The Effect of Silica-Titania Catalyst Loading on the Production of Biodiesel from Palm and Waste Cooking Oil

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The Effect of Silica-Titania Catalyst Loading on the Production of Biodiesel from Palm and Waste Cooking Oil

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Abstract:

A study about the effect silica-titania catalyst loading on biodiesel production from palm oil and waste oil has been conducted. The silica titania catalyst has been synthesized by solid state method and characterized by FTIR and DR UV-Vis to investigate the formation of Si-O-Ti bond from Si and Ti precursors. The synthesized silica-titania catalyst has been applied for biodiesel production with variation of catalyst mass to oil as to be 1%, 3%, 5%, 7% and 9%. The biodiesel products have been examined for their physical properties such as density, flow rate, and acid number. The percentage of conversion obtained to be 91% for waste cooking oil (1.33% FFA) and 33.33% for palm oil (0.374% FFA). The finding is justified that silica-titania catalyst has active acid site formed through Si-O-Ti bonding.

Keywords —**Silica-titania Catalyst, Biodiesel, Titanium Tetrahedral, Transesterification, FFA**

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I. INTRODUCTION

Biodiesel can be produced from transesterification reaction of vegetable oil or its waste cooking oil with a short chain alcohol in the existence of a catalyst [1,2]. In general, the catalyst used for biodiesel production can be homogeneous or heterogeneous catalyst. The main constraint in the usage of a homogeneous catalyst is in the complicated separation process of catalyst from the product obtained due to same phase of catalyst and product. Besides, the usage of homogeneous catalyst yields soap by product that gives another obstacle related to lower biodiesel product and more catalyst consumption [3,4]. Therefore, the application of heterogeneous catalyst gives more benefit compared to that of homogeneous catalyst. The separation process of heterogeneous catalyst and product is more simple due to different phase [5].

The silica-titania is one of important heterogeneous catalysts in titanosilicate group related to broad applications in the field of catalysts, photocatalysts, dielectricum material and others. Previously, two series of silica-titania with SiO_2 and TiO_2 solid precursors have already been synthesized by solid state method mole variations of precursors and calcinations temperatures [6].

This study emphasizes on the silica-titania synthesis with titanium tetrahedral framework. Based on study literature, the formation of titanium tetrahedral framework is shown through the formation of Si-O-Ti bonding. The formation of Si-O-Ti bonding increases the Bronsted acid , hence, the surface acidity increased. This matter is due to geometrical different between Si and Ti [7].

The application of this two series of silica-titania in biodiesel production from palm oil is shown by the physical properties of their products in relation to density, viscosity, and boiling point, which shows that titanium tetrahedral coordination has effect on the biodiesel production.

In relation to their physical properties, the quality of biodiesel production is better ongoing with the increased titanium tetrahedral fraction in silica-titania catalyst. However, previous work has not yet investigated the percentage of conversion from vegetable oil to biodiesel and optimization of catalyst mass in the transesterification reaction. Therefore, this work investigates on the optimization of catalyst mass to produce biodiesel from palm oil and its waste oil. The application of this two types of vegetable oil with different in %FFA is to study the role of SiO_2-TiO_2 as acid or base catalyst. .

II. MATERIALS AND METHODS

A. Materials and Instruments

This study needs materials used for catalyst synthesis and biodiesel production. The synthesis of silica-titania catalyst needed TiO_2 (Across), SiO_2 (Sigma Aldrich), and toluene (Merck). The materials for biodiesel production included palm oil (Bimoli), waste oil, and methanol (Merck), and also several materials used for acid number analysis.

The instrumentations used in this study included instruments for synthesis and characterization of catalyst. Apparatus for synthesis consisted of glassware, hot plate, furnace, balance, thermometer, stirrer, centrifuge, ultrasonic and rotary evaporator.

Instruments used for characterization and analysis of physical properties consisted of titration, picnoimeter, viscometer, FTIR, and DR UV-Vis.

B. Procedure

The preparation for silica-titania catalyst followed the optimum condition obtained from previous investigation [6, 8, 9] that is the mole ratio of Si and Ti was set to be 1:0.5 using SiO_2 and TiO_2 solid precursors, calcinations temperature of 450°C for 8h [6]. The catalyst obtained was characterized by FTIR and DR UV-Vis to justify the formation of silica-titania.

