



Production of biodiesel from non-edible *Jatropha curcas* oil via transesterification using Bi_2O_3 – La_2O_3 catalyst



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ABSTRACT

The simultaneous esterification and transesterification of *Jatropha curcas* oil (JCO) was carried out in the presence of Bi_2O_3 (1–7 wt.%) modified La_2O_3 catalyst at atmospheric pressure. The catalyst were characterized by X-ray diffraction (XRD), BET surface area, desorption of CO_2 (TPD- CO_2) and NH_3 (TPD- NH_3). Under the optimal reaction condition of methanol/oil molar ratio of 15:1, 2 wt.% of catalyst amount and a reaction temperature of 150 °C for 4 h, the highest conversion of biodiesel obtained was 93%. This catalyst maintained 87% of FAME conversion after three times of successive reuse.

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1. Introduction

During this past decade, energy supply and its security have become a major issue around the world. Combustion of fossil fuels provides energy that makes technological advancement and economic growth of a country possible. Fossil fuels cause the emission of greenhouse gases and other type of air pollutant that leaves a negative impact on the environment [1–3]. With the increasing demand of energy, the resources of fossil fuel deplete rapidly leading to search of alternative renewable and sustainable energy. Development of biodiesel as an alternative fuel has been receiving international attention as the global production of biodiesel reached over 22.5 billion litres in 2012 [4]. Biodiesel is a fuel containing mono-alkyl ester of vegetable oils or animal fats which have similar physicochemical and fuel properties as petroleum based diesel fuels [5,6]. Biodiesel is advantageous due to its biodegradability, biorenewability and environmental friendly nature [7–9].

Biodiesel or also known as fatty acid methyl ester (FAME) is produced via transesterification of vegetable oils or animal fats in presence of short chain alcohol, catalyzed by either acids or bases. It consist of three consecutive and reversible reactions where the triglyceride are converted to diglyceride first, next to monoglycer-

ide and finally to glycerin [10,11]. Animal fats such as tallow, white grease or lard can be used to produce biodiesel economically, but the limited amount of animal fats available will never be able to meet the world's fuel needs. Moreover, at room temperature, animal fats become solid wax making the production process of biodiesel harder so it is unfavourable compared to vegetable oil [12,13]. Edible vegetable oils such as palm oil, rapeseed oil, sunflower oil and soybean oil are some of the major sources for biodiesel production [14,15]. Unfortunately, using edible oil as source for biodiesel production influence global imbalance to market demand and food supply which in turn increases the oil and biodiesel price [16,17]. To overcome this, a lower cost and non-edible oil such as *Jatropha curcas* is used. *Jatropha curcas* belongs to Euphorbiaceae family is widely known in Malaysia as “jarak pagar”. This multipurpose plant can grow in a very harsh condition without irrigation in a very dry land. Since *Jatropha* have oil content ranging from 30% to 40% of oil, it is viable for biodiesel production and it is possible to get very high fatty acid methyl ester (FAME) conversion up to 97% [18,19].

Homogeneous catalyst such as NaOH, KOH, H_2SO_4 and H_3PO_4 are typically used for transesterification [20–22]. However, the usage of homogeneous catalyst have a few disadvantages where it needs higher production cost since the product needs washing and purification [23]. Furthermore, homogeneous catalyst also causes equipment corrosion. On the other hand, the use of heterogeneous catalyst can decrease the problem associated with the homogeneous catalyst because heterogeneous catalyst can be

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separated from the liquid products easily and can be designed to give higher activity, selectivity and longer catalyst lifetimes [24]. One of the most important properties of heterogeneous catalyst is its reusability which makes it environmental friendly.

For oil containing high amount of free fatty acid (FFA) such as *Jatropha* oil, an acid and basic-catalyzed two-step method was used to produce biodiesel [25,26]. However, this method requires several reactions, washing and separation stages. To overcome this downside, a single step process of simultaneous esterification and transesterification needed with the use of catalyst containing both acid and basic sites [27,28]. La_2O_3 had been used in biodiesel production since La_2O_3 possess both acid and basic site which favours simultaneous esterification and transesterification. La_2O_3 was mostly used as a support to enhance the activity of catalyst such as MgO , ZnO , CaO , Al_2O_3 , ZrO_2 and activated carbon [29–31].

