Preparation and Characterization of Heterogeneous Catalyst from *Gelam* Wood (*Melaleuca leucadendron*) for Biodiesel Production

Abdullah Unlam*,**, Azidi Irwan*, Utami Irawati*‡, Nor Ain**, Nasrullah Hamdani*, Akif Lanang Jaya*

*Department of Chemistry, Faculty of Mathematics and Natural Sciences, Lambung Mangkurat University, Banjarbaru, Indonesia

**Integrated Laboratory, Lambung Mangkurat University, Banjarbaru, Indonesia

(abdullahunlam@gmail.com, airwan@ulm.ac.id, uirawati@ulm.ac.id, ainda.anwar25@gmail.com, 1811012110001@mhs.ulm.ac.id, 1811012210022@mhs.ulm.ac.id)

‡Corresponding Author: Utami Irawati, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Lambung Mangkurat University, Jl. A. Yani KM. 36 Banjarbaru 70714, South Kalimantan, Indonesia, uirawati@ulm.ac.id

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Abstract- Recently, many researchers have explored the potential use of ash as a catalyst, due to the availability of various mineral elements in it. The ashes themselves can be obtained from various agricultural waste of biomass, including from the burning of woods. In this study, the ash that was used as a raw material for a heterogeneous catalyst was obtained from the burning of *gelam* wood (*Melaleuca leucadendron*). After the burning, the ash was sieved to have particles of homogenous size. The ash was then activated with a solution of 1 M H_2SO_4 and 0.1 M KOH, consecutively. Potassium was then impregnated onto the activated ash using 30% and 60% (w/w) KOH solution, followed by calcination at 800 °C for 3 hours. The impregnated catalysts were then characterized with FTIR, XRD, and SEM-EDX. The catalyst was tested for its ability in the transesterification reaction of palm oil by varying the methanol to oil mole ratio, the amount of catalyst used, and the reaction time. The optimal reaction conditions for biodiesel production using this catalyst include a 12:1 methanol to oil mole ratio, 10.0% weight ratio of the catalyst (catalyst weight to the oil volume), 6 hours of reaction time at 65 °C and stirring speed of 600 rpm. By using this catalyst, the biodiesel production reached up to 99.0% in conversion rate, with a product that satisfactorily meets the ASTM D6751 standards in terms of its density, kinematic viscosity, and acid number.

Keywords Catalyst, *gelam* wood ash, palm oil, biodiesel.

1. Introduction

Biodiesel is an alternative fuel to conventional diesel oil (diesel). Biodiesel is produced from the conversion of triglycerides into a mixture of methyl esters. Triglyceride compounds can be derived from sunflowers [1], soybean oil [2], peanut oil [3], corn oil [4], canola oil [5], waste cooking oil [6], non edible oil [7], and rubber seed oıl [8]. Biodiesel production in Indonesia mostly uses palm oil as a raw material.

In producing biodiesel, either acid or base catalyst can be used. Most researchers prefer base catalysts because of their non-corrosive nature [8]. Some basic catalysts commonly used are CaO [8], NaOH, KOH, CH_3ONa, and CH_3OK [9]. In addition, researchers also use ash as an alternative catalyst in biodiesel production. Some examples of ash sources as catalysts are peanut shells [9], tamarind fruit peels [10], rice husks [11], weed [12], acacia wood [13], sugarcane bagasse [14], corn cobs [15]. Those biomasses can be used as a source of catalysts due to their various mineral content.

Like other biomass, the *gelam* tree (*Melaleuca leucadendron*) also produces ash if burned. *Gelam* woods grow in the wetlands area with abundant availability in the province of South Kalimantan, Indonesia. Recently, *gelam* woods are mainly used for building constructions, firewood and charcoal materials [16]. Despite its high availability and

renewability, research on the use of *gelam* wood as a catalyst in biodiesel production has never been reported. The use of *gelam* wood as a catalyst has not been reported by researchers to date. Thus, a research on using ash from *gelam* wood as a catalyst for biodiesel production is interesting considering the potential benefits. In light of that, this research aims to investigate the synthesis of a heterogeneous catalyst prepared from *gelam* wood, and to examine the activity of said catalysts. In this study, *gelam* wood was burned. The ash produced was then activated with an acid and base solution, followed by a KOH base impregnation and calcination at 800 °C for 3 hours. The performance of the catalyst was then observed in the palm oil transesterification reaction, based on the density, kinematic viscosity, and the acid number of the biodiesel yielded. The ASTM D6751 biodiesel standard was used as a comparison.