The biodiesel production was obtained through transesterification reaction between palm oil (Bimoli) or waste oil and methanol in the presence of silica-titania catalyst. The reaction was conducted in a three bottle neck Erlenmeyer (250 mL) with reflux system provided with a thermometer (360°C) setting at 65°C for 3h. The mole ratio between methanol and vegetable oil is made to be 6:1 [8]. The quantity of catalyst used is varied 1 – 9% of weight of palm oil and waste oil, respectively. Then the mixture was cooled after 5h reaction. Then the separation process between product, catalyst, and excess methanol was done. The first separation to remove catalyst was conducted using a centrifuge. In this separation process, the glycerol formed was separate from the mixture because the glycerol and the catalyst have the same phase.

The following separation was done to evaporate the excess of methanol using a rotary evaporator at temperature higher than boiling point of methanol (64.7°C) [8]. To prove the conversion of palm and waste cooking oil into biodiesel, the product was then determined by density, flow rate, and acid number with the following equation:

Density : $\rho = \frac{m}{V}$

ρ = density (g/mL)

m = (mass of picnometer+sample) - (mass of empty picnometer) (g)

V = Volume of picnometer (mL)

Flow Rate : $\frac{V}{t}$

V = Volume of sample (mL)

t = flow rate time (s)

$$\text{Acid Number} = \frac{56{,}1 \text{ x V x N}}{m}$$

V = Volume of titration (mL)

N = Normalitas of KOH

m = Mass of sample (g) [10].

Furthermore, the acid number, %FFA and % conversion, can be determined by the following equations, respectively

III. RESULTS AND DISCUSSION

A. FTIR dan DR UV-Vis spectra of silica-titania catalyst.

The physicochemical of silica-titania catalyst was studied through characterizations by FTIR and DR UV-Vis spectra. Fig. 1 shows the FTIR spectrum of silica-titania catalyst. The spectrum obtained is similar with that of FTIR spectrum from previous investigation [7]. The main peak of this FTIR spectrum is the absorption band of Si-O-Ti. This absorption band appeared very weakly at wave number of 960 cm^{-1}[6].

In order to confirm the existence of titanium tetrahedral fraction, the FTIR result was confirmed by deconvolution spectra of DR UV-Vis (Fig.2). The titanium tetrahedral fraction has the absorption band at wavelength from 200 to < 270 nm [7]. On the basis of deconvolution spectrum of DR UV-Vis, the percentage of titanium tetrahedral fraction was found to be 31%. The FTIR result was found in agreement with that of DR UV-Vis spectrum.

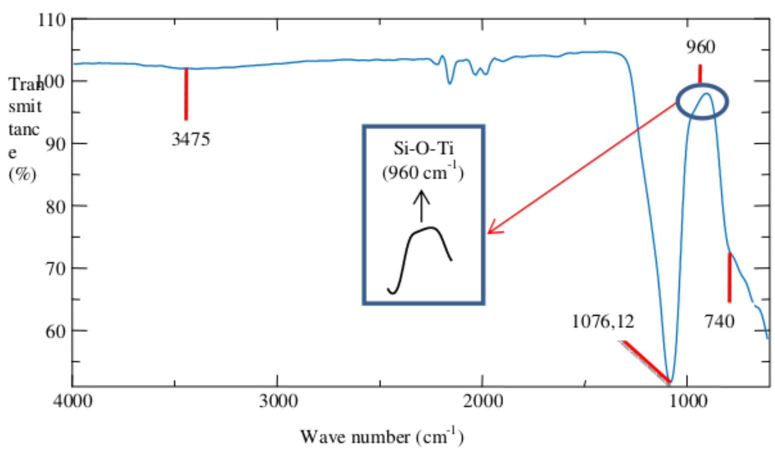

Fig. 1. FTIR spectrum of synthesized silica-titania catalyst.

