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RESEARCH NOTE

Ultrasound Assisted in Situ Esterification of Rubber Seeds Oil for Biodiesel Production

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ABSTRACT

Since the conventional esterification method requires long processing time and gives low yield, the intensification of this process is still an interesting subject to be investigated. To reduce the oil extraction cost from seeds which accounts for almost 70% of total processing cost, in situ esterification has been recently introduced. The objective of this study was to produce biodiesel from rubber seeds oil through in situ esterification, assisted by ultrasound irradiation at 42 kHz for 30 min. The experiment was carried out in two stages of catalyst additions: H_2SO_4 (varied from 0.1-1% v/v) and NaOH (0.1%) and the esterification was conducted at 60 ^oC for 30 min under ultrasound frequency of 42 kHz. The results showed that the optimum yield of fatty acid methyl ester (FAME) was 35% which correspond to yield of biodiesel of 92.5% under conditions of rubber seed ratio to methanol 1:1.75 (w/v), catalyst H_2SO_4 loading of 1% and catalyst KOH loading of 0.1%.

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1. INTRODUCTION

Biodiesel is conventionally synthesized via esterification process, where the extracted oil reacts with alcohol in the presence of acid or alkali catalysts. Non-edible plant oils have been realized as promising crude oils for the production of biodiesel. The use of non-edible oils in developing countries is now more interesting since it does not compete with edible oils and the price is also not that expensive [1, 2]. Rubber seeds are one of the non-edible oil sources and their availability are very abundant in Indonesia, with approximately 40-50% oil contents [3, 4]. The oil must be extracted and refined prior to its use. The cost of extraction itself can account for 75% of overall production cost, which subsequently influences the price of oil. This cost will also affect the biodiesel price in the market. Therefore, there is a need to alternate technology to reduce the price by accelerating the process by implementing extraction and reaction at the same time. The in situ esterification is an approach to produce biodiesel from oil-bearing materials instead of its direct synthesis from the oil [5]. The process is influenced by many variables such as raw material particle size, the molar ratio of alcohol to oil, catalyst concentration, reaction temperature, reaction time and mixing speed intensity. In the in situ esterification method, the alcohol acts as an extracting solvent and a (trans)esterification reagent, so both oil extraction and transesterification reactions are carried in one single step [5]. This method eliminates the requirement of the oil extraction and refinement, so the biodiesel production process could lower the costs and maximize the product yield [6]. The application of this method has been reported in several works including esterification without catalyst, in the presence of catalyst and enzymatic catalyst [7-11]. However, although the use of in situ esterification is able to reduce the extraction step, however the esterification still requires long reaction time (~ 2 h), therefore another technology is required to reduce this reaction time. One of the alternatives is the implementation of ultrasound irradiation.

The use of ultrasound in the biodiesel production has shortened reaction time and enhanced the total FAME

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yields under milder reaction conditions, compared to the conventional method [10, 12, 13]. The study also showed that the base-catalyzed in situ esterification requires 20-40 min of processing time in the presence of ultrasound [14]. However, most of the applications of in situ production of biodiesel has been for low free fatty acid (FFA) oil, which only require base catalyst for biodiesel production. To the best of knowledge of authors, the implementation of in situ process for production of biodiesel using high FFA content oil is still limited. The high content of FFA will determine the amount of acid and base catalyst in the transesterification and esterification reactions. Therefore, the objective of this research was to determine the optimum catalyst concentration and ratio of raw materials to methanol in the production of biodiesel from rubber seed by in situ (tras-)esterification method assisted by ultrasonic irradiation.

2. MATERIALS AND METHOD

2. 1. Materials Rubber seeds were obtained from rubber plantation Kendal Indonesia. Methanol (95%), H_2SO_4 (98%) and KOH were obtined from Merck, Germany. The reactor used consisted of a three-necked flask equipped with stirrer and a thermos-batch ultrasonic unit was used for ultrasound experiment. The experimental set up is shown in Figure 1. The rubber seed oil was analyzed for its content and the results are presented in Table 1.

2. 2. Biodiesel Production Rubber seeds were peeled and the kernels were crushed, macerated and dried in an oven at 55 ^oC for 2 hours. Kernels (100 g) were inserted into a flask assembled by a mixer/stirrer (see Figure 1) and assembled in an ultrasonic bath. Methanol which was mixed with a catalyst was added and the mixture was stirred and heated at 60 ^oC for 30 min at atmospheric pressure. Due to the high content of FFA, the in situ esterification was carried out in two consecutive stages.

