Production and Characterization of Biodiesel from Rapeseed Oil through Optimization of Transesterification Reaction Conditions

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Abstract: In this study, biodiesel fuel was produced from rapeseed oil via transesterification method. The optimum reaction conditions were determined by varying alcohol type and its concentration considering their influence on the yield and properties of produced biodiesel. Methanol and ethanol were alcohol used in the transesterification process. The density of biodiesel was measured at 15 °C according to EN ISO 12185 test method and its viscosity was determined at 40 °C by using a Brookfield digital viscometer (DV-II+ Pro). Shimadzu Auto-Calculating Bomb calorimeter CA-4AJ was used to measure the high heating value. The optimum transesterification conditions found were alcohol:oil ratio of 18:1, 1% of potassium hydroxide as catalyst, 60 min of reaction time, 60 °C of reaction temperature and stirring speed of 650 rpm. Biodiesel properties under these conditions satisfied the regulatory standards and are slightly similar to those of mineral diesel tested in same conditions. Using methanol gives better results compared to ethanol.

Key words: Biodiesel, transesterification reaction, optimization process, rapeseed oil.

Nomenclature

ROME Rapeseed Oil Methyl Ester (biodiesel obtained from rapeseed oil by using methanol)
ROEE Rapeseed Oil Ethyl Ester (biodiesel obtained from rapeseed oil by using ethanol)
HHV Higher Heating Value
EN European Standards
ISO International Organization for Standardization
ATSM American Society for Testing and Materials

1. Introduction

Uncertainty about the availability and price of crude oil has focused attention on alternative transport fuels. Given a sustainability criterion, biofuel is one of the most relevant alternatives from renewable source. It includes several fuels types such as biodiesel and bioethanol. These fuel types appear to be attractive options for the transportation sector due to their similar inherent properties compared to fossil-based fuels, especially the auto-ignitibility [1].

Recently, there have been several investigations which highlighted the potentialities of biodiesel as an alternative fuel [2]. It is the most feasible alternative for diesel fuel in terms of compliance with fuel standard. The outstanding advantage of using biodiesel as engine fuel is its adaptability to a diesel engine without any major modification. Biodiesel offers many other advantages including environmentally friendly emission profile, biodegradability, higher combustion efficiency and easy availability [3, 4]. On the other hand, biodiesel can be produced from oil reach plant such as rapeseed, sunflower, soybean, palm, algae, etc. and hence its development creates a massive new demand in agricultural economy [1].

Technically biodiesel is made by mixing alcohol with vegetable oil, animal fat, algae or recycling cooking oil. These raw materials content triglycerides
molecules and others components depending on their nature. In the process called transesterification, triglycerides react with alcohol in the presence of a catalyst to yield biodiesel (fatty acid alkyl ester) and glycerol as by-product [5-9]. The main objective of this transesterification is to lower the viscosity of the feedstock oil close to that of petro-diesel. Although the procedure does not imply complicated chemical reactions, it is particularly difficult to conduct it properly. This calls for the highest industrial standards to ensure the quality of biodiesel which must adhere with the international standard.

In order to identify the most critical issues for a sustainable production of biodiesel by transesterification, certain criteria should be taken into account. This should include several parameters such as the nature and amount of catalyst, alcohol/vegetable oil molar ratio, the reaction time and temperature and the type of alcohol used [10-13]. However, the nature of alcohol and its concentration are believed to be the most important variable affecting the yield and properties of biodiesel [14, 15]. Several types of alcohol have been explored for biodiesel production; however, methanol and ethanol are the most often used in the transesterification of vegetable oil [16]. Methanol is particularly preferred because its reaction with triglycerides is quick and efficient. Its short-chain molar size contributes for the avoidance of steric hindrance which disturbed the reaction process and lowered the biodiesel yield [14]. As for ethanol, it is more expensive and less reactive compared to methanol. It forms an azeotrope with water which makes difficult it’s recovery from biodiesel product [17, 18]. Despite these disadvantages, ethanol is attracting a lot of interest in the production of biodiesel. It can be obtained from biological sources easily available. Hence biodiesel involving ethanol is completely bio-base and renewable. However, the reactivity of the alcohol depends strongly on the feedstock and the reaction conditions as well.

