Non-edible Oil of *Cerbera manghas* L. Seed From Seram Island-Maluku as Oil Source of Biodiesel Production

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Abstract. Conversion of the *Cerbera manghas* L seed oil into biodiesel using homogenous catalys through esterification and transesterification stage have been done. Esterification process was carried out using mole ratio of methanol/oil 1:3, temperature 60-65°C for 2 hours with acid catalyst 1.25% H₂SO₄. The products were separated between methanol and triglycerides continued by transesterification process using mole ratio of methanol/oil 1:6. Initially, the methanol have been reacted to NaOH catalyst using the catalyst variation 0.1; 0.2; 0.3; and 0.4%, for 2 hours and the reaction temperature 60-65°C. Biodiesel results were analysed by FTIR, GC-MS, and ASTM. The result show that the optimum biodiesel obtained on weight of catalyst is 0.3% NaOH. ASTM the test of results were obtained the properties of the biodiesel there were: specific density of 0.8808 g/cm³, kinematic viscosity of 6.511 mm²/s, flash point 168°C, pour point 9°C, cloud point 12°C and conradson carbon residual 0.165 wt%.

1. Introduction

Biodiesel is a renewable fuel energy source, derived from plant oils and animal fats. This material is converted to methyl ester fatty acids (fatty acids methyl esters) that are environmentally friendly and can help the availability of diesel oil. Biodiesel is generally produced through the transesterification of vegetable oils or animal fats with short-chain alcohols (generally methanol or ethanol) with the help of a catalyst [1–3] or through direct transesterification fatty acids from the hydrolysis of oil with alcohol. The catalyst used in the biodiesel production process generally uses homogeneous catalysts. The advantage of this catalyst is that it does not require a high temperature and pressure in the reaction. Sodium hydroxide and potassium hydroxide are commonly used as homogeneous alkali catalysts and alkali-catalyzed transesterification is most often used commercially [4–6].

Vegetable oil is a raw material for biodiesel production that is renewable, environmentally friendly, can be produced on a large scale, and the manufacturing process is easy and fast. Vegetable oil consists of edible oil (can be consumed) and non-edible oil (not consumed). More than 95% of the raw material for biodiesel production comes from edible oil because it has quite a lot of resources [7,8]. Various edible oils such as sunflower oil [9], cottonseed oil [10], and coconut oil [11] can be used as sources of biodiesel. However, the use of edible oil has a weakness, namely competition with human consumption so that it can increase the cost of biodiesel production.

To overcome the weakness, many researchers are interested in developing non-edible oils [12,13]. This is due to several advantages possessed by non-edible oils, namely not suitable for human consumption because of the presence of several toxic components in oil, plants that produce non-edible oils do not require special soil conditions to grow as well as food crops, and costs cultivation is
much lower because these plants can still be maintained with high yields without intensive care [14,15]. In addition to jatropha, another non-edible oil Cerbera manghas L also can be used as a base for biodiesel derived from non-edible oil. Based on study shows the Cerbera manghas L seeds contain oil of 46-64% [16]. Meanwhile, according to the other study, the Cerbera manghas L seeds contain quite a lot of oil (54.33%) [17], so it have the potential to be used as raw material for biodiesel production. The content of oil and the type of content of the Cerbera manghas L compound depends on the location of the growth of this plant. In Indonesia, Cerbera manghas L can growing in various regions, one of which is in the village of Kamarian, West Seram Regency, Maluku Province.

To date, The research on the application of Cerbera manghas L seed oil from the Seram Islands of Maluku Province as a source of oil for biodiesel production based on the literature never conducted. This research has purpose to determine the compounds contained in Cerbera manghas L oil using FT-IR and GC-MS as well as the biodiesel quality produced using the ASTM test.

2. Experimental

2.1 Materials
The materials have used for this study are Cerbera manghas L fruit, methanol (Merck), H2SO4 (p.a) H3PO4 (p.a), NaOH monohidrat (Merck), destillet water, Na2SO4 anhidrous, Whatman 41.

