

# *The Role of Temperature on Biodiesel Production from Palm and Waste Cooking Oils Catalyzed By Silica-Titania Catalyst*

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**Abstract** - The optimization of reaction temperature on biodiesel production from palm and waste cooking oils has been investigated in the range of 50-70°C. The reaction has used silica-titania catalyst obtained from solid state reaction between solid precursors of silica and titania. The biodiesel products have been characterized by FTIR (Fourier Transformation Infra-Red) and examination of several properties such as density, flow rate, and acid number. The results show the FTIR spectra of biodiesel products are very similar with that of palm oil or waste cooking oil. The biodiesel product from palm oil shows an optimum temperature of 65°C. At this temperature, it shows the lowest density with the highest flow rate and the highest percentage of conversion, i.e. 33.33%. The biodiesel product from waste cooking oil shows the optimum temperature of 55°C that it performs the lowest density with the highest flow rate and the highest percentage of conversion, i.e. 57.1%.

**Keywords** - Biodiesel; Silica-Titania Catalyst; Density; Flow Rate; Temperature; Acid Value.

## I. INTRODUCTION

Biodiesel is an alternative fuel possessing several benefits with regarding to friendly environmental, non toxic, degradable, and less CO emission. This alternative fuel can produce from renewable and sustainable sources such as vegetable oil and animal fat [1]. The vegetable oils include unconsumed oils (hazelnut and castor oils) and consumed oils (palm, sunflower, and coconut oils). Palm oil is the highest consumption and production level vegetable oil[2],[3]. In general, the consumed palm oil contains very low free fatty acid. Indonesia is a very potential country for developing biodiesel production from palm oil because Indonesia is the biggest country in the world to produce palm oil[4]. In addition, the chemical composition and physico-chemical properties of biodiesel from palm oil are similar to that of diesel from petroleum oil and friendly environmental [5].

The waste cooking oil is also potential as raw material for biodiesel production. The advantages of this oil are more economics, available, and easily found in domestic house, restaurant, and industry as residues that can reduce cost of waste management and maintenance. Discharging waste directly to environment can damage the environment due to high content free fatty acid in waste cooking oil. Therefore, in order to overcome the problem the waste cooking oil can be advantaged as raw material for biodiesel production.

The utilization of palm or waste cooking oil as biodiesel sources depends on the type of catalyst used particularly heterogeneous catalyst group. Catalyst with acidic properties is more effective for waste cooking oil. The acid catalyst is non sensitive for free fatty acid and capable for both transesterification and esterification reactions simultaneously. The utilization of a base catalyst opens the possibility to yield

soap besides methyl ester (biodiesel). The application of base catalyst group is more effective for palm oil with low FFA (< 0.5%) [6], [7].

Silica-titania catalyst is one of many catalysts applied for biodiesel production. Previous investigations on silica-titania catalyst include addition of sulfate in silica-titania catalyst, correlation of titanium tetrahedral coordination with physical properties of biodiesel, effect of mol ratio of Si and Ti to produce silica-titania applied as catalyst for biodiesel production, and effect of catalyst mass for biodiesel production. On account of that reasons, the present work focuses on temperature optimization in biodiesel production from palm and waste cooking oils applying silica-titania catalyst synthesized by solid-state method. The silica-titania catalyst was synthesized using optimum condition obtained from previous works [8], [9], [10], [11]. The optimization of temperature has been conducted on the basis of several examinations of biodiesel products such as density, flow rate, and acid number.

## II. RESEACH METHOD

### 2.1 Material and Equipment

The required materials were used for synthesis of catalyst and biodiesel production. The synthesis of silica-titania catalyst required  $\text{SiO}_2$  (Sigma Aldrich),  $\text{TiO}_2$  (Merck), and toluene. The materials used for biodiesel production include palm oil (bimoli), waste cooking oil, and methanol (Merck). The equipments used in this work were required for catalyst synthesis and characterization.

The equipments for catalyst synthesis include glassware, hot plate, oven, balance, stirrer, centrifuge, rotary evaporator, thermometer, furnace, and ultrasonic. The instruments for characterization and examination of physical properties are FTIR spectrophotometer, picnometer, and viscometer.