In the other hand, the concentration of alcohol expressed by the molar ratio of alcohol to triglycerides, is one of the most important variables affecting the transesterification efficiency, the cost and properties of biodiesel [19, 20]. The stoichiometric molar ratio of the transesterification reaction requires three moles of alcohol and one mole of triglyceride to yield three moles of fatty acid alkyl esters (biodiesel) and one mole of glycerol (by-product). Since this process is a reversible reaction, the molar ratio should be higher than that of the stoichiometric ratio in order to increase the miscibility and thus enhance the contact between alcohol molecules and triglycerides [21, 14]. As a result, a molar ratio of 6:1 is normally used in industrial process to obtain a high yield of biodiesel. Moreover, it is assumed that the accepted alcohol to glycerides molar ratios can be fixed between 6:1 and 30:1 [22]. However, it must be optimized because a high molar ratio increases the solubility of glycerol in the ester phase. This effect shifts the equilibrium reaction toward the reactants side thus lowering the biodiesel yield. This also favors difficult separations of the glycerol from the biodiesel [23]. Transesterification reaction is run to a specific time during which the conversion rate increases gradually [24]. However, with the reversibility of the transesterification reaction as explained above, long reaction time can decrease the production yield and lead to more time consuming. The reaction time can be limited from 20 min up to several hours depending sometimes on the type of catalyst and the amount of reactants. For a transesterification reaction catalyzed by a base substance, the yield of biodiesel reaches a maximum at about 120 min while longer reaction time is need when acid catalyzer is used. This is because base catalyzers exhibit high reactivity compared to an acid catalyst [25]. Furthermore, during the transesterification process, energy is supplied to reactants by heating process controlled by the reaction temperature. High temperature increases the reaction rate and thus improves the yield of produced biodiesel. However, the reaction temperature must be less than the boiling point of alcohol to avoid its loss through
vaporization. High temperature beyond the optimum level can be favorable for the soap formation which results in a low biodiesel yield. Reaction temperature is usually chosen between 60 to 80 °C depending on the alcohol and oil type used [26, 27]. Since the boiling point of ethanol is 78 °C, the reaction temperature should be less than 78 °C. In order to obtain efficient conversion rate of biodiesel, the transesterification should be carried out in the presence of catalyst. New trends are oriented toward effective bifunctional heterogeneous catalysts for biodiesel production. Even though these catalysts can simultaneously carry out the esterification of Free Fatty Acids (FFAs) from the raw material and transesterification of triglycerides, they are still under research and not easily available [28]. Generally, in industrial scale homogeneous and heterogeneous acid or base catalysts are used in biodiesel production. However, homogeneous catalysts lead to some drawbacks such as soap formation, reactor corrosion and difficult recovering. As for heterogeneous catalysts, they are non-corrosive, environmentally friendly and can be easily separated from the products [29]. Among the heterogeneous catalysts, sodium hydroxide (NaOH) and potassium hydroxide (KOH) are the most used in the transesterification process [30]. In fact, transesterification of refined and crude oil with 1% either sodium hydroxide or potassium hydroxide catalyst gives successful conversion. However, KOH is most used for biodiesel manufacturing due to its advantage revealed by some researchers such as Rashid and Anwar [31]. They investigated the effects of KOH and NaOH concentrations on the transesterification of rapeseed oil by varying their concentration from 0 to 1.5% (based on the weight of raw oil). They reported that potassium hydroxide (KOH) catalyst with 1% of concentration exhibited the best behavior. Moreover, these results are in line with those obtained by Nye et al. [32] and Tomasevic and Siler-Marinkovic [33], in a transesterification process using frying oils as raw material.

In the view of this above discussion, some transesterification parameters such as catalyst, reaction time and temperature are the easiest factors which can be properly controlled during the industrial process for biodiesel production. However, molar ratio of alcohol to oil and the type of alcohol seem to be the major challenges that associate with the transesterification of vegetable oil. Their optimization is relatively complicated because of their strong influence on the fuel properties of the produced biodiesel. Therefore, it is important to establish optimum conditions for the transesterification of rapeseed oil using suitable process optimization that can be easily employed in large scale production of biodiesel.