2.2 Sample preparation
At first, the Cerbera manghas L seeds were cleaned from the skin and shell then dried in the sun to dry. The dried Cerbera manghas L seeds are crushed until smooth then pressed using a press tool. Continued with the degumming process was carried out by adding 20% H3PO4 of 0.5% oil, heated at 80 °C for 15 minutes to form a phosphatide compound which settles at the bottom of the oil. The precipitate formed was separated then the oil washed with warm water at 60 °C until clear.

2.3 Esterification reaction
A total of 20 g of Cerbera manghas L oil was put into a three-neck flask, then it was esterified using methanol (oil/methanol ratio 1:3) with 1 M H2SO4 catalyst 1.25% of the total weight of oil and methanol then the mixture stirred with medium speed on temperature of 60-65 °C for 2 hours. The reaction mixture is cooled until forming two layers. The upper part of methanol and methyl ester, the lower layer of triglycerides. The layers are separated using a separating funnel.

2.4 Synthesis of biodiesel through a transesterification reaction
The triglycerides resulted from esterification process then transesterified using methanol (oil/methanol ratio 1:6) and NaOH catalyst was added with the variations of weight 0.05%; 0.1%; 0.2%; 0.3%; 0.4%. NaOH solids were dissolved in methanol for 10 minutes. The mixture is refluxed at 60-65 °C for 2 hours. The reaction mixture is cooled and formed 2 layers, namely from top to bottom biodiesel (methyl ester), glycerol. Biodiesel and glycerol layers are separated using a separating funnel. Then the methyl ester layer washed with distilled water in a separating funnel to remove the remaining glycerol. The methyl ester is evaporated to remove the remaining methanol. The methyl ester was added Na2SO4 anhydrous to bind the remaining water and it filtered with the filter paper Whatman-41.

2.5 Characterization of biodiesel
The resulting methyl ester (biodiesel) was tested using FTIR and GC-MS. Test the physical character with the ASTM standard method of specific density 60/60°F (ASTM D 1298), 40 °C kinematic viscosity (ASTM D 445), pour point (ASTM D 97), flash point (ASTM D 93), cloud point (ASTM D 97), and the residual carbon conradson (ASTM D 189).
3. Results And Discussion

3.1 Preparation of Cerbera manghas Seeds L
The oil extraction process of Cerbera manghas L seeds is carried out using the principle of pressing. Cerbera manghas L seeds that have been separated from the seed shell as shown (Fig. 1a), then dried in the sun (Fig. 1b) to evaporate the water contained in them. After drying the seeds of Cerbera manghas L are crushed until smooth (Fig. 1c) and then pressed using a press tool (Fig. 1d). The oil produced from this process is yellowish black because it still contains impurities derived from the skin and chemical compounds such as alkaloids, phosphatides, carotenoids, and others that are dark in color.

![Figure 1](image)

Figure 1 Preparation process of Cerbera manghas L seeds
(a) Results of separation from the skin (b) Seeds dry out
(c) Scouring (d) Pressing equipment

The impurity compounds contained in Cerbera manghas L. oil are separated by a degumming process. The purpose of degumming is to separate oil from gum which consists of phosphatides, proteins, carbohydrates, residues, water, and resins [18]. The degumming process is carried out by adding 20% H₃PO₄ to the oil which is then heated to accelerate the formation of phosphatide compounds. After 15 minutes of heating time, two layers are formed where the phosphatide compounds are bound and deposited at the bottom of the oil. The precipitate formed is then separated using a separating funnel, and the oil is washed with warm water at a temperature of 60 °C to clear. The oil was produced in this process has a clear yellow color.