2. Materials and Methods

2.1. Materials

Some of the materials used in the study were *gelam* wood, palm oil which was obtained from PT. Bitung Manado Oil Ltd (Bimoli), methanol (E-Merck), KOH (E-Merck), H_2SO_4 (E-Merck), universal pH indicators (E-Merck), Whatman 42 filter paper (E-Merk), boiling stones, and distilled water. All the chemicals used were of chemical grades and used as received without further purification.

2.2 *Method*

2.2.1 *Preparation of ash*

After its skin was peeled off, *gelam* wood was cut into small pieces and then air-dried under the sun. Dried *gelam* wood was then burned into ashes on a stove. After all the wood has been completely burned, the ashes were sieved using a 60 mesh sieve. The ashes were then stored in a tightly closed bottle. Some of the ash was calcined at 800 °C for 3 hours.

2.2.2 Ash Activation

As much as 25 g of the ashes were refluxed in 125 mL of 1 M H_2SO_4 solution at 100 °C and stirred with a 500 rpm speed for 3 hours. This ash mixture was then poured into a Buchner funnel to be neutralized using distilled water. The ashes were then dried at 105 °C, followed with another reflux procedure using 125 mL of 0.1 M KOH solution in the same condition as the previous reflux process. Some of the ashes were then calcined at 800 °C for 3 hours.

2.2.3 KOH impregnation on ash

As much as 8.81 g of dry ash was impregnated with 2.65 g of KOH (30% w/w), and as much as 6.85 g of ash was impregnated with 4.11 g of KOH (60% w/w). The KOH solution itself was prepared by dissolving KOH in 50 mL of distilled water. The dry ash-KOH mixtures were then heated to evaporate the water content.

2.2.4 Calcination

The KOH-impregnated *gelam* wood ashes were then calcined in a furnace at 800 °C for 3 hours. The activated ash which was impregnated by KOH (30% w/w) was labeled as Catalyst-1, while the one being impregnated with KOH (60 % w/w) were labeled as Catalyst-2. Both of the catalysts were then characterized using FTIR, XRD, and SEM-EDX instruments

2.2.5 Catalyst Activity

The catalysts were then studied for their performance in the transesterification reaction of palm oil. The catalysts examined here were: (i) untreated ash, (ii) calcinated ash, (iii) activated calcinated-ash, (iv) Catalyst-1 and (v) Catalyst-2. For each catalyst, the amount of the catalyst added was adjusted to have a 5.0% (w/v) concentration of catalysts in the oil. All of the transesterification reactions were carried out at 65 °C for 3 hours with a 600 rpm of agitation speed, where the mole ratio of methanol:oil was 6:1. After the reaction was complete, the products were then poured into a separating funnel and left to stand for 24 hours. Catalysts that satisfyingly produced biodiesel were then tested further, where the methanol:oil mole ratio was varied (6:1, 9:1, 12:1). This further examination was also carried out by varying the amount of the catalyst (5.0; 7.5; 10.0%), and reaction time (3; 5; 6 hours)

2.2.6 Determination of Biodiesel Conversion Value

Conversion in this study was determined through the following calculations:

$$\underset{(x\ \text{mole})e}{\text{Palm oil}} + \underset{(3x\ \text{mole})}{3\ \text{methanol}} \rightleftharpoons \underset{(3x\ \text{mole})}{3\ \text{methyl ester (biodiesel)}} + \underset{(x\ \text{mole})}{\text{glycerol}}\ldots \quad (1)$$

$$x\ \text{mol palm oil} = x\ \text{mol glycerol} = \text{theoretical number of moles of glycerol} \ldots \quad (2)$$

$$\text{mol glycerol theoretical} \times \text{WM glycerol (92 g/mol)} = \text{theoretical weight of glycerol} \ldots \quad (3)$$

$$\text{Conversion (\%)} = (\text{actual glycerol weight} / \text{theoretical glycerol weight}) \times 100\% \ldots \quad (4)$$

2.2.7 Characterization of Biodiesel

Characterization of biodiesel characterization was conducted based on ASTM D6751 standards for density (D445), kinematic viscosity (D1298), and acid number (D664).

