Optimizing production of biodiesel from Nyamplung oil with microwave power setting

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Abstract. This experimental study examined the processing of biodiesel from Nyamplung oil by microwave power regulation. The aim is to optimize biodiesel yields based on the time process and heating temperature of 650 °C. A suitable method for analyzing biodiesel results is to set the 400 Watts microwave power to 10%, 30%, and 50% of the maximum power. In this process a 1% w/w KOH catalyst is used with a variety of molar ratios of oil-methanol such as 1:6, 1:9, and 1:12. The results showed that the 1:6 molar ratio is the highest percentage of biodiesel obtained with a variation of power of 10% or equal to 40W. The fastest time in this process is 10 minutes. In contrast, the 1:12 molar ratio is the lowest obtained percentage of biodiversity. The higher the temperature and the molar ratio used the fewer biodiesel products are obtained.

Keywords: biofuel, transesterification, catalyst, temperature

1. Introduction
Nyamplung or Calophyllum inophyllum L. is one of the plants that have the potential to be a better source of biofuel compared to jatropha. Methyl ester fatty acids from Nyamplung seed oil meet all requirements for major biodiesel in the United States standards (ASTM D 6751-06) and European Union standards (EN 14214) [1]. Dried seeds of Calophyllum inophyllum have free fatty acids (FFA) of 17.77% [2]. The advantage of Nyamplung as a biofuel is that the seeds have a higher yield of 40-70%, compared to other types of plants, such as jatropha is 40-60%, and oil palm is 46-54%. In addition, users do not compete with food interests [3]. As with other biofuel plants, Nyamplung seeds also require heating treatment in their processing starting with degumming, esterification, and transesterification.

The first time a conventional biodiesel treatment process used conventional high-power heaters or heating elements and mechanical stirring. In this method, electric heaters are used for heating and electric motors for the mixing of reactants. Conventional heating depends on the convection current and thermal conductivity of the reaction mixture. In general, the reaction temperature is used below the temperature of the boiling point of methanol. Biodiesel yield from 80 to 100% can be obtained depending on the raw material, the amount and type of alcohol, the catalyst quantity, reaction temperature, and reaction time. Conventional biodiesel processing with a batch system is very slow where the reaction time exceeds one hour and the separation stage is more than 5 hours [4-6], and they use more energy [7].
Along with the development of advanced technology, the use of microwave radiation has been widely used by previous researchers as a substitute for conventional methods [8-10]. Microwave heating has the advantage of being more evenly heated because it does not transfer heat from the outside but generates heat from inside the material. The heating can also be selective, meaning that it depends on the dielectric properties of the material. This will save energy for heating. In addition, these waves can propagate through liquids where the heating process takes place more effectively and the biodiesel processing stage becomes shorter [11].

Based on some literature that has been studied that the irradiation of the microwave can be used as a heat source in the transesterification process only a few minutes. The characteristics of microwave dielectric depend on the temperature, frequency, and catalyst [12]. Many works, e.g. [4,5], explain that there are several variables that affect the quality of biodiesel, such as reaction temperature, reaction time, catalyst, molar ratio, reactant purity, and free fatty acid content. However, although, the temperature of the microwave heating takes place very quickly, it requires setting the time to maintain a constant temperature. The longer the time, the higher the temperature in the microwave. Here, it is not needed in the transesterification process. The purpose of this paper is to optimize the production of biodiesel from Nyamplung oil by controlling microwave power. For comparison, microwave power is regulated by variations of 10%, 30%, and 50%.

2. Materials and methods

![Nyamplung oil (Crude oil)](image)

**Figure 1.** Nyamplung oil (Crude oil)

| Characteristics         | Before Degumming (Crude oil) | After degumming (Refined oil) |
|-------------------------|-----------------------------|-------------------------------|
| Free fatty acid content | 19.82 %                     | 15.21 %                       |
| Color                   | Dark green and thick         | Reddish yellow and thick      |

**Table 2.** Specifications of microwave oven

| No. | Name               | Specification          |
|-----|--------------------|------------------------|
| 1.  | Model              | R-222Y(w)              |
| 2.  | Voltage input      | 220 Volts              |
| 3.  | Power              | 400 Watts              |
| 4.  | Current output     | 3.6 Amps               |
| 5.  | Frequency          | 2450 Hz                |
Figure 1 shows the Nyamplung oil obtained from Kebumen, Central Java [13]. The characteristics of Nyamplung oil before and after degumming are shown in Table 1. In this study, a Sharp brand Microwave oven is used in the processing of biodiesel. Table 2 shows the equipment specifications with heat control ranging from 10%, 30%, 50%, 70%, to 100%, respectively.