A number of studies have been reported on the production of biodiesel using La_2O_3 catalysts. Sun et al. [32] reported by using 5 wt.% La_2O_3 catalyst amount, methanol to oil molar ratio of 30:1 at reaction temperature of 200 °C for 5 h, the FAME yield was 56%. Some studies were done using La_2O_3 as support for transesterification. Sun et al. [33] used rapeseed oil with $\text{La}_2\text{O}_3/\gamma\text{-Al}_2\text{O}_3$ catalyst with reaction conditions of 2 h, methanol/oil molar ratio of 50:1, 10 wt.% catalyst loading at 200 °C and the yield obtained was 46%. Yan et al. [34] used zinc loaded on lanthanum for reaction with waste cooking oil and obtained 96% FAME using 36:1 methanol/oil molar ratio for 3 h with 3 wt.% catalyst loading at 200 °C. The leaching activity that causes the catalyst deactivation was also reported. Endalew et al. [35] researched $\text{La}_2\text{O}_3\text{-ZnO}$ catalyst for transesterification and reported that the yield of FAME was 30% with reaction conditions of 5 wt.% catalyst loading, 6:1 methanol to oil molar ratio, at temperature of 60 °C for 3 h. Most of the studies reported previously requires high temperature (200 °C), catalyst amount (5–10 wt.%) and methanol/oil molar ratio (20:1 to 50:1). More importantly, leaching is also found to be one of the hurdles which reduce the stability of the catalyst [35].

The purpose of this study was to develop a new type of bi-functional $\text{Bi}_2\text{O}_3\text{-La}_2\text{O}_3$ catalyst for simultaneous esterification and transesterification reaction. The optimum reaction parameters such as reaction temperature, catalyst amount and methanol/oil molar ratio for transesterification was analyzed to determine the stability of $\text{Bi}_2\text{O}_3\text{-La}_2\text{O}_3$ catalyst. The catalysts were synthesized via impregnation method. The characterization of the catalyst were appraised by X-ray diffraction (XRD), N_2 adsorption (BET), temperature programmed desorption of CO_2 (TPD- CO_2) and temperature programmed desorption of NH_3 (TPD- NH_3), respectively.

2. Experimental

2.1. Materials

Jatropha curcas L. oil was purchased from Bionas Sdn Bhd, Malaysia. This oil was used for the reaction without further treatment and purification. La_2O_3 (Sigma–Aldrich), Bi_2NO_3 (Sigma–Aldrich), commercial anhydrous methanol (Merck, 99.7%) were purchased from Fisher Scientific. Analytical reagent grades were applied throughout the experimental.

2.2. Catalyst preparation

The catalysts were prepared by impregnation method followed by calcination of the precursor [28]. In a typical method of catalyst preparation, La_2O_3 powder (Sigma–Aldrich) were impregnated with appropriate amount of Bi_2NO_3 solutions (Sigma–Aldrich) under vigorous stirring at room temperature for about 3 h followed

by drying 100 °C for 12 h. The resultant precursor was then calcined at 650 °C for 5 h. The process was repeated to obtain desired Bi_2O_3 loading (1, 2, 3, 5, 7 wt.%) and denoted as 1BiLa, 2BiLa, 3BiLa, 5BiLa, and 7BiLa respectively.

2.3. Catalyst characterization

The powder X-ray diffraction analysis carried out using Shimadzu diffractometer model XRD6000. $\text{Cu K}\alpha$ radiation was used to generate the crystalline sample's diffraction at room temperature. The $\text{Cu K}\alpha$ radiation was generated by Philips glass diffraction X-ray tube (broad focus 2.7 kW type). Samples were mounted on a sample holder and the basal spacing was determined via powder technique using 2θ range of 20–90° and scanning speed of 2.000 (deg/min).

Brunauer–Emmett–Teller (BET) surface area was measured using nitrogen adsorption at –96 °C. Approximately 0.5 g of catalyst was degassed before heated at 150 °C overnight by vacuum of inert gas purging. Then the sample placed in vacuum chamber for analysis. The analysis was conducted using Thermo Finnigan Sorptomatic 1900 series nitrogen adsorption/desorption analyzer.