3.2 Esterification of Cerbera manghas L oil
The free fatty acid content contained in bintaro oil is quite high at 5.82%, so clear bintaro oil produced in the degumming process is esterified first. Esterification is the process of converting some of the free fatty acid content into methyl esters. In other words, the amount of free fatty acids contained in oil will be relatively reduced [19,20]. In this process, 20 g of Cerbera manghas Loil was poured into a three neck flask, then it is esterified with methanol and the aid of an acid catalyst. The use of an acid catalyst is intended to avoid saponification in this process and the reaction can take place quickly. After 2 hours of reflux takes place, the oil from the esterification reaction is cooled to form two successive layers from top to bottom methyl esters and triglycerides. The esterification reaction mechanism of the trigliseride using acid catalyst in methanol shows in Fig. 2.
3.3 Transesterification reaction

The transesterification reaction will convert esters from fatty acids in the form of complex triglycerides to simpler ester mixtures and glycerol as by-products. The separated triglycerides in the esterification stage were reacted with methanol and alkaline alkaline catalyst NaOH with variations in weight of 0.05%; 0.1%; 0.2%; 0.3%; 0.4% at 65 °C for 2 hours. The temperature of the reaction used must be close to the boiling point of methanol. The reaction mixture is cooled and two layers are formed, namely from top to bottom methyl ester, glycerol. The layers of methyl ester and glycerol are separated using a separating funnel.

The methyl ester is then washed with distilled water in a separating funnel which was previously heated at 60 °C to dissolve the remaining glycerol. The washing process aims to remove the remaining glycerol and the remains of methanol which is still present in methyl esters. The washing process will
form two layers, the upper part of the methyl ester and the bottom is distilled water and the remaining glycerol. The biodiesel conversion results presented in graphical form can be seen in Fig. 3 and the transesterification reaction mechanism of the triglyceride show in Fig. 4.

Figure 4 Transesterification reaction mechanism of triglyceride
The graph in Fig. 3 shows that with increasing catalyst concentration, biodiesel conversion tends to increase. The highest biodiesel conversion was obtained by using 0.3% NaOH catalyst. At catalyst use of 0.4% or more than 0.3% there is a decrease in biodiesel conversion. The decrease was due to excessive use of catalysts resulting in reduced biodiesel yield. The use of excessive catalysts can lead to saponification reactions. The higher the concentration of the catalyst used, the higher the conversion of transesterification products, but for high concentrations of catalysts it could cause a decrease in products because it could damage the fatty acid content [21].

3.4 Biodiesel analysis with FT-IR spectrometer
Analysis of the results of biodiesel with FTIR spectrophotometer was carried out to prove the presence of ester groups in transesterification products. The presence of esters, which can be seen in the absorption of C = O and C-O groups compared to the results of identification of spectrophotometer FTIR Cerbera manghas L oil and Cerbera manghas L biodiesel can be seen in Table 1.

![Figure 5 FT-IR spectra of biodiesel from Cerbera manghas L oil](image)

Table 1. FT-IR spectra comparison of the oil Cerbera manghas L and biodiesel

| Oil (cm⁻¹)   | Biodiesel (cm⁻¹) | Functional group |
|--------------|------------------|-----------------|
| 1247.00      | 1170.81          | C-O             |
| 1715.71      | 1743.68          | C=O             |
| 722.35       | 722.35           | -CH=CH          |
| 1458.21      | 1436.99-1458.21  | -CH₂⁻           |
| 1363.70      | 1363.70          | -CH₃             |
| 3468.08      | 3468.08          | OH              |

In Table 1 shows that the presence of esters for bintaro oil has a strong absorption bond that arises from the carbonyl group (C = O) at a wavelength number of 1715.71 cm⁻¹ and C-O group at uptake of 1247.00 cm⁻¹. Biodiesel uptake results showed a change in the absorption of the C = O group to 1743.68 cm⁻¹ and C-O group also found in uptake of 1170.81 cm⁻¹. Whereas in the absorption area of 722.35 cm⁻¹ it occurs in alkene groups (CH = CH). Uptake in the wavelength region of 1436.99-1458.21 cm⁻¹ which is absorption for CH₂ and 1363.70 cm⁻¹ is absorption for CH₃. The shift between
1170.81 cm\(^{-1}\) to 1247.00 cm\(^{-1}\) represents the C-O range adjacent to CH\(_3\) which indicates the presence of a methyl ester group. Uptake at 346.08 cm\(^{-1}\) is a typical absorption area for OH from glycerol [22].