The objective of this present research is to determine the best operation conditions for the transesterification of rapeseed oil with respect to their influence on biodiesel fuel properties and its refining process. Our strategy is to optimize the molar ratio of alcohol to rapeseed oil and the nature of alcohol while keeping constant other transesterification parameters such as catalyst type and its concentration as well as the reaction time and temperature. The optimal value for each parameter involved in the optimization process is determined according to the biodiesel yield and its fuel properties. Biodiesel properties are also tested and compared with recommended international standard values and those of mineral diesel. Findings with respect to optimum reaction conditions will also be discussed in the light of those of international standards and other researchers to perform a sustainable biodiesel production from rapeseed oil.

2. Materials and Method

2.1 Raw Material and Chemicals

Rapeseed oil namely Brassica napus (purchased from the Nisshin Oillio Group, Ltd.) is selected as a feedstock for the biodiesel production. It is an agriculture product widely cultivated around the world. It belongs to the preferred oil stock for large scale production of biodiesel [34]. Methanol (CH₃OH;
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99.8%) and ethanol (C\textsubscript{2}H\textsubscript{5}OH; 99.5%) are alcohol types used in transesterification reaction catalyzed by potassium hydroxide (KOH) pellets (85.0%). Acetic acid is acid used during the washing process performed after reaction.

2.2 Transesterification Process

The transesterification procedure used to produce biodiesel is described in the schematic diagram as shown in Fig. 1. For each sample, 100 g of rapeseed oil was poured into a glass flask of 500 mL. The oil was then preheated in thermal batch to eliminate moistures. The required amount of alcohol (methanol or ethanol) and catalysts (potassium hydroxide) established for each sample was measured and mixed in an Erlenmeyer flask. The solution was stirred on magnetic plate to dissolve the KOH pellets. This is to make methoxide/ethoxide solution which is then added into the preheated oil. The reaction takes place and is run for the required time under stirring condition and at ambient pressure. Alcohol concentration was varied by making samples with 6:1; 9:1; 12:1; 15:1; 18:1 and 21:1 of molar ratio. For better estimation of their effects, the following transesterification parameters were kept constant for each experiment: 1% of potassium hydroxide (KOH) as catalyst; 60 °C of reaction temperature; 30 minutes of reaction time and a stirring speed of 650 rpm. After completion of reaction, samples were set to decantation for phase separation by gravity. The biodiesel floats on the top of the reaction vessel while the glycerol sinks in the bottom.

2.3 Purification Process

The biodiesel is then removed and treated with acetic acid to neutralize any unreacted of hydroxide, methoxide or ethoxide. It is also washed with water to remove soluble salts (from potassium hydroxide), unreacted alcohols and partial glycerides. Any water that might have mixed with the biodiesel is removed by heating process. As for the first washing process, the crude biodiesel was mixed with water at 30% by volume and acetic acid at 1% by volume (to neutralize the free fatty acid). After 30 min under stirring process, the mixture as formed was put to settle for two days. In the second and third washing process, the crud biodiesel was mixed with water for half of volume ratio and put

![Schematic diagram of transesterification reaction and purification process.](image-url)
under stirring condition for 30 min. After sedimentation, the crude biodiesel is removed and put under heating process about 10 min at 150 °C which gives the final product ready for use as biodiesel.

2.4 Characterization Process

The produced biodiesel was measured quantitatively and characterized by its density, viscosity and high heating value. The density of biodiesel was measured at 15 °C according to EN ISO 12185 test method and by means of Eq. (1) [35]. Dynamic viscosity of biodiesel was determined by using a Brookfield digital viscometer (DV-II+ Pro) connected to a water bath with circulating pump (Brush less DC Pump, model DC 40-2470). Measurement was done at 40 °C for three different shear rates (7.4 s⁻¹, 15 s⁻¹ and 37 s⁻¹) and according to the standard test method ASTM D445. Results were in triplicate to minimize errors and average was made. Since kinematic viscosity at 40 °C is the parameter required by biodiesel and petroleum diesel standard, it was deducted from the dynamic viscosity and the density by using Eq. (2). The calorific value of biodiesel samples was measured by using the Shimadzu Auto-Calculating Bomb calorimeter CA-4AJ according to ASTMD240-14 standard test method [36]. It was calibrated for 26,456 J/g at 20 °C with standard benzoic acid. Measurement was repeated three times for accuracy purpose and average was done to obtain final value.