3. Results and discussion

3.1 Catalyst from Gelam Wood ash

The following is a picture of some of the ashes, i.e. (i) untreated ash, (ii) calcinated-ash, (iii) activated calcinated-ash, (iv) Catalyst-1 and (v) Catalyst-2. Based on these figures, it can be observed that each ash being used as catalysts gave products with different in colors from one another. Calcinated ashes shown in Figure 2 has a lighter color due to the calcination process of the ash. This discoloration is due to the loss of charcoal in the ash. The activated calcinated-ash shown on Figure 3 appeared to have a brighter color than the calcinated-ash, which might have come as a result of the activation treatment using acid and base solutions.

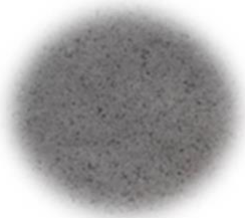

Fig. 1. untreated ash

Fig. 2. ash+calcination,

Fig. 3. ash+ activation+calcination,

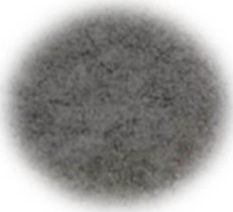

Fig. 4. Catalyst-1

Fig. 5. Catalyst-2

Meanwhile, as can be observed from Figure 4 (Catalyst-1) and Figure 5 (Catalyst-2) showing the color of catalyst-2 is darker than catalyst-1 because of the higher KOH.

3.2 FTIR, XRD, and SEM-EDX Analysis

The *gelam* wood ash and catalyst were characterized using FTIR, XRD, and SEM-EDX. Based on the FTIR analysis, the organic groups and minerals present in the *gelam* wood ash and catalysts can be determined (Figure 6.)

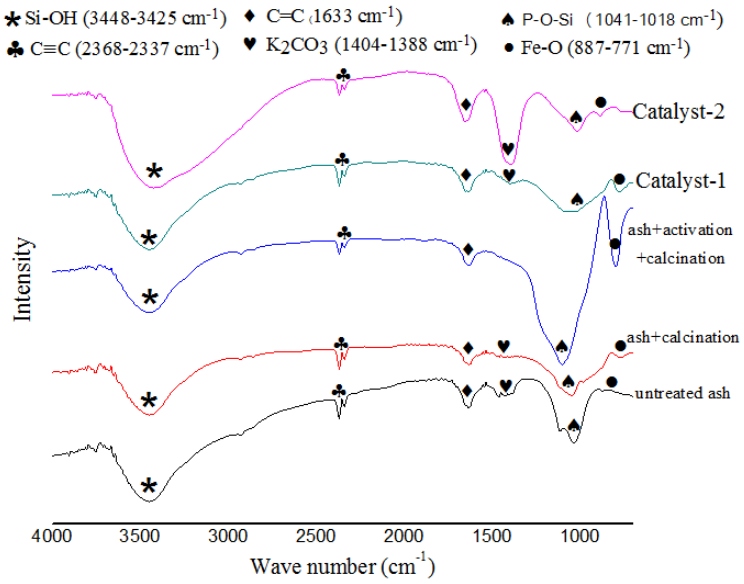

Fig. 6. FTIR spectra of *gelam* wood ash and catalyst

As can be observed from the FTIR spectrum shown in Figure 6, a broad peak appears at 3448-3425 cm^{-1}, which according to Wicaksana *et al.* [12] might come from the Si-OH vibration. The band at 2368-2337 cm^{-1} comes from C≡C vibrations, forwhile the peak at 1633 cm^{-1} appears as a result of C=C vibrations [17]. Peaks showing the presence of K_2CO_3 minerals appear at 1404-1388 cm^{-1}, the peak appears at 1041-1018 cm^{-1} that comes from PO-Si bond vibration, while the

peak at 887-771 cm^{-1} indicates the presence of Fe-O [18]. From Figure 6, it appears that the intensity of the K_2CO_3 in Catalyst-2 is greater compared to Catalyst-1, which might relate to the amount of KOH being impregnated onto the catalyst.