Catalysts basicity and acidity study were performed using Thermo Finnigan TPDRO 1100 apparatus equipped with thermal conductivity detector. Catalysts basicity was studied with temperature programmed desorption using CO_2 as probe molecule (TPD- CO_2). Samples were pretreated at 250 °C for 30 min under He stream. Then the temperature decreased to 50 °C and pure CO_2 were adsorbed for 1 h before flushed with helium for 20 min. The CO_2 desorption was carried out between 50 to 850 °C under helium flow (10 °C/min, 30 mL/min). Catalysts acidity was studied with temperature programmed desorption using NH_3 as probe molecule (TPD- NH_3). Samples were pretreated at 250 °C for 30 min under nitrogen stream. Then ammonia adsorption was performed for 2 h at room temperature before samples were flushed with helium for 30 min to remove excess ammonia. The NH_3 desorption was carried out between 50 to 800 °C under He flow (15 °C/min, 20 mL/min).

The concentration of the elements in the catalyst was determined using inductively-coupled plasma-atomic emission spectrometer (ICP-AES) analysis. The sample was prepared by dissolving 0.025 g sample in 8 M nitric acid before diluted to give nominal concentration of 5–50 ppm. The analysis was done using Perkin–Elmer Emission Spectrometer Model Plasma 1000.

2.4. Catalytic activity

Catalytic activity evaluated using transesterification of crude JCO (14% FFA content, molecular weight of 892.19) with methanol. Transesterification was carried out in BERGHOF high pressure autoclave reactor with Nitrogen flow and continuous stirring at 400 rpm (Fig. 1). The mixed metal oxide catalysts (varied from 1 to 5 wt.%), 20 g JCO and methanol (molar ratio from 5:1 to 40:1) was mixed and stirred in the autoclave at required time and temperature. When reaction completed, the catalysts were separated by centrifugation before heating the mixture to remove excess methanol. Finally, the reaction mixture containing glycerol and FAME was separated. The conversions of oil were calculated by the recovered glycerol using the following equations [36]:

$$\text{Conversion (\%)} = \frac{\text{mole of experimental glycerol}}{\text{mole of theoretical glycerol}} \times 100\% \quad (1)$$

The experiments were repeated for twice and the conversions variations were estimated.

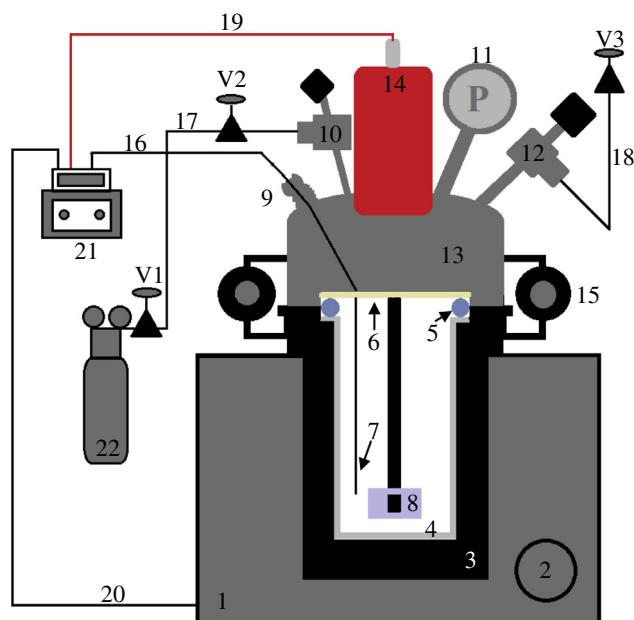


Fig. 1. Schematic diagram of the reaction setup. 1-Heater, 2-safety temperature controller, 3-reactor vessel, 4-PTFE insert, 5-PTFE O-ring, 6-PTFE lid lining, 7-K-type thermocouple, 8-TPFE covered stirrer, 9-temperature regulation, 10-standard fittings (N₂), 11-pressure measurement, 12-special fittings, 13-reactor lid, 14-closure, 16-thermocouple connector line, 17-stainless steel line (input), 18-stainless steel line (output), 19-stirring connector line, 20-temperature controller line, 21-controller, 22-inert gas (N₂), V1~V3-stop valves.