3. Results and discussion

3.1 Testing the characteristics of microwave temperature

In this test, the measurement of the microwave temperature is carried out by setting the power from 10%, 30%, 50%, 70%, to 100%. This value shows the use of microwave energy from 40 W, 120 W, 200 W to a maximum power of 100 W. Figure 2 shows the measurement of the microwave temperature and it appears that the setting of 10% reaches a temperature of up to 40°C with a time of 50 min, while the shortest time reaches 6 min at 95°C with maximum power (100%).

![Figure 2. Temperature characteristics of microwave oven](image)

3.2 Transesterification process

The transesterification process is the main stage of this study. Table 3 shows the main components of oil that are converted to methyl esters and glycerol. In this process, a 1% w/w KOH catalyst is used with three variations of the oil-methanol molar ratio of 1: 6, 1: 9, and 1:12 respectively. There are only 3 variations of microwave power used starting at 10% (10 min), 30% (4 min), and 50% (2 min) of maximum power. The time and power settings are carried out to get the average reaction temperature reaching 65°C. According to [14], that the optimal temperature in the transesterification process with a KOH catalyst is 65°C.

Based on Table 3, it can be seen that the largest percentage of biodiesel is obtained at a molar ratio of 1:6. In the 10% power setting with a heating time of 10 min, the biodiesel yield was 83.45%. The Arrhenius equation states that temperature is a factor that affects the constant value of the reaction speed (k). That is, the temperature increases, the reaction speed will increase as well. However, these results differ from the Arrhenius equation because at temperatures above the optimum temperature a little biodiesel is obtained. The decrease in the percentage of biodiesel above the optimum temperature is due to the possibility of evaporation of methanol due...
to heating exceeds the boiling point temperature. This effect causes contact between reactants is not optimal and imperfect [15].

Table 3. Transesterification from Nyamplung oil

| No. | Molar ratio | Sample | Microwave power (%) | Final Temperature (°C) | Methyl Ester (%) | Glycerol Crude (%) |
|-----|-------------|--------|---------------------|-----------------------|-----------------|-------------------|
| 1.  | 1:6         | X1     | 10                  | 63                    | 83.45           | 16.55             |
|     |             | X2     | 30                  | 69                    | 81.56           | 18.44             |
|     |             | X3     | 50                  | 71                    | 81.25           | 18.75             |
| 2.  | 1:9         | Y1     | 10                  | 63                    | 77.15           | 22.85             |
|     |             | Y2     | 30                  | 65                    | 78.71           | 21.29             |
|     |             | Y3     | 50                  | 67                    | 74.97           | 25.04             |
| 3.  | 1:12        | Z1     | 10                  | 62                    | 76.24           | 23.76             |
|     |             | Z2     | 30                  | 67                    | 72.84           | 27.16             |
|     |             | Z3     | 50                  | 70                    | 72.51           | 27.49             |

3.3 Effects of microwave power and molar ratio

The molar ratio is the mole ratio between oil and methanol in the transesterification process. The molar ratio is one of the important factors that can affect biodiesel production. Stoichiometrically, the reaction of 1 mole of triglycerides requires 3 moles of alcohol. According to [16], the use of excessive amounts of alcohol can increase the production of alkyl esters, however, the separation of biodiesel and glycerol is difficult. Conversely, a little alcohol will produce a little alkyl ester. Therefore a mole comparison between alcohol and triglycerides requires a careful calculation so that the reaction results can be maximized. In addition, the reaction temperature produced by microwave heating depends on power characteristics and operating time. That is, apart from the molar ratio of the reaction temperature produced in the transesterification process depends on the Microwave power regulation which greatly influences the biodiesel yield. In [17], there are several factors that can reduce biodiesel yields such as base catalysts and high temperatures.

3.3.1 Nyamplung oil methyl ester

The final product is Nyamplung oil methyl ester after the crude glycerol is separated. Figure 3 shows the percentage of Nyamplung oil methyl ester yield for various power settings and molar ratios. Based on these graphs, it can be seen that the highest percentage obtained at 1:6 molar ratio of 83.45%, while the lowest percentage at the 1:12 molar ratio of 72.513%.
Based on Figure 3, it can be seen that the decrease in the percentage of Nyamplung oil methyl ester yield occurs with increasing molar ratio in each Microwave power setting. Conversely, the greater the microwave power and the molar ratio the smaller the percentage of methyl ester produced. Figure 4 shows the Nyamplung biodiesel production for 3 variations of microwave power with a molar ratio of 1:6.