TABLE 1. Rubber seed oil analysis

Parameter	Value
Free fatty acid (%)	10.39-16.48
Density (g/mL)	0.90-0.93
Vicosity (cp)	0.404-0.774
Palmitat acid (%)	0.75
Linoleat acid(%)	14.34
Colour	Dark brown
Refractive index	1.42
Iodine value	132.3
Saphonification value	193.5
Mositure content (%)	7.9

At the first stage, the concentration of H_2SO_4 and the ratio of rubber seed to methanol was varied in the range of 0.1-1% (v/v) and 1:2 (w/v), respectively. After 10 min, at the second stage, base catalyst was added to the solution. In this stage, the ratio of rubber seed to methanol was varied in the range of 1:1.5-1:3 (w/v) and catalyst KOH was 0.1% (w/v). After the extraction-reaction in ultrasonic bath (30 min at 42 kHz) was completed, the product was decanted for 3 h to let glycerol and biodiesel separate due to gravitation. The biodiesel was filtered and methanol was separated using distillation. The biodiesel was kept for further analysis. Biodiesel product was then measured for its physical properties according to ASTM 445 for kinematic viscosity and ASTM 127 for density, while acid value was determined based on ASTM D974 method.

2. 3. Analysis Method Biodiesel product was analyzed for its physical properties and FAME concentration. Concentration of methyl ester in biodiesel product was analyzed using Gas Chromatography (HP 5890, with OV-17 column and FID detector). The operation used nitrogen as gas carries with flow rate of 28 ml/min. The final temperature was set at 275 ^oC. The initial temperature was set at 125 ^oC that kept constant for 3 min, furthermore the temperature was increased with a rate of 15/min until 275 ^oC. To compare with commercial product, the biodiesel produced by Pertamina was used as a standard and benzyl alcohol was used as internal standard. The yield of FAME was calculated using the following equation [15]:

$$FAME\ yield(\%) = \frac{weight_{biodiesel} \cdot FAME_{content}}{FAME_{oil}} x100\% \quad (1)$$

$$Biodiesel\ yield(\%) = \frac{denisty \cdot Vol\ biodiesel}{weight_{oil}} x100\% \quad (2)$$

3. RESULTS AND DISCUSSION

Prior to experiment, the rubber seed kernel was analyzed for its fat/oil content. The oil content was 28.65% (wb) or 40% (db) which was in the range of 40-50% and in agreement with Ketaren [16].

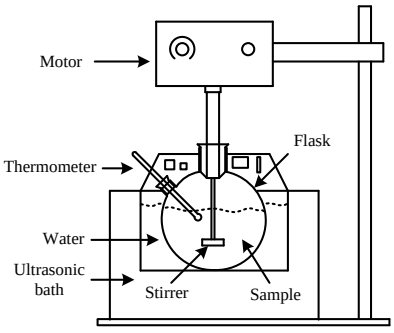

Figure 1. Experimental set up

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In the process of biodiesel production, it is necessary to remove water from seeds, because the water can disrupt the process of biodiesel production. The dehydration at 55 ^{o}C for 2 h, reduced the moisture content from 7.78 to 4.04% which was adopted in our experiment. The drying process does not change significantly the rubber seeds composition [17].

According to Leung and Guo [18] and Eevera et al., [19] when reaction temperature increases beyond the optimal temperature, the yield of the biodiesel product decreases due to acceleration of the saponification reaction of triglycerides. Depending on the oil used, the optimal temperature ranges from 50 to 60 ^{o}C. The reaction temperature must be less than the boiling point of alcohol in order to ensure minimum vaporization. When the reaction temperature closes or exceeds the boiling point of methanol (68 °C), the methanol will vaporize and form a large number of bubbles which may inhibit the reaction [20]. To increase the extraction-reaction rate, vigorous mixing was utilized to increase the rate of collision between the reactants and to homogenize the reaction mixture. The alcohol (methanol) and the triglyceride source (vegetable oil/animal fat) are immiscible and tend to form two layers. Vigorous mixing increases the mass transfer rate by dispersing the alcohol as fine droplets in the triglyceride phase, thereby increasing the contact surface area between the two immiscible reactants [21]. The ultrasound was employed to increase the mass transfer and promote the reaction [22-26].