3.5 Biodiesel analysis by GC-MS

Methyl esters (biodiesel) produced from the 0.3% transesterification reaction was then analyzed using GC-MS to determine the type of methyl ester contained in bintaro seed oil. The number of peaks in the results of the chromatogram shows the amount of each esterification methyl ester separately and produces peaks with different retention times. Esters with short carbon chains will first come out after that followed by esters with long carbon chains. The basis of separation by gas chromatography is the distribution of samples between phases. One phase is the stationary phase and the other phase is the mobile phase of gas that passes through the stationary phase [23]. The column (stationary phase) used is non-polar, whereas in general esters are polar. Short chain esters are more polar than long carbon chain esters. According to the law like dissolve like, the ester with a longer chain will be stuck in the column, while the ester with a short chain will pass along the mobile phase out of the column. The content of methyl esters from GC-MS analysis on biodiesel biodiesel seed oil can be shown in Fig. 6.

![Figure 6 Chromatogram of biodiesel of Cerbera manghas L oil](image)

In Fig. 6 the chromatogram of the GC-MS test results showed three peaks detected as fatty acid methyl ester. The detected peak is the first peak with a retention time of 17.127 minutes (Molecular weight 270 g/mol) with the persetation 28.11% is methyl palmitate (C\(_{17}\)H\(_{34}\)O\(_2\)), the second peak with a retention time of 21.514 minutes (Molecular weight 296 g/mol) with the persetation 64.03% is methyl oleate (C\(_{18}\)H\(_{36}\)O\(_2\)), the third peak with a retention time of 22.115 minutes (Molecular weight 298 g/mol) with the persentation 7.86% is methyl stearate (C\(_{19}\)H\(_{38}\)O\(_2\)). Based on the molecular weight of the methyl palmitate compound (270 g/mol) and GC-MS test results, the fragmentation pattern can be shown in Figure 7. The fragmentation pattern of methyl oleate (296 g/mol) is shown in Fig. 8 and Fig. 9, Fig. 10, and Fig. 11 shows the methyl stearate fragmentation pattern (298 g/mol).
Figure 7 Fragmentation pattern of methyl palmitate compounds
Figure 8 Fragmentation pattern of methyl oleate compounds
Figure 9 Fragmentation pattern of methyl oleate compounds (continued)
Figure 10 Fragmentation pattern of methyl stearate compounds
3.6 Analysis of the physical properties of biodiesel by the ASTM method

Testing the physical properties of biodiesel from *Cerbera manghas* L seed oil was obtained by the ASTM (American Standard for Testing and Materials) examination method compared to the standard diesel oil and biodiesel SNI. The test results are shown in Table 2.

**Table 2. Characteristics of biodiesel from *Cerbera manghas* L.**

| Properties               | Biodiesel | ASTM D6751-08 | Methods          |
|--------------------------|-----------|---------------|------------------|
| Specific density 60/60 °F (g/ml) | 0.8808    | 0.850-0.890   | ASTM D 1298      |
| Kinematic viscosity 40°C (mm²/s) | 6.511     | 1.9 – 6       | IKU/5.4/TK-02    |
| Flash point (°C)         | 168       | 100 min.      | IKU/5.4/TK-03    |
| Pour point (°C)          | 9         |               | IKU/5.4/TK-04    |
| Cloud point (°C)         | 12        | 18 min.       | IKU/5.4/TK-04    |
| Conradson carbon residual| 0.165     | 0.05 max.     | ASTM D 189       |

**Figure 11** Fragmentation pattern of methyl stearate compounds (continued)
The characteristics of biodiesel were obtained with the physical characteristics of standard diesel oil. Biodiesel characteristics must be compared with standard diesel oil specifications, such as ASTM specifications for biodiesel [24].