Figure 4. Nyamplung oil methyl ester with microwave power regulation for 1:6 molar ratio

3.3.2 Crude glycerol
Glycerol or glycerin is a by-product of the transesterification process. Figure 5 shows the graph of crude glycerol after being separated from methyl ester. It is seen that the highest crude glycerol obtained by a 1:12 molar ratio of 27.48% based on 3 variations of microwave power. On the contrary, the lowest crude glycerol was obtained at a molar ratio of 1:6 of 16.55%. That is, the smaller the percentage of methyl ester caused by a lot of glycerol, and vice versa, the greater the
percentage of methyl ester showing less glycerol. It can be stated, a little crude glycerol is inversely proportional to the methyl ester produced.

![Figure 5. Effect of microwave power and molar ratio VS crude glycerol](image)

In contrast to methyl ester, based on Figure 5 it appears that an increase in the percentage of crude glycerol is proportional to the setting of a large microwave power and a high molar ratio. The greater the molar ratio, the greater the crude glycerol produced. Likewise, microwave power regulation, except for 10% power decreased from 22.85% to 21.29%, all of them almost experienced an increase in the percentage of crude glycerol.

3.3.3 Discussion

Optimal conditions are obtained from the processing of Nyamplung biodiesel oil using microwave-assisted techniques with power regulation. To verify the benefits of microwave irradiation, this technique is applied to oils of 1:6, 1:9, and 1:12 molar ratios. The microwave oven is equipped with power regulators ranging from 10%, 30%, 50%, 75%, and 100% which allow temperature regulation and microwave power. Microwave output power varies up to 400 watts, controlled via the regulator button. The temperature is set to 65°C, so the reaction time is different from each power setting.

The base-catalysed transesterification results of Nyamplung oil was investigated by changing the catalyst (KOH) to oil ratios (% w/w) and alcohol to oil ratio (% w/w). The highest version was obtained with a catalyst ratio of 1% KOH to oil is the molar ratio of methanol to the oil of 1:6 for 50 min, and under these conditions, the Nyamplung oil methyl ester yield was 83.45%. Figure 3 shows methyl ester percentage with variations in microwave power at different alcohol/oil molar ratios. The higher the microwave power the faster the transesterification process, however, it produces less methyl ester and vice versa it produces large crude glycerol. Figure 5 shows the results of processing biodiesel by setting the microwave power. It can be seen that the 50% power setting is the largest producer of crude oil, while the 10% power setting produces less crude oil.

One of the most important variables affecting the yield is the molar ratio of alcohol to triglycerides. In this study, a 1:6 molar ratios have given a yield of over 80%. Biodiesel processing in industry, 1:6 molar ratios (oil/alcohol) is usually used to obtain a more optimal yield of methyl ester [5,18]. In many studies, biodiesel with the best properties is obtained by using potassium hydroxide as a catalyst [19-22]. Analysis with a 1% by weight KOH catalyst resulted in a conversion
that succeeded in providing the best results and ester viscosity in most of the literature reviewed. It also showed better results in this study, but the results were obtained using microwave-assisted techniques with microwave power and time regulation.

Applying the microwave technique as explained earlier in the methodology section shows that the application of microwave energy increases the reaction rate for the conversion of crude oil to biodiesel, and encourages the reaction balance towards biodiesel production. The highest biodiesel yield (83.45%) was obtained by applying microwave irradiation for ten minutes at 10% power settings compared to 30% and 50% power. As shown, however, this result is achieved with a 1:6 molar ratios compared to other molar ratios. It is also proven that large power usage and exceeding the optimal reaction time will cause a decrease in biodiesel yield. Like, setting 30% power is obtained by methyl esters of 77.15, while at a setting of 50% power with methyl esters of 76.24%. Apart from the reaction, alcohol, and oil molar ratio, catalyst, reaction time, water-content, FFA, and stirring intensity, one of the important variables that affect biodiesel yield from the transesterification process is the reaction temperature [23-25]. The reaction rate is largely determined by the reaction temperature, therefore, according to [18], this reaction usually has to be carried out close to the boiling point of alcohol at atmospheric pressure.

4. Conclusions

- Microwave power regulation affects the time and temperature of the transesterification process which affects Nyamplung oil methyl ester.
- The greater the molar ratio, the higher the percentage of crude glycerol produced and inversely proportional to the yield of the methyl ester. The 1:6 molar ratio is the best composition compared to 1:9, and 1:12 in the processing of biodiesel Nyamplung oil.
- The higher the microwave power the faster the time of the transesterification process, however, it will produce less methyl ester and produce high crude glycerol.

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6. References
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