\[
\rho_{\text{biodiesel}} = \frac{m_{\text{biodiesel}} - m_{\text{glycerine}} - m_{\text{water}}}{m_{\text{glycerine}}} \times \rho_{\text{water}} \tag{1}
\]

where, \(\rho\) (kg/m³) and \(m\) represent the density and the mass, respectively.

\[
v = \left[ \frac{q}{\rho} \right] \times 10^2 \tag{2}
\]

where, \(v\) = kinematic viscosity in mm²·s⁻¹; \(q\) = dynamic viscosity in mPa·s; \(\rho\) = density of biodiesel at 40 °C.

3. Results and Discussion

3.1 Effect of Molar Ratio and Alcohol Type on Biodiesel Yield and Transesterification

In this study, the biodiesel fuels obtained via transesterification of rapeseed oil with methanol and ethanol are denoted ROME (Rapeseed Oil Methyl Ester) and ROEE (Rapeseed Oil Ethyl Ester), respectively. The effect of alcohol to molar ratio and the nature of alcohol on the biodiesel yield are presented in Fig. 2. The yield of biodiesel obtained during transesterification increased with increase in

![Fig. 2](image-url)  
Effects of molar ratio and alcohol type on the quantity of produced biodiesel fuel under.
concentration of alcohol. The highest yield was obtained at molar ratio of 18:1 for methanol and ethanol as well. Further increase in concentration of alcohol added to the reaction mixture led to decreasing the yield of biodiesel in both cases. It could be explained by the fact that the transesterification reaction was still incomplete for molar ratio less than 18:1. As a result, an increase in alcohol concentration increases the miscibility of the solution which improves the contact between alcohol molecules and triglycerides and then shifts the reaction toward completion [37, 14]. For the molar ratio of 21:1, the yield of biodiesel is reduced because of an increase of glycerol solubility caused by the high concentration of alcohol. In fact, since the solubility of the products is increased, a part of glycerol remains in the biodiesel phase and drives the reaction equilibrium backward to the left and thus lowering the biodiesel amount in both case (ROME and ROEE) [38, 39]. This effect has led to difficult separation of the glycerol from biodiesel particularly in the production of ROEE with molar ratio of 15:1 up to 21:1. To ensure phase separation by gravity, 10 g of water was added to the reaction products followed by a shot stirring process. Even though this operation adds more cost and time to the transesterification reaction, it was effective to recover the biodiesel phase from the by-product. Furthermore, for any molar ratio performed, the amount of ROME is greater than the ROEE one. Thus, according to the transesterification conditions used in this study, methanol is considered to be more reactive with rapeseed oil compared to ethanol. It reacts quickly with triglycerides due to its short-chain molar size. This property contributes strongly to the avoidance of steric hindrance effects and water absorption from atmosphere during transesterification reaction and hence improves the yield of biodiesel [40, 41, 18]. However, using ethanol could achieve an acceptable biodiesel yield despite difficulties noticed during the transesterification procedure. Therefore, the optimum molar ratio to produce high amount of biodiesel is 18:1 and methanol is more favorable for the transesterification of rapeseed oil for biodiesel production.

3.2 Effect of Molar Ratio and Alcohol Type on Fuel Properties of Biodiesel

3.2.1 Effect on Biodiesel Density

Density is an important property of biodiesel since it is used to make mass/volume conversion and calculate the kinematic viscosity properties [42]. It can be used also to provide directional prediction of engine output power and fuel consumption due to the difference mass fuel injected [35, 43, 44]. The densities of biodiesel are generally higher than those diesel fuels. They depend on their fatty acid composition and the purity of the feedstock.