### 2.2 Procedure

The method for synthesis of silica-titania was combined from the best conditions showed in the previous works [12],[8], [10]. The mol ratio of Si/Ti was 1:0.5 while the solid state temperature reaction conducted at 450oC for 8h [11]. The silica-titania catalyst was then applied in the reaction of tranesterfication between palm oil and methanol and followed in the reaction of waste cooking oil and methanol. The procedure to produce biodiesel was chosen from the best performance of silica-titania in the previous studies [12],[8], [11]. The mixture of oil source, methanol and catalyst was placed in reflux system for 5h in various temperatures (50, 55, 60, 65, and 70oC). The mol ratio of oil to methanol was 1:6

while the ratio of catalyst to oil was 7% of oil mass. The catalyst was then removed by centrifuge after reaction and continued by heating the product above the boiling point of methanol to remove the excess methanol. In order to investigate the role of temperature, the biodiesel product was characterized by FTIR and analyzed several properties such as density, flow rate and acid number [9].

Table 1. Table of sample label

| Sample            | Label | Sample            | Label |
|-------------------|-------|-------------------|-------|
| Palm Oil          | PO    | Waste Cooking Oil | WCO   |
| 50 <sup>o</sup> C | BPO50 | 50 <sup>o</sup> C | WCO50 |
| 55 <sup>o</sup> C | BPO55 | 55 <sup>o</sup> C | WCO55 |
| 60 <sup>o</sup> C | BPO60 | 60 <sup>o</sup> C | WCO60 |
| 65 <sup>o</sup> C | BP065 | 65 <sup>o</sup> C | WC065 |
| 70 <sup>o</sup> C | BPO70 | 70 <sup>o</sup> C | WCO70 |

## III. RESULTS AND DISCUSSION

### 3.1 FTIR Characterization of Biodiesel Product

The FTIR is used to study the change in characterization of functional groups of oil source to yield biodiesel applying optimum reaction temperature on the basis of FTIR spectra of palm and waste cooking oils as oil sources and biodiesel products.

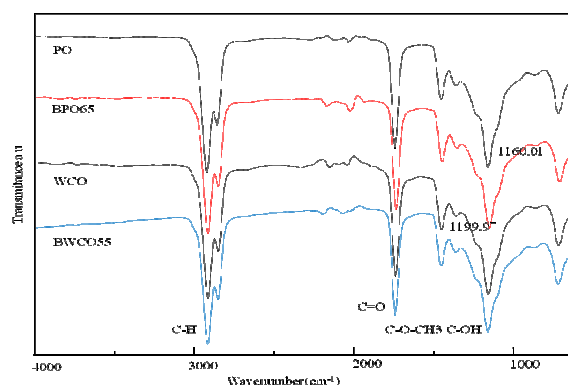


Fig 1. FTIR spectra of palm oil, waste cooking oil, and biodiesel products applying optimum reaction temperature

As shown in Fig.1 the FTIR spectra of biodiesel products are not too different from that of oil sources. There is a change in chemical bonding attributed to absorption band at wavenumber range of 1170 – 1200  $\text{cm}^{-1}$  showed the vibration of C-O-CH<sub>3</sub> of methyl ester[8]. The shifts of absorption peaks due to conversion of palm oil and waste cooking oil, respectively, to biodiesel are shown at wave number of 1160.01  $\text{cm}^{-1}$  and 1199.97  $\text{cm}^{-1}$ . In addition, the absorption

band at wavenumber of 1743  $\text{cm}^{-1}$  of oil sources and biodiesel products indicated C = O vibration. The shift of absorption peak is due to conversion of ester group of oil sources to form methyl ester of biodiesel product[13].

### 3.2 Examination on several parameters of biodiesel product

#### 1) Flow rate

The flow rate examination is used to determine viscosity since viscosity is in reversed correlation with its flow rate. On the other hand, increased flow rate causes a decrease in viscosity.

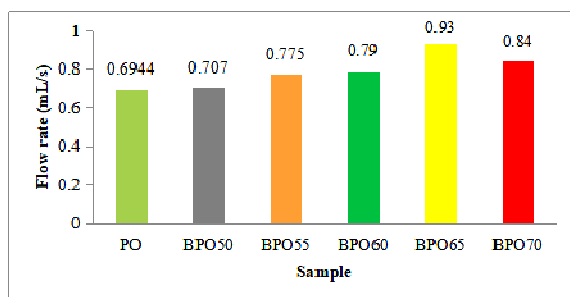


Fig.2 Flow rates of biodiesel products and palm oil (bimoli)