To determine the minerals constituting the catalyst and their crystallinity, the catalysts produced in this study were analyzed using XRD. The result of XRD analysis is shown below on Figure 7. Based on that figure, the mineral compositions in the catalysts comprise SiO_2 (2θ = 21°, 26°), K_2CO_3 (2θ = 28°, 33°), Fe_3O_4 (2θ = 29°, 31°), and FeO (2θ = 35°, 42°).

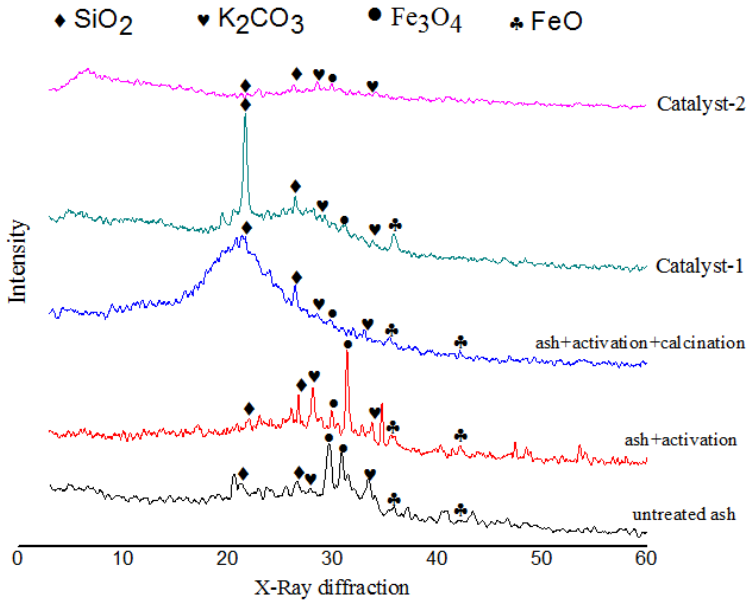

Fig. 7. The diffraction patterns of *gelam* wood ash and catalyst

From Figure 7, it can also be observed that the diffraction patterns of untreated ash and calcinated ash are similar. Thus, it can be inferred that the calcination treatment does not affect the mineral composition or crystallinity. As for the activated calcinated-ash, a decrease in the diffraction pattern intensity is observed in the area of 2θ = 28°-33° which represents a 2θ value for the K_2CO_3 and Fe_3O_4 minerals. This indicates that the crystallinity of those two compounds in the activated calcinated-ash has decreased due to the activation treatment with acid and base solutions. On the other hand, the diffraction pattern of the activated calcinated-ash shows an increase in intensity in 2θ = 21°, which corresponds to the area for SiO_2 minerals. This increase in intensity indicates that the activation treatment with acid and base can increase the crystallinity of the mineral SiO_2.

The diffraction pattern of Catalyst-1shows an increase of intensity in 2θ = 21°, that indicates that impregnating KOH base to the catalyst can increase the crystallinity of SiO_2. However, adding a higher concentration base (i.e: 60% (w/w KOH that was being added to Catalyst-2) caused the SiO_2 crystallinity of to decrease again. Based on the diffraction pattern of Catalyst-2, it can be observed that SiO_2, K_2CO_3, and Fe_3O_4 minerals in the catalysts are amorphous, of which K_2CO_3 is a mineral commonly used as the active compound of a heterogeneous catalyst.

Catalysts-1 and Catalyst-2 were then analyzed by SEM, and the result was compared with untreated ash. The result of SEM analysis for Catalyst-1 is shown in Figure 9, showing a better crystallinity when compared to untreated ash, which SEM result was shown in Figure 8. Figure 10 shows the SEM result for Catalyst-2 in which the crystallinity of the catalyst did not appear. The white spots that can be seen in Figure 10 are a manifestation of the element potassium (K) as a result of KOH impregnation onto Catalyst-2. This element will then act as active sites in heterogeneous catalysts for the conversion of palm oil to biodiesel.

