Abstract
Background: Transesterification catalyzed by solid base catalyst is a brilliant technology for the noble process featuring the fast reaction under mild reacting condition in biodiesel production. Heterogeneous base catalysts are generally more reactive than solid acid catalysts which require extreme operating condition for high conversion and biodiesel yield. In the present study, synthesis of biodiesel was studied by using edible (palm) or non-edible (Jatropha) feedstock catalyzed by heterogeneous base catalysts such as supported alkali metal (NaOH/Al₂O₃), alkaline-earth metal oxide (MgO, CaO and SrO) and mixed metal oxides catalysts (CaMgO and CaZnO).

Results: The chemical characteristic, textural properties, basicity profile and leaching test of synthesized catalysts were studied by using X-ray diffraction, BET measurement, TPD-CO₂ and ICP-AES analysis, respectively. Transesterification activity of solid base catalysts showed that > 90% of palm biodiesel and > 80% of Jatropha biodiesel yield under 3 wt.% of catalyst, 3 h reaction time, methanol to oil ratio of 15:1 under 65°C. This indicated that other than physicochemical characteristic of catalysts; different types of natural oil greatly influence the catalytic reaction due to the presence of free fatty acids.

Conclusions: Among the solid base catalysts, calcium based mixed metal oxides catalysts with binary metal system (CaMgO and CaZnO) showed capability to maintain the transesterification activity for 3 continuous runs at ~ 80% yield. These catalysts render high durability characteristic in transesterification with low active metal leaching for several cycles.

Keywords: Transesterification, Palm oil, Jatropha oil, Solid base catalyst, Alkaline-earth metal oxide, Mixed metal oxides

Background
Industrialization processes continue to grow globally in par with human population which leads to the growing worldwide demand for energy as well as for petrochemical resources, coal and natural gases. This phenomenon has caused the depletion rate of fossil energy resources to increase exponentially and caused alarming environmental problems to the society. Recently, many fuel developers have showed interests in alternative renewable fuels to substitute or blend with petroleum-based fuels. An alternative fuel shall be easily available, environment friendly and techno-economically competitive [1,2].

Biodiesel plays a major role in energy sector due to its similar combustion properties with petroleum. Furthermore, biodiesel is sometimes more superior than petroleum diesel with improved physical and chemical properties, such as higher flash point, higher cetane number, ultralow sulfur content, better lubricity, improved biodegradability, and smaller carbon footprint [3-5]. Chemically, biodiesel is a mixture of methyl esters with long-chain fatty acids and is typically made from transesterification reaction of biological triglyceride sources such as vegetable oil and animal fats with alcohol in the presence of catalyst. This process reduces the viscosity to a value comparable to that of diesel and hence improves combustion [6].

According to Meng et al. (2009), biological feedstock supply for biodiesel production covers more than 75% of the overall production cost [7]. The favorable properties in selecting the best biodiesel feedstock include lowest oil price, high oil content, favorable fatty acid composition (saturated or unsaturated acid), low cultivation maintenance and costs, controllable growth and harvesting season, consistent seeds maturity rates and potential market for agricultural by-products [8].
In general, biodiesel feedstock can be divided into 4 main categories which are: (a) edible vegetable oil, (b) non-edible vegetable oil, (c) waste or recycled oil and (d) animal fats [9]. The most common feedstock employed in biodiesel production is edible and inedible oil from oleaginous plants grown in different regions. Soybean oil, sunflower oil, rapeseed oil and palm oil have been used as edible feedstock in biodiesel synthesis [10]. In Malaysia, biodiesel production is synonymous to palm oil as oil palm plantations possesses higher productivity per hectare of oil palm with lowest oil production cost per unit as compared to other vegetable oils like rapeseed and soybean [11,12]. A hectare of oil-palm plantation produces approximately 3.62 tonnes/ha/year of oil, 5–9 times higher than other oil producing crops like soybean, sunflower and rapeseed, which produces 0.4, 0.46 and 0.68 tonnes/ha/year, respectively [13]. This keeps the price of palm-based biodiesel competitive enough to meet the demand of commodity market [9,14].

Instead of edible palm oil, non-edible feedstock is getting interest as a biodiesel feedstock in biodiesel production. Amongst the varieties of non-edible plant oil, Jatropha is the most favorable for biodiesel production as they meet the demand of commodity market [9,14]. The formation of soap was observed in base catalyzed transesterification of high acid oil with low biodiesel yield.

By drawing on this, an attempt has been made in the present work to produce biodiesel from edible (palm) and non-edible (Jatropha) oils using heterogeneous base catalysts: (a) supported alkali metal catalysts– sodium hydroxide supported with alumina (NaOH/Al2O3), (b) alkaline-earth metal oxides– magnesium oxide (MgO), calcium oxide (CaO) and strontium oxide (SrO) and (c) calcium-based mixed metal oxides– (CaMgO and CaZnO). This study was aimed to investigate the versatility of solid base catalysts with different chemical characteristic for transesterification of edible and non-edible biodiesel feedstock. The physicochemical properties (chemical composition, textural properties and basicity) of synthesized solid base catalysts produced thru wet impregnation and co-precipitation techniques were investigated. Furthermore, effects of the catalyst loading and reaction time towards catalyst activity were investigated in order to optimize transesterification conditions. The reusability of the solid catalysts was evaluated by batch experiment and the reasons for the deactivation of the catalyst were also discussed by performed the catalyst leaching test.