Density of produced biodiesel is presented in Table 1. It can be observed that values decrease slightly with a variation of molar ratio of alcohol to rapeseed oil. This is due to the fact that the density of vegetable oil (rapeseed oil: 0.893 g/mL) and those of alcohol (methanol: 0.792 g/mL; ethanol: 0.789 g/mL) are slightly closed. As a result, the variation of the molar ratio cannot induce significant variation on the density of ROME and ROEE as well [45]. Densities values of biodiesel are slightly higher than those of mineral diesel (0.806 g/mL) measured in the same conditions. From the molar ratio of 9:1 to 18:1, values of ROEE give a slight decreasing trend which may be due to the issues noticed during the transesterification process. However, density of ROME is in good accordance with the standard limits referred [46].

3.2.2 Effect on Biodiesel Viscosity

Viscosity is the most important property to control during fuel processing. It influences the engine starting characteristic and the performance of fuel injection system. The fuel-air mixture impacts strongly the efficiency of the fuel combustion process in a diesel engine. The quality of this mixture depends in turn on the fluidity of the fuel which is directly related to the viscosity [47-50]. Kinematic viscosity at 40 °C is the
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Table 1  Density of ROME and ROEE in (g/mL) (values measured at 15° according to EN ISO 12185 and compared with Japanese biodiesel specification values [46]).

| Molar ratio | (6:1) | (9:1) | (12:1) | (15:1) | (18:1) | (21:1) | Limits     |
|-------------|-------|-------|--------|--------|--------|--------|------------|
| $\sigma_{\text{ROME}}$ | 0.866 | 0.865 | 0.864 | 0.863 | 0.865 | 0.863 | 0.860-0.900 |
| $\sigma_{\text{ROEE}}$ | 0.869 | 0.861 | 0.856 | 0.854 | 0.853 | 0.854 | 0.860-0.900 |

Table 2  Kinematic viscosity (40 °C; mm²/s) of ROME compared with Japanese biodiesel specification values [49].

| Molar ratio | $\nu$ (at 7.5·s⁻¹) | $\nu$ (at 15·s⁻¹) | $\nu$ (at 37.5·s⁻¹) | Limits |
|-------------|---------------------|-------------------|---------------------|--------|
| 6:1         | 6.28                | 5.05              | 4.88                | 3.5-5  |
| 9:1         | 5.94                | 4.89              | 5.10                | 3.5-5  |
| 12:1        | 4.88                | 4.19              | 4.26                | 3.5-5  |
| 15:1        | 4.87                | 3.99              | 4.38                | 3.5-5  |
| 18:1        | 3.84                | 3.49              | 4.26                | 3.5-5  |
| 21:1        | 5.57                | 4.47              | 4.50                | 3.5-5  |

Table 3  Kinematic viscosity (40°C; mm²/s) of ROEE compared with Japanese biodiesel specification [49].

| Molar ratio | $\nu$ (at 7.5·s⁻¹) | $\nu$ (at 15·s⁻¹) | $\nu$ (at 37.5·s⁻¹) | Limits |
|-------------|---------------------|-------------------|---------------------|--------|
| 6:1         | 6.23                | 5.76              | 5.71                | 3.5-5  |
| 9:1         | 5.75                | 4.70              | 5.33                | 3.5-5  |
| 12:1        | 5.29                | 4.75              | 5.10                | 3.5-5  |
| 15:1        | 5.06                | 4.65              | 5.09                | 3.5-5  |
| 18:1        | 4.61                | 4.50              | 5.14                | 3.5-5  |
| 21:1        | 5.33                | 5.03              | 5.52                | 3.5-5  |

Table 4  Kinematic viscosity (40 °C; mm²/s) of diesel and rapeseed oil measured at experimental conditions and compared with European petrodiesel standard (EN 590) values [47].

| Samples       | $\nu$ (at 7.5·s⁻¹) | $\nu$ (at 15·s⁻¹) | $\nu$ (37.5·s⁻¹) | Limits |
|---------------|---------------------|-------------------|-------------------|--------|
| Mineral diesel fuel | 2.50                | 2.50              | 2.97              | 2.0-4.5 |
| Rapeseed oil  | 38.21               | 37.41             | 37.22             | -      |