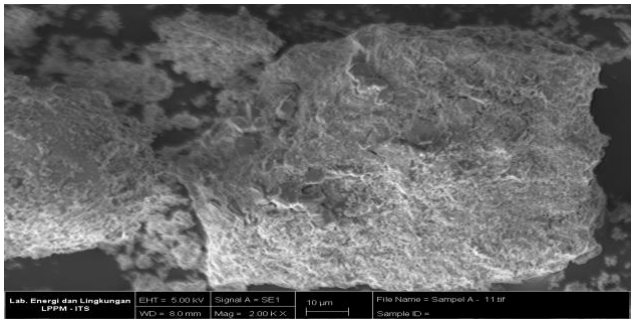

Fig. 8. SEM image of untreated ash

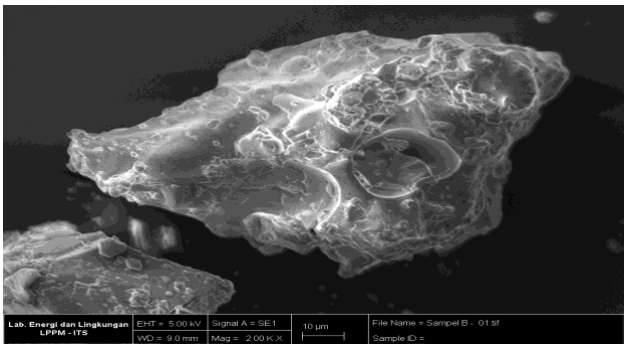

Fig. 9. SEM image of Catalyst-1

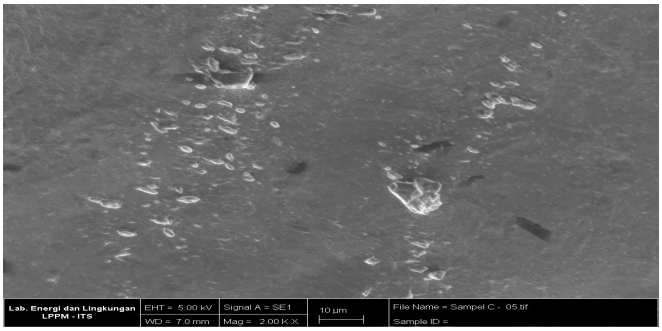

Fig. 10. SEM image of Catalyst-2

The catalysts were also analyzed by EDX to determine the elements that they contained. The result of the EDX analysis is shown in Table 1. From the table, it can be concluded that the main elements being present in Catalyst-1 and Catalyst-2 are O, Fe, C, Si, and K. Compared to the untreated ash, the content in both Catalyst-1 and Catalyst-2 has decreased. This is also confirmed with the result of XRD analysis which was shown in Figure 7. Based on Table 1, it can be seen that the elemental content of Si in Catalyst-1 and Catalyst-2 are higher than untreated ash. This shows that the activation treatment in the preparation of Catalyst-1 and Catalyst-2 were able to increase the SiO_2 content. The carbon content in Catalyst-1 and Catalyst-2 is higher than the carbon content in ash without treatment, which is very likely due to a decrease in other elements, especially Fe. The Fe content in the ash without treatment fell from 33.17% to 0.07% (Catalyst-1) and 0.13% (Catalyst-2).

Table 1 shows that the potassium (K) content in the untreated ash is higher when compared to Catalyst-1. This is probably the potassium originally present in the untreated ash was dissolved during the activation treatment when Catalyst-1 was being prepared. It is interesting to note that even though the preparation of Catalyst-1 involved impregnation of KOH onto the catalyst, the potassium content in Catalyst-1 is still lower than its initial amount. A different result was obtained for Catalyst-2, in which the amount of KOH being impregnated to the catalyst was two times higher than in Catalyst-1. Thus, compared to the untreated ash, the potassium content in Catalyst-2 has increased.

Table 1. Composition of elements found in the *gelam* wood ash catalyst*

Element	Untreated ash (Blanko)		Catalyst-1 (KOH 30%)		Catalyst-2 (KOH 60%)	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
O	63.15	82.30	64.18	66.98	70.59	73.04
Fe	33.17	12.38	0.07	0.02	0.13	0.04
C	2.65	4.61	14.83	20.62	12.39	17.08
P	0.57	0.38	0.73	0.40	1.35	0.72
Si	0.33	0.24	19.72	11.72	14.94	8.80
S	0.11	0.07	0.31	0.16	0.48	0.25
K	0.03	0.01	0.02	0.01	0.05	0.02
Na	0.00	0.00	0.09	0.06	0.05	0.04
Mg	0.00	0.00	0.01	0.01	0.00	0.00
Al	0.00	0.00	0.01	0.01	0.00	0.00
Ca	0.00	0.00	0.00	0.00	0.00	0.00

*Component analysis was done using EDX

3.3 Transesterification of Palm Oil into Biodiesel

Biodiesel production through the transesterification reaction of palm oil was carried out using (i) untreated ash, (ii) calcinated-ash, (iii) activated calcinated-ash, (iv) Catalyst-1 and (v) Catalyst-2. The reaction conditions for the reactions were as what has been described in the Methodology section. Table 2 presents the layer formation in palm oil transesterification.