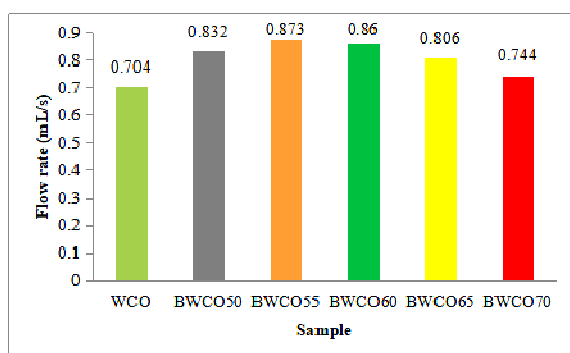


Fig. 3 Flow rates of biodiesel products and waste cooking oil

Fig. 2 shows the flow rates of biodiesel products at temperature range of 50 – 70°C compared to that of palm oil (bimoli). Increasing temperature until 65°C causes faster movement of compound molecules related to higher kinetic energy yielding larger molecule collisions[14]. Besides, at 65°C all methanol reacted with triglyceride (three molecules fatty acid in vegetable oil). Then the flow rate of biodiesel product decreased at temperature above 65°C (i.e. 70°C). When the temperature increased above the boiling point of methanol, bubbles formed on liquid surface retarded phase change[15].

Fig. 3 shows the increased flow rates of biodiesel products due to increased temperature until 55°C, then the flow rates decreased at increasing temperature until 70°C. This

phenomenon is related to natural properties of acid catalyst effective for high FFA vegetable oil that at 55°C all FFA in waste cooking oil converted to methyl ester[16]. The decreased flow rate above 55°C is also due to less interaction between vegetable oil and methanol since most methanol at that temperature changed to gas phase [14].

#### 2) Density

Density is the second assay of biodiesel products from oil sources (palm and waste cooking oils). The result is as follows:

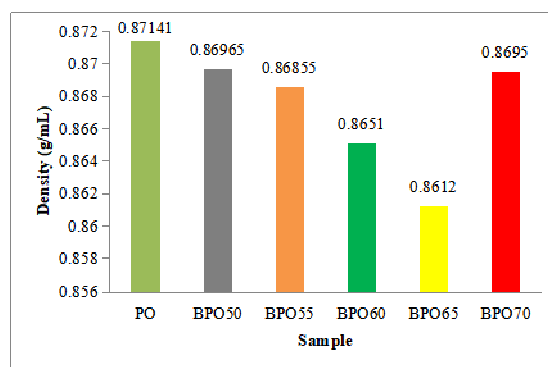


Fig. 4 Densities of biodiesel products and palm oil (bimoli)

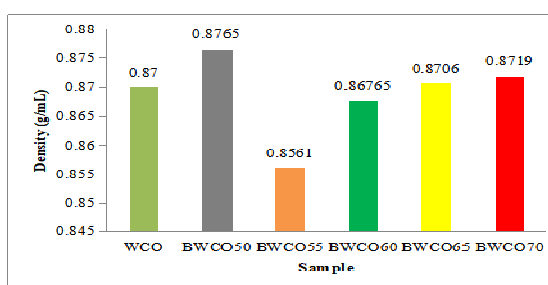


Fig. 5 Densities of biodiesel products and waste cooking oil

Fig. 4 shows the densities of biodiesel products from palm oil continually reduced at temperature range of 50-65°C, but the density increased again at 70°C. This trend is consistent with that of flow rate of biodiesel products from palm oil. Fig. 5 shows a rather peculiar pattern of density attributed to densities of biodiesel products from waste cooking oil. Temperature of 50°C is not enough to reduce density of biodiesel product lowered than that of waste cooking oil source. The increased density of biodiesel product at 50°C compared to that of waste cooking oil source is probably related to increased material mass due to catalyst effect. However, the density of biodiesel product decreased at 55°C attributed to conversion of waste cooking oil to biodiesel due to silica-titania catalyst role [17].

### 3) Acid value and percentage of conversion

The assay of acid value is to determine the FFA content in biodiesel products compared to that of palm and waste cooking oil sources. Fig. 6 and Fig. 7 presented the results of acid value of biodiesel products and oil sources.