3. Results and discussions

3.1. Catalyst characterization

The XRD patterns for Bi₂O₃–La₂O₃ mixed metal oxides with different Bi₂O₃ loading on La₂O₃ support were shown in Fig. 2. La₂O₃ support occurred as intensified peak at 2θ of 26.2°, 27.8°, 30.0°, 46.2°, 52.1° and 55.4° (JCPDS File No. 65-3185). These peaks show the presence of cubic La₂O₃. A tetragonal Bi₂O₃ peak was found at 2θ of 48.6° (JCPDS File No. 27-0051). The XRD pattern also indicates presence of separate metal oxide crystalline phases with no formation of other new metal oxide phases in the catalysts [37]. When

the loading of Bi₂O₃ was increased from 3% to 7%, Bi₂O₃ peaks can be observed clearly. As the content Bi³⁺ increase, the intensity of La₂O₃ decreases, suggesting that the dispersion of Bi₂O₃ was saturated and covers the La₂O₃ phase of the catalyst [38]. The shift of the diffraction angle of La₂O₃ peak of 5BiLa, and 7BiLa may be due to the doping of Bi₂O₃ which change the lattice parameters of La₂O₃, resulted the XRD peak shifts in the opposite direction. So, if the dopant leads to an increase in lattice constant then the diffraction angle 2θ decrease.

The specific surface area of the catalyst is shown in Table 1. Unloaded La₂O₃ catalyst has smallest surface area of 4 m² g^{−1}. When Bi₂O₃ loaded on La₂O₃, it was well dispersed onto the surface of the support, increasing the surface area. The surface area of 1BiLa, 2BiLa, 3BiLa, 5BiLa, and 7BiLa are 26, 29, 31, 34 and 35 m² g^{−1}, respectively. The surface area of prepared catalyst were found to be higher compared to previous study using Zn₉La₁, Zn₃La₁ and CaO/La₂O₃ catalyst which was 16.8, 15.7 8.5, m² g^{−1} respectively [31,39]. This shows that addition of Bi₂O₃ content increases and enhances the surface area.

The peak in the high and low temperature zone can be attributed to desorption of NH₃ and CO₂ from the strong and weak acid/basic sites, respectively. The peak in the high temperature region above 700 °C can be due to the desorption of NH₃ or CO₂ from strong Brønsted and Lewis type acid/basic site and peaks below 360 °C may be attributed to the desorption of NH₃ or CO₂ from weak acid/basic sites. Basic strength of the catalysts was measured by TPD–CO₂ as the profiles are shown in Fig. 3. 1BiLa and 2BiLa catalysts shows a maximum CO₂ desorption peak at 820 °C in the presence of very strong basic strength which also indicating the interaction of CO₂ with catalyst. When loading of Bi₂O₃ increased from 3 to 7 wt.%, the CO₂ desorption peaks appeared to be broad with two peaks at temperature 720 and 780 °C corresponding to a strong basic strength in the catalysts. 5BiLa and 7BiLa have a small peak at 290 °C corresponding to a low basic strength [40]. When Bi₂O₃ were loaded, it dispersed on the surface of the catalyst hindering the adsorption of CO₂ onto the active sites of La₂O₃. Hence, when the loading of Bi₂O₃ increases, the basic strength in the catalyst reduces. Fig. 4 shows the TPD–NH₃ profile for the catalysts. 1BiLa and 2BiLa catalyst gave small desorption peak in the range of 200 to 360 °C which indicates a weak acid strength. 3BiLa, 5BiLa, and 7BiLa catalysts showed a narrow peak at higher range of 700–820 °C which attributes to presence of strong acid strength. 5BiLa also shows a shoulder peak at 700 °C corresponding to a