Tabel 2. Palm oil transesterification

Catalyst	Formed 2 layers
Untreated ash	No
Ash+calcination	No
Ash+activation+calcination	No
1	No
2	yes

A further study was then carried out to investigate the optimum reaction conditions in the formation of biodiesel. The result is presented in Table 3 below.

Tabel 3. Biodiesel conversion using Catalyst-2

Mole ratio methanol:oil	Amount of catalyst (%)	Time (hour)	Conversion (%)
1:6	5.0	3	13.32
1:6	7.5	3	34.68
1:6	10.0	3	36.37
1:6	10.0	5	86.7
1:6	7.5	6	92.5
1:6	10.0	6	96.43
1:9	5.0	3	10.57
1:9	10.0	6	97.8
1:12	5.0	3	7.4
1:12	7.5	3	11.84
1:12	10.0	3	25.0
1:12	10.0	6	99.0

Description: weight of catalyst to volume of oil

The methanol:oil mole ratio variations were 6:1; 9:1 and 12:1. The conversion values for each said mole ratio were 96.43; 97.8; and 99.0%, respectively The highest conversion rate was obtained when the amount of catalyst used was

10.0% (w/v) to the oil volume, with a reaction time of 6 hours. The conversion value was determined using Equation 4. The kinematic viscosity value of the three biodiesel is as follows: 8.5; 7.03; and 5.3 (mm^2/s). Based on these values, the kinematic viscosity that meets the ASTM D6751 standard is the biodiesel produced from a 12: 1 mole ratio of methanol:oil.

The biodiesel giving optimum result was then tested for its density and acid number. The results for those two parameters are shown in Table 4.

Table 4. Physical and chemical properties of biodiesel

Property	ASTMD6751		Result
kinematic viscosity @40°C (mm^2/s)	D445	1.9-6.0	5.3
Density @15°C (kg/m^3)	D1298	800-880	853
Acid number (mgKOH/g)	D664	Max. 0.5	0.33

Based on Table 4, it can be concluded that the biodiesel produced with a methanol:oil mole ratio of 12:1, in which the amount of catalyst was 10.0%, along a 600 rpm of agitation speed and a 6-hour reaction time at 65 °C has produced biodiesel that satisfactorily meets the ASTM D6751 standard.

3.4 Catalyst Stability

To investigate the stability of the heterogeneous catalysts produced in this study, a catalyst was used repeatedly in biodiesel production. The stability of Catalyst-2 is determined by its conversion value, as shown in Table 5.

Table 5. Conversion value biodiesel use Catalyst-2

Repetition	Conversion value (%)
1	26.64
2	9.0

Reaction conditions: methanol:oil mole ratio (1:12), catalyst 10% (wt catalyst to oil volume), reaction time 6 hours, stirring speed 600 rpm, temperature 65°C

Based on the conversion values biodiesel in Table 5, it can be observed that the ability of the catalysts has decreased after the first and second usage. This result shows that the KOH impregnated onto *gelam* wood ash easily dissolves when it is used as a catalyst. This implies the need for further research on heterogeneous catalysts made from *gelam* wood, especially to increase the stability of the catalyst.

4. Conclusion

Gelam wood ash is a potential raw material to produce heterogeneous catalysts for the conversion of palm oil to biodiesel, but its stability remains to be an area of further research. A catalyst from *gelam* wood that gave an optimum performance was prepared by burning the wood into ashes, followed by activation using H_2SO_4 dan KOH. The activated ash was then impregnated with a 60% KOH solution was used, and then the catalyst was calcinated at 800 °C for 3 hours. The optimal reaction conditions for biodiesel production using this heterogeneous catalyst prepared in this study include a 12:1 methanol to oil mole ratio, 10.0% weight ratio of the catalyst to the oil volume), 6 hours of reaction time at 65 °C along with stirring speed of 600 rpm.

5. Acknowledgments

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