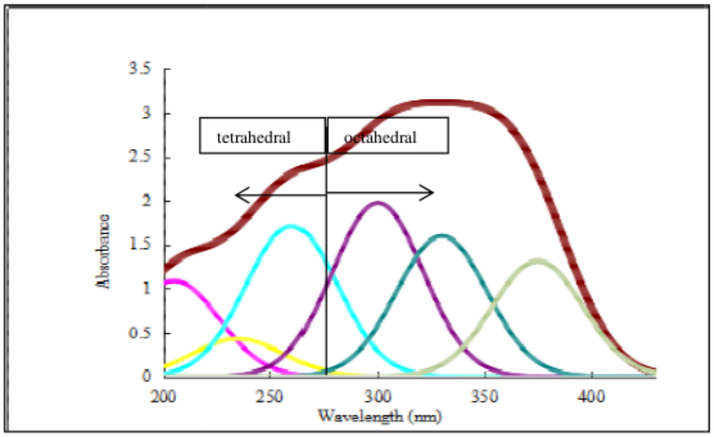

Fig. 2. Deconvolution of DR UV-Vis spectra of SiO_2-TiO_2

B. The effect of mass variation of catalyst on biodiesel physical properties.

1) Density and Viscosity (Flow Rate)

The effect of mass variation of catalyst on biodiesel product are shown in Fig. 3 – 6. Fig. 3 and 4 show the effect of mass variation of catalyst on densities of biodiesel from palm oil and its waste oil, respectively.

Fig. 5 and 6, respectively, shows the effect of mass variation of catalyst on viscosity represented by flow rate of biodiesel from palm oil and its waste oil, respectively. The density of palm oil and its waste oil before reaction is measured to be 0,90900 and 0,87105 g/ml, respectively.

With regard to Fig.3, the usage of 7% mass of silica-titania catalyst to vegetable oil could reduce density from 0,90900 to 0,8654 g/ml. The density of biodiesel from palm oil in then increased after increasing mass of catalyst more than 7%. This assumes with increased density in biodiesel or absorption of sample on the surface of catalyst hindering enzyme function.

Fig. 4 shows the density of biodiesel from waste oil. For the conversion of waste cooking oil, the usage of 1% mass of catalyst shows optimal condition in decreasing density. The addition of more catalyst causes increased biodiesel density. In general, the

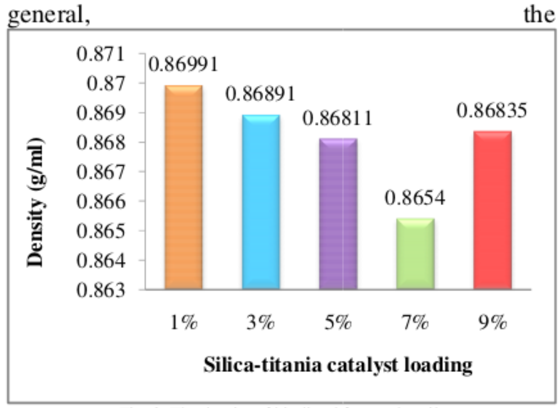

Fig. 3. The density of biodiesel from palm oil.

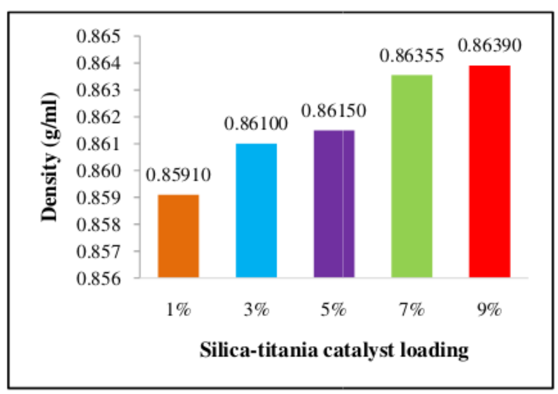

Fig. 4. The density of biodiesel from waste cooking oil.

densities of biodiesel from palm oil and its waste cooking oil are in the standard ASMTI rank.

Fig. 5 shows the mass variation of catalyst on flow rate of biodiesel from palm oil. Before reaction, the flow rate of palm oil is measured as 0,81235 ml/s and the flow rate increased until catalyst mass of 7%. This finding shows that increasing of catalyst mass until 7% could reduce viscosity or increase flow rate until 1,17645 ml/s. The increasing of catalyst mass more than 7% causing flow rate reduced. Fig. 6 shows the effect of mass variation of silica-titania catalyst on biodiesel product from waste cooking oil. Before reaction, the flow rate of waste cooking oil was measured as 0,8000 ml/s and the usage of 1% catalyst mass causing the flow rate increased significantly until 1,0630 ml/s. Then the flow rate reduced by increasing catalyst mass. The result of density examination is in agreement with that of flow rate examination.