3. 1. Effect of Catalyst Concentration Catalyst is considered as the important parameter in the production of biodiesel. Therefore, in this experiment the catalyst loading was also investigated. The acid catalyst was varied between 0.1-1% while base catalyst was set constant at 1%. Catalyst KOH was not set as variable because it will produce yellowish deposits (soap) in the lower layer of biodiesel product which will reduce the yield [9].

Figure 2 shows the physical properties of biodiesel produced as function of catalyst H_2SO_4 loading in the solution. There was no significance changes of density and viscosity by variation of H_2SO_4 catalyst. The density of biodiesel varies from 0.846 to 0.891 g/mL which was in the order of the biodiesel standard (SNI 04-7182-2006) which is in the range (0.85-0.89 g/mL). The lowest density was shown at catalyst loading of 0.1% while the highest was obtained at 0.25% of H_2SO_4. From catalyst loading of 0.5 to 1%, the density of biodiesel had a constant trend. The density of fuel has an effect to the break up of fuel injection [27, 28]. Increasing fuel density will increase fuel droplet diameter and lead to an increase of penetrations in combustion chamber [29].

Figure 2 also depicts that variation of H_2SO_4 catalyst from 0.1 to 1% increases the kinematic viscosity from 1.81 to 2.65 mm^2/s which was in the order of standard kinematic viscosity of SNI (2.3–6.0 mm^2/s). This viscosity will interfere with the injection process and fuel atomization. High fuel viscosity can cause early injection due to high line pressure, which moves the combustion of the fuel closer to top dead center, increasing the maximum pressure and temperature in the combustion chamber [28, 29].

Figure 3 shows the positive trend of FAME yield by increasing catalyst concentration with highest yield of 34.74%, at 1% of H_2SO_4 catalyst. The low FAME yield was due to the low quality of rubber seeds and perhaps the acid catalyst concentration was not in the optimum range for producing high yield [30]. However, the amount of acid catalyst should be limited since it will have corrosion effects [31]. Overall, Figure 3 also shows that the application of ultrasound could improve the yield of biodiesel.

3. 2. Effect of Rubber Seed to Methanol Ratio The ratio of alcohol to seeds is one of the important factors that affects the conversion efficiency as well as production cost of biodiesel. Figure 4 depicts that increasing seed:methanol ratio will increase the density and viscosity of biodiesel.

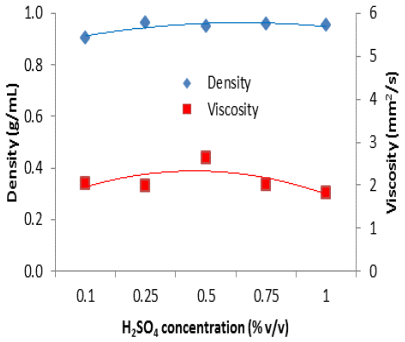

Figure 2. Effect of catalyst concentration (v/v%) of H_2SO_4 on density and kinematic viscosity of biodiesel

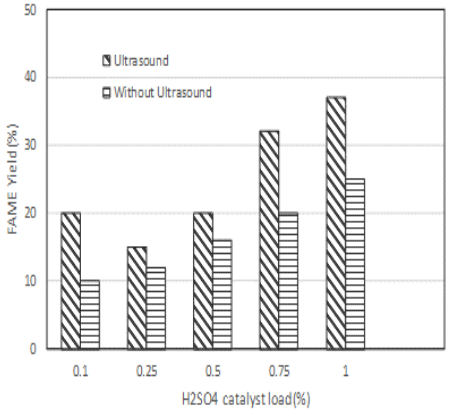

Figure 3. Effect of catalyst concentration on yield of FAME for experiment without and with ultrasound

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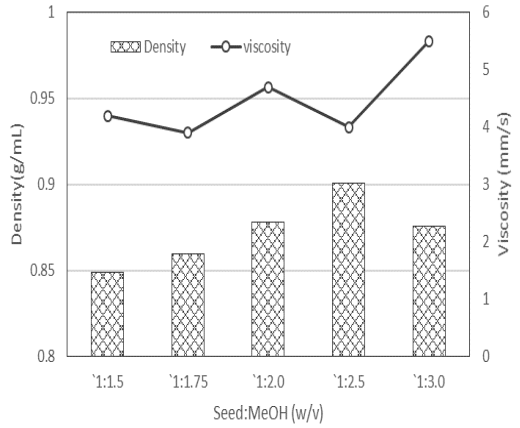

Figure 4. Effect of raw material ratio to methanol on density and viscosity of biodiesel

For the density, the positive trend was identified when the seed:methanol ratio increased from 1:1.5 to 1:2.5 and then declined afterwards. After ratio of 1:2.5, the solution become viscous because of the too much alcohol in the solution, therefore the density decreased. Moreover, at higher ethanol concentration, the chain length will increase and decreases with the increase in the number of double bonds (unsaturated level) [32-35]. However, overall, the density and viscosity are still in the range of physical properties, standard of SNI.