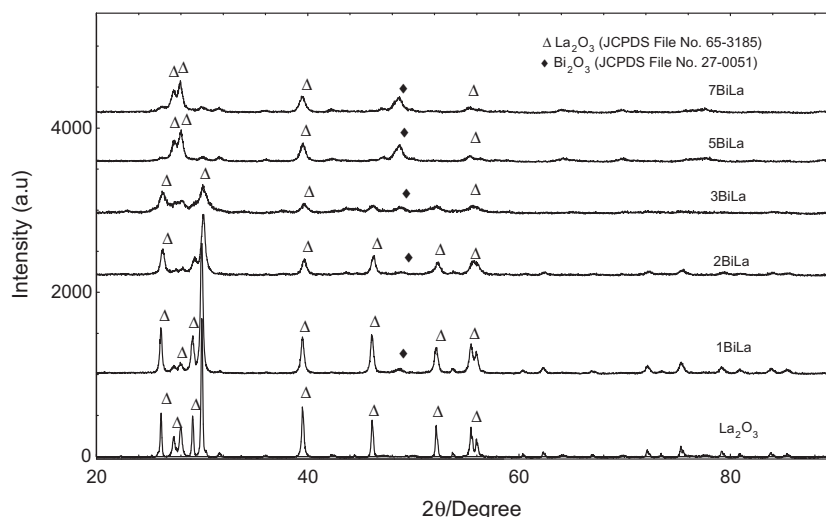
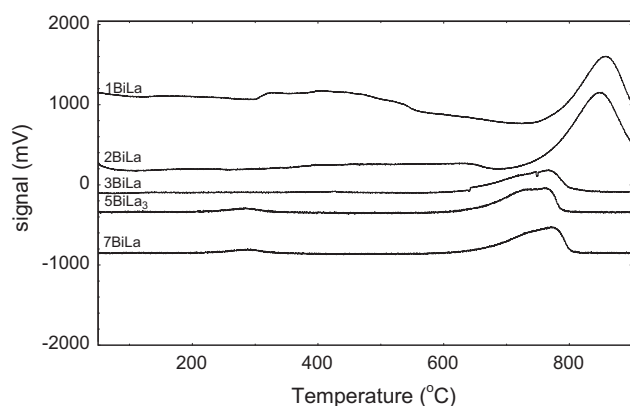
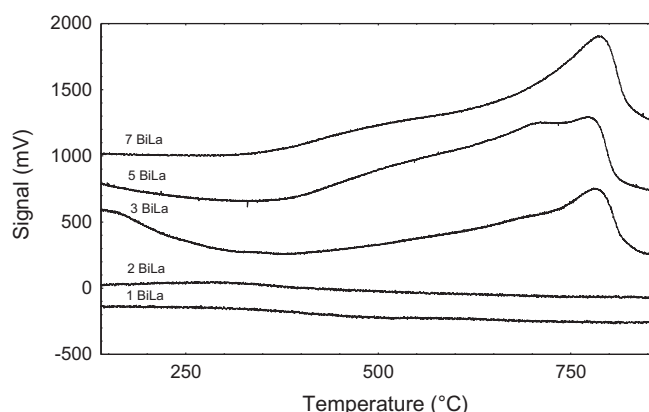


Fig. 2. XRD patterns of La₂O₃, 1BiLa, 2BiLa, 3BiLa, 5BiLa and 7BiLa catalysts calcined at 650 °C (5 °C/min) for 5 h: (1BiLa) 1 wt.% of Bi₂O₃/La₂O₃; (2BiLa) 2 wt.% of Bi₂O₃/La₂O₃; (3BiLa) 3 wt.% of Bi₂O₃/La₂O₃; (5BiLa) 5 wt.% of Bi₂O₃/La₂O₃ and (7BiLa) 7 wt.% of Bi₂O₃/La₂O₃. (Δ) La₂O₃, (◆) Bi₂O₃.

Table 1

BET surface area of La_2O_3 , 1BiLa, 2BiLa, 3BiLa, 5BiLa and 7BiLa catalysts.