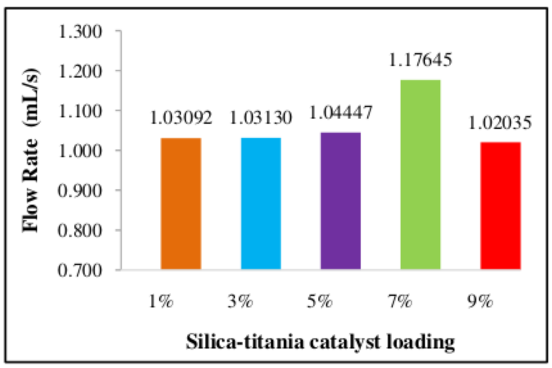

Fig. 5. The flow rate of biodiesel from palm oil.

The decreasing of flow rate after optimal condition gained is due to several factors. The first factor is related to salt of fatty acid (soap) from FFA and TiO_2 unreacted not to form silica-titania. The second factor is the excess of catalyst mass increasing viscosity impeding diffusion between reactant and catalyst causing lower ester production. The third factor is that the excess of catalyst mass causing biodiesel absorption on the surface of catalyst [11,12]

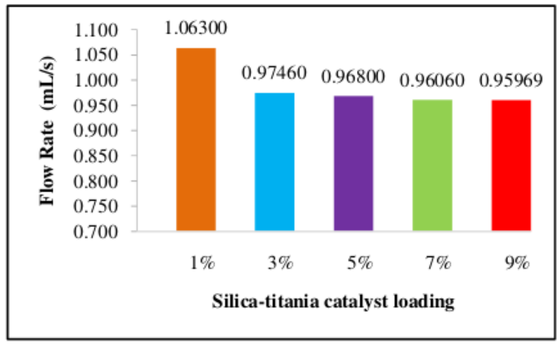

Fig. 6. The flow rate of biodiesel from waste cooking oil.

2) Acid Number and Percentage of Conversion

On the basis of Table 1, the 1% of catalyst mass increasing conversion until 3%. The addition of catalyst until 9% did not increase the % conversion or stable at 33.33%. In the case of biodiesel from waste cooking oil, the usage of 1% catalyst mass causing fast increasing until addition of 3% and 5% of catalyst mass. The results are in agreement with that findings of density and viscisity. However, the % conversion at 7% of catalyst mass yields same value as that of the usage of 1% of catalyst mass, and decreased again at the usage of 9% of catalyst mass (Table 2).

TABLE 1.
ACID NUMBER, %FFA AND % CONVERSION OF PALM OIL TO BIODIESEL

Sample	Acid Number	% FFA	% Conversion
Palm oil	1,122	0,561	
1%	0,935	0,4675	16,67
3%	0,748	0,374	33,33
5%	0,748	0,374	33,33
7%	0,748	0,374	33,33
9%	0,748	0,374	33,33

TABLE 2.
ACID NUMBER, %FFA AND % CONVERSION OF WASTE COOKING OIL TO BIODIESEL

Sample	Acid number	% FFA	% Conversion
Waste cooking oil	14,96	7,48	
1%	1,309	0,6545	91,25
3%	1,496	0,748	90
5%	1,496	0,748	90
7%	1,309	0,6545	91,25
9%	1,496	0,748	90

On the basis of % conversion, generally the results show that waste cooking oil gave % conversion much higher than that of palm oil. On the basis of literature study, a catalyst with acid character is suitable for vegetable oil with high value of FFA, and a catalyst with base character is suitable for vegetable oil with low FFA (< 5%). Therefore, it can be deduced that silica-titania catalyst is a heterogeneous acid catalyst due to its high acticity toward waste cooking oil with % FFA of 7.84%.

IV. CONCLUSION

The silica-titania catalyst synthesized from solid silica and titania using solid state method can be applied fro biodiesel production. The optimum mass of silica-titania catalyst obtained by this investigation is found to be 7% of catalyst mass for biodiesel product from palm oil and 1% of catalyst mass for biodiesel product from waste cooking oil. The silica-titania catalyst is attributed as heterogeneous acid catalyst due to higher percentage conversion of waste cooking oil (higher % FFA) to biodiesel product compared to that of palm oil (lower % FFA).

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