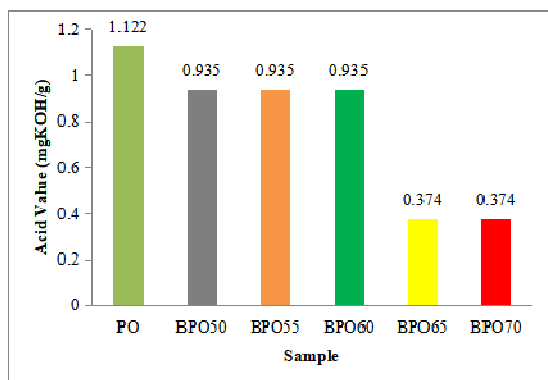


Fig. 6 Acid values of biodiesel products and palm oil (bimoli)

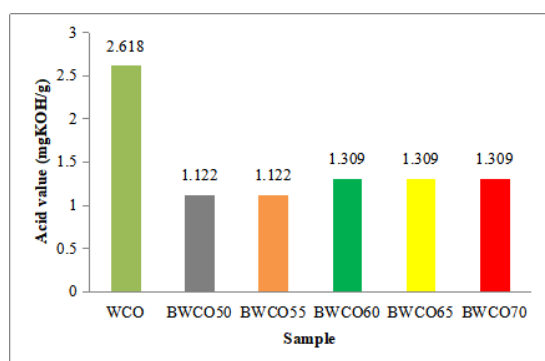


Fig. 7 Acid values of biodiesel products and waste cooking oil

Fig. 6 shows acid values of biodiesel products (50 – 70°C) lower than that of palm oil (Bimoli). The acid value of palm oil (Bimoli) is found to be 1.122 mg KOH/g and biodiesel products (65° and 70°C) are lower, i.e. 0.374 mg KOH/g. Fig. 7 shows acid values of biodiesel products (50° – 70°C) lower than that of waste cooking oil source. The acid value of waste cooking oil is found to be 2.618 mg KOH/g and the acid value of biodiesel products give the lowest number of 0.561 mg KOH/g.

Based on acid number data, the % conversion of palm and waste cooking oils to biodiesel can be determined. Table 2 and Table 3 present data of % FFA and conversion of oil sources to biodiesel.

Table 2. %FFA and conversion of palm oil to biodiesel

| Sample | Acid number | % FFA  | % conversion |
|--------|-------------|--------|--------------|
| PO     | 1.122       | 0.561  |              |
| BPO50  | 0.935       | 0.4675 | 16.67%       |
| BPO55  | 0.935       | 0.4675 | 16.67%       |
| BPO60  | 0.935       | 0.4675 | 16.67%       |
| BPO65  | 0.374       | 0.374  | 33.33%       |
| BPO70  | 0.374       | 0.374  | 33.33%       |

Table 3. %FFA and conversion of waste cooking oil to biodiesel

| Sample | Acid number | % FFA  | % conversion |
|--------|-------------|--------|--------------|
| WCO    | 2.618       | 1.309  |              |
| BWCO50 | 0.561       | 0.561  | 57.1%        |
| BWCO55 | 0.561       | 0.561  | 57.1%        |
| BWCO60 | 0.545       | 0.6545 | 50%          |
| BWCO65 | 0.6545      | 0.6545 | 50%          |
| BWCO70 | 0.6545      | 0.6545 | 50%          |

Table 2 presents that FFA content (%) of biodiesel products lower than that of palm oil (Bimoli). This is due to conversion of FFA in vegetable oil to FAME (Fatty Acid Methyl Ester) or biodiesel product [11]. The highest conversion (33.33%) at 65°C is shown by conversion of palm oil to biodiesel. Table 3 presents that % FFA of biodiesel products showed an increased trend compared to that of waste cooking oil. This is related to the role of silica-titania acid catalyst more appropriate to be used for vegetable oil with high FFA content[18]. Therefore, this is resonable that silica-titania catalyst is more effective for waste cooking with higher FFA content rather than that of palm oil (Bimoli) with lower FFA content. The percentage conversion of palm and waste cooking oils to biodiesel products are found to be 33.33% (65°C) and 57.1% (55°C), respectively.

### IV. CONCLUSIONS

This study found the optimum reaction temperature of 65°C and 55°C for biodiesel production from palm oil and waste cooking oil, respectively. The optimum temperature for conversion of waste cooking oil to biodiesel (55°C) is found to be lower than that of palm oil (65°C). A conclusion can be drawn that silica-titania catalyst is more effective and

appropriate to be used for catalytic action on vegetable oil with high FFA content.

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