Parameter required by biodiesel and petroleum diesel standard. Tables 2 and 3 list the kinematic viscosity values of produced biodiesel at different share rate and compared with biodiesel requirements values. For sake of comparison, kinematic viscosity of conventional diesel and rapeseed oil was determined and reported in Table 4. By means of Figs. 3 and 4, it can be observed that values of kinematic viscosity for ROME and ROEE decease when the molar ratio is increased from 6:1 to 18:1 and increased at 21:1 molar ratio. There is also a similar tendency when share rate was increased during the measurement. ROME and share rate of 15·s⁻¹ give the lowest kinematic viscosity values. Particularly the molar ratio of 18:1 with the lowest viscosity value (3.49 mm²/s) can be considered as an optimal molar ratio in the transesterification process of rapeseed oil using methanol. Likewise, viscosities values of ROME decline faster than those of ROEE and they are the closest to diesel one as shown in Fig. 5. So, using methanol in the transesterification is the most effective to decease the viscosity of the rapeseed oil [51]. The large differences between viscosity of rapeseed oil and biodiesel observed in Fig. 6 were due to the conversion of large and branched triglyceride molecules into lighter straight chain of methyl or ethyl ester (biodiesel) molecules. This result shows that the transesterification process greatly improved the viscosity property of rapeseed oil comparable to those of mineral diesel. This difference in viscosity can be used to monitor biodiesel production by transesterification [52].

3.2.3 Effect on High Heating Value

The higher heating value (HHV) is another important property of biodiesel characterized before it...
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Fig. 3  Kinematic viscosity of ROME under different share rates and compared with mineral diesel values.

Fig. 4  Kinematic viscosity of ROEE at different share rates and compared with mineral diesel values.

Fig. 5  Kinematic viscosity of produced biodiesel compared to those of mineral diesel.
is used as fuel in diesel engine. It is defined as a thermal energy released by the combustion of unit quantity of fuel and hence characterizes the energy content of the fuel [53, 54].

The HHV measurements of ROME and ROEE are tabulated in Tables 5 and 6 respectively. Values are triplicated and compared to those of convention diesel measured in same experimental conditions. The HHV of the biodiesel samples is relatively high which ranged from 39.145 to 40.83 MJ/kg. Although, these values fall well within the required standards, they are slightly lower (approximately 10%) than those of diesel. This result is normally acceptable and can be explained by presence of oxygen in biodiesel (11% by weight) and also by the low percentage of carbon and hydrogen in biodiesel [55, 56]. It can be emphasized that data are very spare because of no regular increase or decrease tendency when the molar ratio is increased. However,
the two types of biodiesel (ROME and ROEE) have their maximum heating value at the same molar ratio (12:1) while ROME has the highest values (40.83 MJ/kg).

4. Conclusion

The optimization process employed in this study resulted in a high yield of biodiesel with good fuel properties. The molar ratio of alcohol to rapeseed oil and the nature of the alcohol have significant influence in the yield and properties of the produced biodiesel. Based on the results, the following points are summarized:

- The optimum reaction conditions for the transesterification of rapeseed oil during the optimization process are: an alcohol/oil molar ratio of 18:1, 1% of potassium hydroxide as catalyst, 30 minutes a reaction time, a reaction temperature at 60 °C and a stirring speed of 650 rpm. Biodiesel produced under these conditions gave the best yield in which those obtained from ROME were higher than those of ROEE. Alcohol amount below optimal parameters results in an incomplete conversion of alkyl ester, hence in low biodiesel yield. Beyond optimal conditions higher alcohol amount results in an increase of glycerol solubility furnishing a low yield of biodiesel.

- Biodiesel preparation process greatly improved the properties of rapeseed oil in term of density, viscosity and high heating value. However, using methanol gave the better results than ethanol.

- Fuel properties of produced biodiesel are not only within recommended level but slightly similar to those of mineral diesel hence making the biodiesel more suitable for diesel engine operation.

- Despite its feasibility, transesterification of rapeseed oil involving ethanol led to some difficulties especially in the separation of the glycerol from the ester which required additional procedures for the production process. However, ethanol can be obtained from the biomass, so its use in transesterification process requires a lot of consideration for environmental prospects.

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