The mixture was titrated with 0.5 N hydrochloric acid solution to the end point. The end point at which the colour changes was recorded. All experimental procedures were performed in triplicate. Saponification values of oil samples were calculated by using Equation 5.

\[
\text{Saponification value} = \frac{56.1 \times (B-S) \times N}{W} \quad \text{(Equation 5)}
\]

Where,
B = Volume of hydrochloric acid solution in millilitre used for blank
S = Volume of hydrochloric acid solution in millilitre used for samples
N = Normality of hydrochloric acid solution
W = Weight of oil sample in gram

2.3.3 Peroxide Value. The method for determination of peroxide value proposed by Canesin et al. (2014) [17] was performed with few modifications. Each oil sample was weighed to 5.0 g into a 250 mL Erlenmeyer flask. Then, 30 mL of solvent mixture (a mixture of glacial acetic acid and chloroform) was added into the Erlenmeyer flask containing oil sample. The flask was swirled until the sample was completely dissolved. Then, 0.5 mL of potassium iodide solution was added into the mixture by using a micropipette. The mixture was thoroughly shaken and allowed to stand for exactly one minute. Then, 30 mL of distilled water was immediately added into the mixture.

The mixture was covered by using a stopper and shaken vigorously to liberate the iodine from chloroform layer. Then, the mixture was titrated with 0.1 M sodium thiosulfate solution by using starch solution as indicator. The end point at which the colour changes was recorded. A blank determination was conducted at the same time and all experimental procedures were performed in triplicate. Peroxide values of oil samples were calculated by using Equation 6.

\[
\text{Peroxide value} = \frac{1000 \times (S - B) \times N}{W} \quad \text{(Equation 6)}
\]

Where,
- \(S\) = Volume of sodium thiosulfate solution in millilitre used for samples
- \(B\) = Volume of sodium thiosulfate solution in millilitre used for blank
- \(N\) = Normality of sodium thiosulfate solution
- \(W\) = Weight of oil sample in gram

2.4. Functional Group Analysis of Oil
The functional group analysis of oil samples was performed using Perkin Elmer FTIR Spectrum 100 spectrometer.

3. Results and Discussion

3.1. Physical Properties Analysis of Oil
Physical properties of olive cooking oil A, olive cooking oil B and palm cooking oil were studied to determine their potential as viable feedstock for biodiesel production. The physical properties tested were density and specific gravity. Table 1 shows the results of physical properties analysis and standard limits as recommended by ASTM D6751 and EN 14214.

| Physical Property | Olive Cooking Oil A | Olive Cooking Oil B | Palm Cooking Oil | ASTM D6751 | EN 14214 |
|-------------------|---------------------|---------------------|------------------|-------------|----------|
| Density (kg/m³)   | 895                 | 897                 | 904              | -           | 860 - 900 |
| Specific gravity  | 0.88                | 0.88                | 0.89             | 0.87 - 0.90 | -        |

Table 1. Physical properties of olive cooking oil A, olive cooking oil B and palm cooking oil.
3.1.1. Density. The density values for olive cooking oil A and olive cooking oil B obtained were respectively 895 kg/m³ and 897 kg/m³. Palm cooking oil has the highest density value of 904 kg/m³ as compared to the olive cooking oils. It can be observed that the density values of olive cooking oil A, olive cooking oil B and palm cooking oil are within the acceptable EN standard. Density is specified as an important property of biodiesel feedstock to determine the fuel injection performance of its derived biodiesel [18]. Ayoola et al. (2016) [19] reported that biodiesel derived from higher density feedstock was found to have greater density values and greater fuel mass to be injected in a diesel engine. A higher density biodiesel would lead to a low air-fuel ratio, giving a richer mixture and improving the diesel engine performance [20].

3.1.2. Specific Gravity. The specific gravities of olive cooking oil A, olive cooking oil B and palm cooking oil were found to be 0.88, 0.88 and 0.89 respectively, which fall in the range recommended by ASTM standard. Specific gravity, also known as relative density, is described as one of the most basic biodiesel feedstock property because of its correlation with cetane number, heating value, fuel storage and fuel transportation of biodiesel [21]. A high specific gravity vegetable oil can be processed into a high specific gravity biodiesel. Ejikeme et al. (2011) [22] reported that the greater the specific gravity of biodiesel, the higher its energy content. Similar trends had been reported by Alamu et al. (2007) [23] that biodiesel with higher specific gravity had higher energy content, giving a better mileage and increased power.

3.2. Chemical Properties Analysis of Oil

Chemical properties of olive cooking oil A, olive cooking oil B and palm cooking oil were studied to ascertain their acceptability and stability in terms of biodiesel production. The chemical properties tested were acid value, free fatty acid content, saponification value and peroxide value. Table 2 shows the results of chemical properties analysis and standard limits as recommended by ASTM and EN.

| Chemical Property       | Olive Cooking Oil A | Olive Cooking Oil B | Palm Cooking Oil | Standard Limit         |
|-------------------------|---------------------|---------------------|------------------|------------------------|
| Acid value (mg KOH/g)   | 0.45 (± 0.02)       | 0.41 (± 0.02)       | 1.08 (±0.07)     | 0.50 (max.) 0.50 (max.) |
| FFA content (%)         | 0.23 (± 0.01)       | 0.21 (± 0.01)       | 0.54 (± 0.04)    | -                      |
| Saponification value    | 187.93 (± 1.40)     | 188.40 (± 0.81)     | 198.22 (± 0.81)  | -                      |
| Peroxide value (meq/kg) | 8.00 (± 2.00)       | 8.00 (± 2.00)       | 6.00 (± 2.00)    | -                      |