Catalyst	Surface area ($\text{m}^2 \text{g}^{-1}$)
La_2O_3	4
1BiLa	26
2BiLa	29
3BiLa	31
5BiLa	34
7BiLa	35

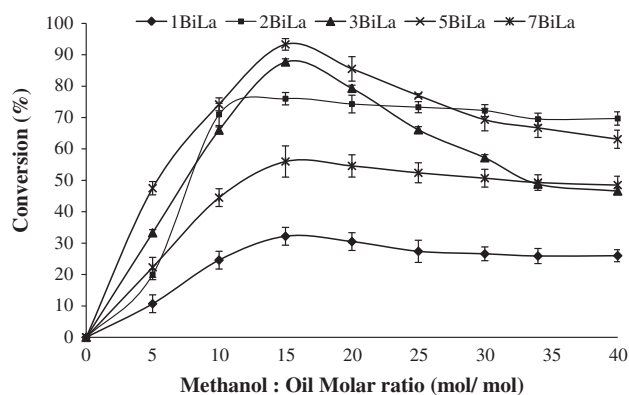
**Fig. 3.** TPD- CO_2 of $\text{Bi}_2\text{O}_3/\text{La}_2\text{O}_3$ mixed metal oxide catalysts.**Fig. 4.** TPD- NH_3 of $\text{Bi}_2\text{O}_3/\text{La}_2\text{O}_3$ mixed metal oxide catalysts.

strong acid strength. The results show that the addition of Bi_2O_3 enhanced the acid strength of the catalysts. Increasing the bismuth loading changes the intensity of XRD peaks where the intensity for La_2O_3 decreases. The basicity of the catalysts were in agreement with the intensity of the peak in which decrease of intensity La_2O_3 peak enhance of total amount of CO_2 absorb [6].

3.2. Catalytic activity

3.2.1. Effect of methanol/oil molar ratio

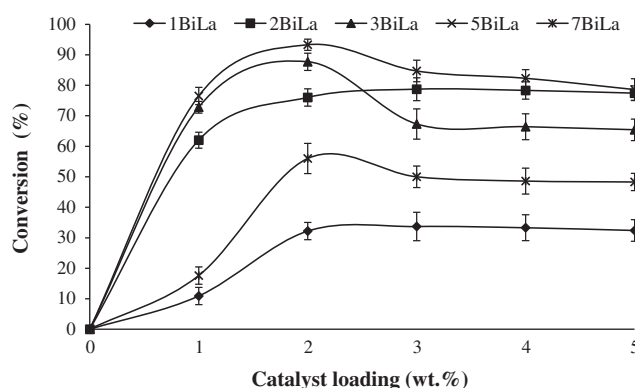
Stoichiometrically, methanol/oil molar ratio required for a complete transesterification is 3:1. Since transesterification is a reversible reaction, methanol is needed in excess to drive the reaction forwards ensuring the triglyceride are completely converted to esters. The effect of methanol/oil molar ratio on the FAME conversion was studied at various methanol/oil molar ratios from 5:1 to 40:1 as shown in Fig. 5. 1BiLa, 2BiLa, 3BiLa, 5BiLa and 7BiLa catalysts show increasing conversion of FAME as the methanol/oil molar

**Fig. 5.** Effect of methanol/oil molar ratio on FAME conversion. Reaction conditions: reaction temperature = 150°C , catalyst amount = 2 wt.%, reaction time = 4 h.

ratio increases. The maximum conversion i.e. 93% was obtained at 15:1 methanol/oil molar ratio using 5BiLa catalyst. Further addition of methanol beyond 15:1 molar ratio did not increase the conversion. Excess methanol decreases the conversion. This was due to the polar hydroxyl group in methanol acting as emulsifier making it more difficult to separate the FAME and glycerol [41]. Previously, Babu et al. [42] used ML-3 catalyst with higher methanol/oil molar ratio (42:1) compared to 5BiLa (15:1) to achieve the same conversion.