**a. Specific density**
Specific density of biodiesel from *Cerbera manghas* L seed oil is 0.8808 g/cm$^3$ has according to The ASTM standard 0.850-0.890 g/cm$^3$. If the biodiesel has a specific density exceeding the provisions, an imperfect reaction will occur in the conversion of vegetable oils. The biodiesel will increase engine wear, emissions, and cause damage to the engine [25].

**b. Kinematic viscosity**
Kinematic viscosity was measured by the IKU/5.4/TK-02 / D445 test methods get the results 6.511 mm$^2$/s. The result is out of the ASTM range 1.9 – 6 g/ml. Viscosity is related to the composition of fatty acids and biodiesel purity, viscosity increases with increasing chain length of saturated fatty acids, increases in the length of the carbon alcohol chain, decreases in the chain length of unsaturated fatty acids and the residual increase in -mono, -di and triglycerides in biodiesel [26].

The viscosity of fuel for diesel engines needs to be limited. The viscosity that is too low can cause a leak in the fuel injection pump, while the viscosity that is too high can affect the fast work of the fuel injection device and make it difficult to fade the fuel oil so that combustion becomes less than perfect. The kinematic viscosity of biodiesel produced from *Cerbera manghas* L seed oil is quite high so that when used as diesel engine fuel, the injection results in the combustion chamber easily form fog and facilitate combustion, and are useful for lubricating diesel engines [25].

**c. Flash point**
The flash point is measured by the IKU/5.4/TK-03/D 93 test methods is 168 °C. The result is still in the ASTM range minimum 130 °C. Determination of this flash point is related to safety in storing and handling fuel. A high flash point will facilitate the handling, storage and transportation process because it can reduce the risk of ignition. If the biodiesel has low flash point causes the fuel to burn easily in storage [27].

**d. Pour point**
The pour point is a number that shows the lowest temperature where fuel can still flow or pour if cooled under certain conditions. Pour point was measured by the IKU / 5.4 / TK-04 / D97 test method. The pour point test results have met biodiesel quality standards. In the pour point test associated with viscosity, that is, with the lower viscosity of biodiesel, it is easier for biodiesel to flow under certain conditions. The high content of methyl esters derived from saturated fatty acids will increase biodiesel viscosity, whereas the presence of methyl esters derived from unsaturated fatty acids will reduce the value of viscosity [28].

**e. Cloud point**
The cloud point results show that the biodiesel yield from *Cerbera manghas* L seed oil was obtained 12 °C. Based on the pour point, it can be concluded, with the lower pour point of the methyl ester, the easier the biodiesel to flow so that the combustion process is faster [28]. The faster the fuel flows, the faster the fuel will turn into fog with a higher temperature, then the fog point (cloud point) has a higher temperature than the pour point.

**f. Conradson's remaining carbon**
Conradson carbon residue is the value of carbon left after evaporation and combustion is exhausted. Conradson's remaining carbon was measured by the ASTM D 189 test methods, the test results obtained were 0.165% wt. Conradson's remaining carbon test results still in SNI biodiesel
specifications range. Low the conradson carbon residual value indicates that the decomposition of triglycerides is getting closer to perfect so that it can reduce air pollution [29].

4. CONCLUSION

Based on the results can be concluded that the biodiesel was carried out through esterification and transesterification reactions using 5 variations of NaOH catalyst concentration, the optimum weight percent of NaOH as a catalyst is 0.3% with a percent conversion of 73.81%. While the quality of the biodiesel produced from Cerbera manghas L seed oil using NaOH catalyst at optimum weight percent through FT-IR test there are functional groups C-O, C = O, -CH=CH-, -CH2, -CH3, and OH. The GC-MS results showed three peaks as fatty acid methyl esters, namely, the first peak with a retention time of 17.127 minutes which was methyl palmitate, the second peak with a retention time of 21.514 minutes which was methyl oleate, the third peak with a retention time of 22.115 minutes which was methyl stearate, and with the ASTM method, the results for specific density (0.8808 g/cm³), kinematic viscosity (6.511 mm²/s), flash point (168 °C), pour point (9 °C), cloud point (12 °C), and conradson carbon residual (0.165% wt).

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