3.2.1. Acid Value and Free Fatty Acid Content. As shown in Table 2, the acid values of olive cooking oil A and olive cooking oil B were 0.45 mg KOH/g ± 0.02 mg KOH/g and 0.41 mg KOH/g ± 0.02 mg KOH/g respectively, which ranged within the limits specified by ASTM and EN standards. However, the acid value of palm cooking oil was 1.08 mg KOH/g ± 0.07 mg KOH/g, which exceeded the maximum limits of 0.50 mg KOH/g as recommended by U.S. and European specifications. Acid value is defined as the amount of potassium hydroxide in milligram that required to neutralize the fatty acid in one gram of oil sample [15]. Also, acid value is a measure of free fatty acid contents in oil. According to Ibeto (2012) [18], high acid value indicated the presence of high percentage of free fatty acids in oil, which was in accordance with the results obtained in this study, as shown in Table 4.2. Olive cooking oil A and olive cooking oil B have free fatty acid contents of 0.23 % ± 0.01 % and 0.21 % ± 0.01 % respectively. Whereas, palm cooking oil having the highest acid value was found to has the highest free fatty acid contents of 0.54 % ± 0.04 %.
3.2.2. Saponification Value. The saponification values of olive cooking oil A, olive cooking oil B and palm cooking oil were 187.93 mg KOH/g ± 1.40 mg KOH/g, 188.40 mg KOH/g ± 0.81 mg KOH/g and 198.22 mg KOH/g ± 0.81 mg KOH/g respectively. It can be observed that the saponification values of three oil samples were ranged between 187 mg KOH/g to 200 mg KOH/g. This range was found typical for the feedstock having predominately fatty acids with chain length between C16 to C18 [24].

3.2.3. Peroxide Value. The peroxide values of olive cooking oil A, olive cooking oil B and palm cooking oil were found to be 8.00 meq/kg ± 2.00 meq/kg, 8.00 meq/kg ± 2.00 meq/kg and 6.00 meq/kg ± 2.00 meq/kg respectively, which are within the limit of 10.00 meq/kg established by Codex Alimentarius International Standard (FAO, 1981).

It can be concluded that olive cooking oil A and olive cooking oil B having higher peroxide value is unstable and would grow rancid easily as well as degrade faster as compared to palm cooking oil having lower peroxide values. Consequently, biodiesel produced from olive cooking oils has a shorter shelf life as it is more susceptible to oxidative degradation.

3.3. Functional Group Analysis of Oil
Fourier Transform Infrared Spectroscopy (FTIR) technique was used to analyse the functional groups present in oils. Figure 2 shows the FTIR spectra of olive cooking oil A, olive cooking oil B and palm cooking oil.

![FTIR spectra of olive cooking oil A, olive cooking oil B and palm cooking oil.](image)

Figure 2. FTIR spectra of olive cooking oil A, olive cooking oil B and palm cooking oil.
The FTIR profile of olive cooking oil A, olive cooking oil B and palm cooking oil are similar with several major absorption bands at around 2922 cm\(^{-1}\) to 2850 cm\(^{-1}\), around 1744 cm\(^{-1}\), around 1464 cm\(^{-1}\), around 1377 cm\(^{-1}\), around 1160 cm\(^{-1}\) and around 720 cm\(^{-1}\). These absorption bands show the characteristic of long chain fatty acids, which predominate in biodiesel feedstock (Sanford \textit{et al.}, 2009).

As seen in Figure 2, the FTIR spectral of olive cooking oil A, olive cooking oil B and palm cooking oil showed absorption bands at around 2922 cm\(^{-1}\) to 2850 cm\(^{-1}\), indicating sp\(^3\) C-H stretch. This shows the presence of aliphatic hydrogen in these three oil samples (Basir, 2013). At around 1744 cm\(^{-1}\), there is an absorption band indicating C=O stretch. This shows the presence of carbonyl group in olive cooking oil A, olive cooking oil B and palm cooking oil.

The weak absorption band appears at around 720 cm\(^{-1}\) shows the methylene rocking vibration in which all the methylene groups rock in phase [22]. This shows the presence of at least four methylene groups in olive cooking oil A, olive cooking oil B and palm cooking oil.

Furlan \textit{et al.} (2010) [25] investigated the oxidative degradation of biodiesel feedstock by using infrared spectroscopy and it was found that the intensity of absorption band in IR spectrum defined the oxidative stability of biodiesel feedstock. A decrease in absorption intensity indicates the susceptibility to oxidative degradation. This means olive cooking oil B is oxidative unstable; however, this problem can be solved if the storage condition of feedstock is controlled.

\textbf{Table 3.} Functional groups present in IR spectra of olive cooking oil A, olive cooking oil B and palm cooking oil.

| Functional group | Wavenumber of Oil Sample (cm\(^{-1}\)) |
|------------------|----------------------------------------|
| C-H stretch      | ~2922.65 to 2853.57                    |
| C=O stretch      | ~1744.25                               |
| CH\(_2\) bend    | ~1463.74                               |
| CH\(_2\) bend    | ~1377.68                               |
| C-O stretch      | ~1159.80                               |
| CH\(_3\) rocking| ~721.07                                |

\textbf{4. Conclusion}

Palm cooking oil having higher density value could produce a higher density biodiesel, which provide a better performance of diesel engine as compared to olive cooking oil feedstock. It also shows that palm cooking oil having higher specific gravity could produce a biodiesel with higher specific gravity, which provide a better mileage as compared to olive cooking oil feedstock. The overall results indicated the suitability of olive cooking oil A, olive cooking oil B and palm cooking oil as biodiesel feedstock.

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