Figure 5 shows the effect of seed:methanol ratio on the yield of biodiesel. Increasing the seed:methanol ratio lead to the increase of yield, however above the ratio 1:2.0 there was no increment of this yield. This is probably due to the fact that excess methanol will disturb the reaction esterification equilibrium. Moreover, although the addition of more alcohol will adhere the reaction to the product side, however too excess alcohol will not be economically feasible.

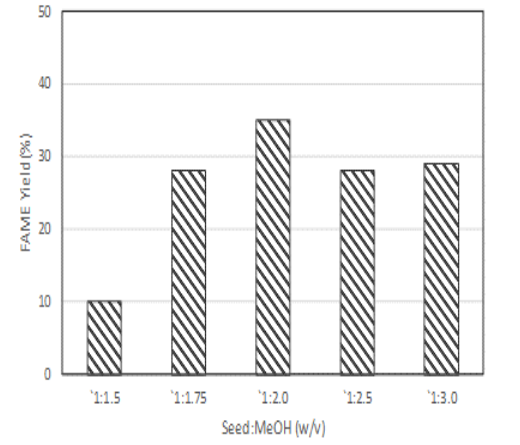

Figure 5. Effect of raw material to methanol ratio on the yield of FAME

In addition, methanol on ester layers can lower the flash point of biodiesel [4]. Therefore, in this experiment we conclude that the optimum process condition for rubber seed in situ esterification assisted by ultrasound irradiation was seed:methanol ratio =1:2.0, H_2SO_4 catalyst 1% and KOH catalyst 0.1%, at temperature of 55-60 ^oC.

3. 3. Biodiesel Product Analysis Besides kinematic viscosity and density, biodiesel was also investigated for its acid values. Acid values indicate the amount of KOH required to neutralize fatty acid in the biodiesel. If the acid number is high, then the level of fatty acid in the biodiesel is also higher. The biodiesel standard is acid values below 0.8 mg KOH/g.

Figure 6 represents the effect of H_2SO_4 catalyst (0.1-1% v/v) to acid values. All the acid values are below 0.8; fluctuating values were observed during the experiment. However, the lowest value of acid number was observed at 0.1% H_2SO_4 catalyst. Since the yield of 1% H_2SO_4 was greater than 0.1%, then we conclude that the optimum H_2SO_4 was 1%.

Referring to the effect of seed:methanol ratio (see Figure 7), the acid value has lowest number in the ratio of 1:1.75, which indicates that less seed was processed in the biodiesel production. Overall almost all of the acid values on biodiesel product met the biodiesel SNI standard (max. 0.8 mg KOH/g).

The comparison between the experiment which utilized ultrasound and that in the absence of ultrasound is shown in Table 2. The use of ultrasound showed significant improvement in reaction time, yield and other physical properties.

The FAME yield was observed to achieve optimum value at 35% at 1% H_2SO_4 catalyst, 0.1% KOH and 1:1.75 seed:MeOH ratio, while the yield of biodiesel was highest at 92% (calculated by Equation (2)). The FAME analysis using GC is shown in Figure 8.

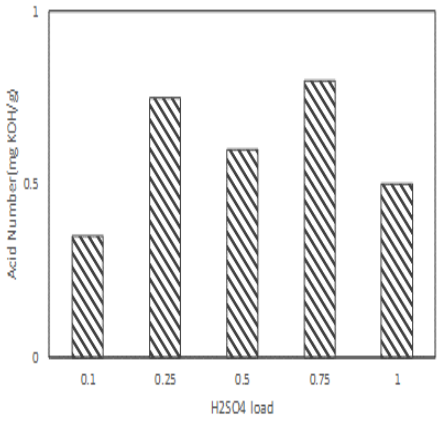

Figure 6. Effect of catalyst concentration on acid value

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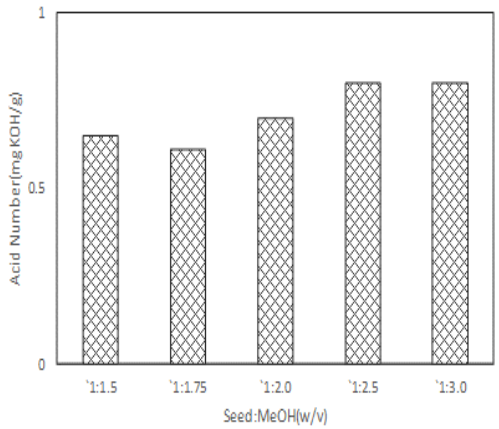