3.2.2. Effect of catalyst amount

The effect of catalyst amount on conversion of FAME was studied by varying the catalyst amount in the range of 0–5 wt.% as shown in Fig. 6. There was no conversion obtained for reaction conducted without catalyst. An adequate amount of catalyst needed to provide active sites for the reaction to convert triglyceride to FAME completely. When the catalyst amount increased, the conversion of FAME also increases. Addition of catalyst increases the reaction rate for transesterification due to which higher conversion of FAME was obtained. 1BiLa and 2BiLa catalysts gives maximum conversion when the catalyst amount is 3 wt.%. 3BiLa, 5BiLa, and 7BiLa catalyst gives highest FAME conversion when the catalyst amount is 2 wt.%. The highest conversion obtained using 2 wt.% of 5BiLa catalyst because 5BiLa possess more active sites for esterification and transesterification reactions. However, further increase in catalyst amount beyond its optimum value decreases the FAME conversion due to catalyst, reactant and product which are resisted to mix [6,43]. From the TPD profiles from Figs. 3 and 4, it was suggested that the catalyst possess both acid and basic sites which is

**Fig. 6.** Effect of catalyst amount on FAME conversion. Reaction conditions: methanol/oil molar ratio = 15:1, reaction temperature = 150°C , reaction time = 4 h.

active for esterification and transesterification reactions. Basic sites in the catalyst favours transesterification while acid sites favours esterification [44,45]. In this study, the JCO used contains high FFA amount hence more esterification reaction needs to take place compared to transesterification. 3BiLa and 5BiLa possess strong basic strength which favours transesterification and strong acid strength favouring esterification reaction hence giving high FAME conversion. Catalyst with larger surface such as 5BiLa gives higher FAME conversion compared to 1BiLa, 2BiLa and 3BiLa. This is due to high surface area provides more surface for the reaction to take place. For catalyst 7BiLa, FAME conversion decreases probably due to the increasing Bi^{3+} content on the active sites of La_2O_3 .

3.2.3. Effect of reaction temperature

The effect of reaction temperature on FAME conversion was shown in Fig. 7. The temperature studied varied from 100 to 170 °C. Transesterification is an endothermic reaction, so the reaction needs higher temperature to react [46]. Insufficient temperature result to incomplete conversion of triglyceride giving low FAME conversion. From Fig. 6, the conversion starts to occur beyond 120 °C. As the temperature of the reaction increases, the FAME conversion also increases. For 1BiLa catalyst, the highest conversion was at temperature 160 °C while for 2BiLa, 3BiLa, 5BiLa and 7BiLa catalyst, highest conversion was at temperature 150 °C. When the reaction temperature increases it will cause the kinetic energy in the molecules increase, making the reaction rate becomes faster. This gives more FAME conversion in a shorter time. Increase in reaction temperature higher than optimum decreases the FAME conversion. Few studies suggested that saponification of triglyceride will proceed beyond optimal temperature, thus decreasing the conversion [47,48].

3.2.4. Effect of reaction time

Reaction temperature is also one of the factors effecting the FAME conversion. In this study, the reaction time was varied from 2 h to 6 h and the conversion is illustrated in Fig. 8. At the starting 3 h reaction, the reactions are slow giving low FAME conversion. At this time, the methanol is starting to mix and disperse into the oil. As the reaction time increased more than 3 h, the conversion of FAME increased since the reaction happens faster. The highest FAME conversion for all the catalysts achieved at 4 h reaction time. Since transesterification is a reversible reaction, prolonging the reaction time more than 4 h causes the reaction to move backwards, decreasing the conversion.

3.2.5. Catalyst reusability

Reusability of a catalyst is essential properties for a heterogeneous catalyst. Ngamcharussrivichai et al. [49] indicated that leaching of catalyst occurs due to the dissolution of active metal

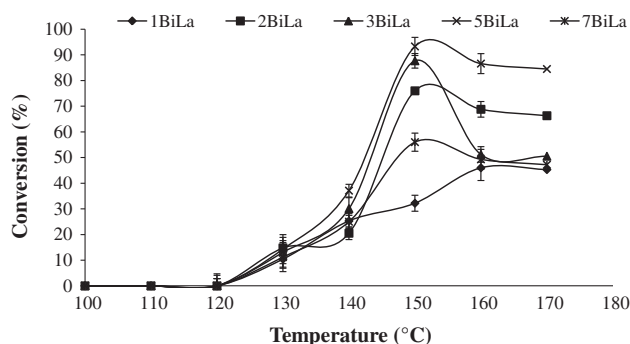


Fig. 7. Effect of reaction temperature on FAME conversion. Reaction conditions: methanol/oil molar ratio = 15:1, catalyst amount = 2 wt.%, reaction time = 4 h.