Figure 7. Effect of rubber seed ratio to methanol on acid value

TABLE 2. Comparison of products with and wihout ultrasound irradiation at conditions: 1% H_2SO_4 catalyst, 0.1% KOH and 1:1:75 seed:MeOH ratio

Parameters	With ultrasound	Without ultrasound	Standard
Viscosity (mm/s^2)	5.1	4.2	2.3-6
Denisty (g/mL)	0.91	0.85	0.8-0.89
Acid number (mgKOH/g)	0.82	0.75	<0.8
FAME yield (%)	25.2	35	
Biodiesel yield (%)	82.3	92.5	
Reaction time (min)	20	10	

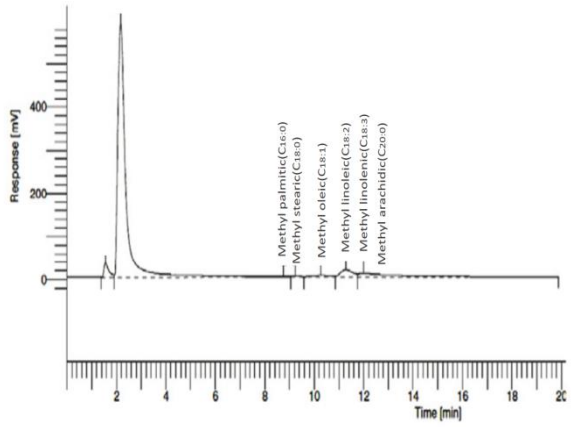

Figure 8. The GC analysis of FAME produced from rubber seed oil after esterification

The FAME contents were deteced within retention times of 8-13 min. The major component in FAME was methyl linoleic (C18:2)=42.5%, followed by methyl oleic (21.2%) and palmitic acid (8.6%).

4. CONCLUSSION

Rubber seed was investigated as non-edible feedstock for biodiesel production to overcome energy problems in replacing diesel fuel. Based on the results, we concluded that using acid catalyst, the highest FAME yield of 35% (92.5 % biodiesel yield) at seed: methanol ratio of 1:1.75 (w/v) with H_2SO_4 1% (v/v) and KOH 0.1% (w/v) was obtained. The experiment was conducted at 55-60 ^oC, 42 kHz frequency of ultrasonic waves, at atmospheric condition for 30 min. The characteristic of biodiesel product met the biodiesel standards according to the Indonesian National Standard/SNI. Furthermore, in situ method with ultrasond can be potentially used for biodiesel production especially for large scale applications.

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Ultrasonic

چکیده

از آنجا که روش استریفیکاسیون معمولی نیاز به زمان فرآوری طولانی داشته و بازده پایینی دارد، بهبود این فرایند هنوز هم یک موضوع جالب است که باید بررسی شود. به منظور کاهش هزینه استخراج روغن از دانه که تقریبا ۷۰٪ از هزینه کل فرایند را شامل می‌شود، فرایند استریفیکاسیون درجا اخیرا معرفی شده است. هدف از این مطالعه تولید بیودیزل از روغن دانه لاستیک از طریق استریفیکاسیون در محل، باکمک امواج فراصوت در ۴۲ کیلو هرتز به مدت ۳۰ دقیقه بود. آزمایش در دو مرحله افزودن کاتالیزور انجام شد: اسید سولفوریک (با غلظت ۰/۱–۱٪ حجمی/حجمی) و سود (۰/۱٪) و استریفیکاسیون در ۶۰ درجه سانتیگراد به مدت ۳۰ دقیقه تحت فرکانس صوت ۴۲ کیلو هرتز انجام شد. نتایج نشان داد که بازده بهینه اسید چرب متیل استر (FAME) ۳۵٪ بود که متناظر با بازده بیودیزل ۹۲/۵٪ بوده که تحت شرایط نسبت دانه لاستیک به متانول ۱: ۱/۷۵ (وزنی/حجمی)، بارگذاری کاتالیزور H_2SO_4 (۱٪) و بارگذاری KOH کاتالیزور (۰/۱٪) حاصل شد.

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