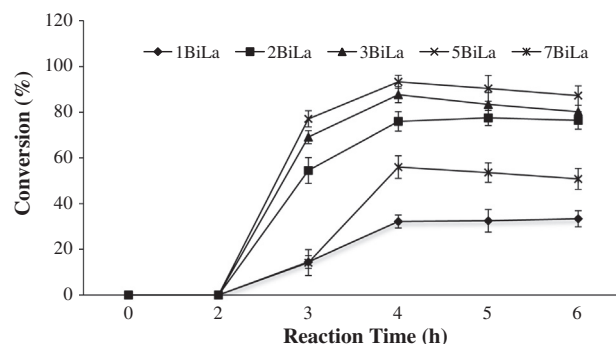


Fig. 8. Effect of reaction time on FAME conversion. Reaction conditions: methanol/oil molar ratio = 15:1, catalyst amount = 2 wt.%, reaction temperature = 150 °C.

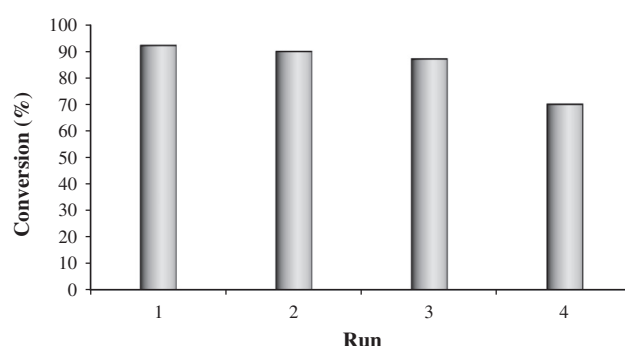


Fig. 9. Catalyst reusability of 5BiLa catalyst. Reaction conditions: methanol/oil molar ratio = 15:1, catalyst amount = 2 wt.%, reaction temperature = 150 °C, reaction time = 4 h.

Table 2

Concentration of element of fresh and used 5BiLa catalysts.

Concentration of element (ppm)	Bi	La
Fresh catalyst	3.871 ± 0.14	47.51 ± 0.29
Used catalyst (3rd run)	3.769 ± 0.19	47.48 ± 0.13

and this can minimize transesterification activity of a catalyst. The purpose of preparing this catalyst was to stabilize the active phase of the catalyst to avoid leaching. For this study, 5BiLa was used with the optimum reaction conditions. The used catalyst was washed with hexane and methanol to remove any unreacted oil, biodiesel and glycerol before dried in oven at 100 °C for 2 h. The catalyst was regenerated by calcining at 600 °C for 1 h to. The reusability result shown in Fig. 9. The conversion of FAME decreases as the catalyst was reused for each run. The data suggested that the catalyst can be reused for three runs with the conversion around 87%. The decrease in conversion after each runs suggesting the active sites of the catalyst might be enclosed by unreacted oil, biodiesel or glycerol [50]. In order to study the leaching of metals in the catalysts, ICP-AES analysis was done using fresh $5\text{Bi}_2\text{O}_3\text{-La}_2\text{O}_3$ and used $5\text{Bi}_2\text{O}_3\text{-La}_2\text{O}_3$. The result was depicted in Table 2. The result shows the concentration of Bi and La decreases a bit after 3rd cycle of reaction with not significant affect the FAME conversion.

4. Conclusions

In this study, a bi-functional catalyst, Bi_2O_3 supported on La_2O_3 catalysts was synthesized using wet impregnation method. 5BiLa gives the highest FAME conversion of 93%, at optimum reaction

conditions (methanol to oil molar ratio 15:1, catalyst amount 2 wt.%, reaction temperature 150 °C for 4 h). The results showed that the high catalytic activity of 5BiLa catalyst can be associated to the well dispersion of Bi₂O₃ on La₂O₃ support which increases the surface area of the catalyst. The catalyst also possesses strong acid basic strengths that favours simultaneous esterification and transesterification reactions. Furthermore, 5BiLa catalyst maintained almost 87% conversion after three successive run.

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