Results and discussion

Catalysts characterization

The crystalline structure of alumina supported alkali metal (NaOH/Al2O3), alkaline-earth metal oxide (CaO, MgO, SrO) and calcium-based mixed metal oxides (CaMgO and CaZnO) base solid catalysts was revealed by X-ray diffraction pattern (Figure 1). The diffraction peaks of NaOH/Al2O3 showed the presence of NaOH (ICPDS File No. 27-0711) and sodium aluminate (NaAlO2) phases (ICPDS File No. 20-1073). The intensity of Al2O3 peaks were reduced when NaOH was introduced into Al2O3 support, this is due to well dispersion of NaOH on the Al2O3 support in the form of monolayer covering the surface of support. The results indicate that NaOH has reacted with Al2O3 thus giving rise to the formation of aluminates and is in good agreement with that reported by Arzamendi J. and Kim’s research groups [28,29]. The diffractogram of MgO clearly showed that the presence of oxide phases at 2θ of 42.9 and 62.2° (ICPDS File No. 4-0829). For CaO, the diffraction pattern depicted intensified peaks at 2θ angles of 32.2, 37.3 and 53.8° (ICPDS File No. 37-1497). SrO catalyst showed main reflection peaks at 2θ = 30.31, 50.54° and 63.04° (ICPDS File No. 1-074-1227). And the other peaks observed indicated the presence of SrCO3 and SrO2H2O phases (ICPDS File No. 1-084-1778 and 28-1222). Strontium compound is difficult to regenerate
by thermal treatment, and their decomposition temperatures are higher than 1200°C [30]. Comparing to MgO and CaO, SrO can be easily absorbed by CO₂ and hydrolyzed by moisture to form carbonate and hydroxide phase, respectively which compatible with the XRD result. The XRD patterns of calcium-based mixed metal oxides catalysts recorded the patterns of both samples (CaMgO and CaZnO) were corresponded to pure oxide, and no new crystalline phase attributable to the formation of mixed oxides could be detected. CaMgO catalyst showed the appearance of cubic CaO and hexagonal MgO phases where CaZnO catalyst also gave diffraction peaks of CaO and wurzite structure of ZnO phase at 2θ angles of 31.7°, 34.4° and 36.2° (JCPDS File No. 36-1451).

The textural properties of the solid base catalysts with various calcination temperatures were determined by BET measurement (Table 1). Results showed that BET surface area of pure γ-Al₂O₃ is 136 m²/g. However, the surface area of the NaOH/γ-Al₂O₃ catalyst was decreased to 52 m²/g after NaOH impregnation and thermal activation. This reflects a significant reduction of pore volume of Al₂O₃ penetrated by Na⁺ ion as shown in Table 1 [28]. The surface area and porosity of alkaline-earth metal oxide: MgO, CaO and SrO catalysts vary with different thermal activation temperature of 600, 800 and 1200°C, respectively. Calcination at high temperature is required to thermally activate the metal carbonate salts to active metal oxide catalyst for transesterification reaction, especially for SrO catalyst which > 1000°C of heating is required to remove the strong

Table 1 Surface area profile of synthesized base catalysts

| Synthesized catalyst                  | Calcination temperature (°C) | BET area (m²/g) | Pore volume (cm³/g) | Pore diameter (Å) |
|--------------------------------------|------------------------------|-----------------|---------------------|-------------------|
| Alkali supported alumina             |                              |                 |                     |                   |
| Al₂O₃                                | -                            | 136             | 0.436               | 132.6             |
| NaOH/Al₂O₃                           | 500                          | 52              | 0.235               | 139.1             |
| Alkaline-earth metal oxide           |                              |                 |                     |                   |
| MgO                                  | 600                          | 29.7            | 0.132               | 160.5             |
| CaO                                  | 800                          | 9.5             | 0.072               | 213.6             |
| SrO                                  | 1200                         | 1.0             | 0.005               | 216.0             |
| Calcium-based mixed metal oxides     |                              |                 |                     |                   |
| CaMgO                                | 800                          | 15.5            | 0.103               | 225.3             |
| CaZnO                                | 800                          | 9.8             | 0.098               | 220.4             |

Figure 1 XRD diffractogram of synthesized solid base catalysts.
bonding of carbonate from the catalyst’s surface. The surface area and pore volume for MgO, CaO and SrO were decreased as the calcination temperature increased, whereby the pore diameter were increased with temperature as the pore structure collapsed with high temperature. For calcium-based mixed oxides catalysts: CaMgO and CaZnO, the textural properties are apparently small when synthesized using coprecipitation method. The precursor of binary metal-based catalyst in carbonate form required high temperature (800°C) to render pure active mixed oxide phase for reaction which led to sintering effect of catalysts.

Several studies have reported that basicity of catalyst is the major key to improve the transesterification activity. Among basicity distribution strength (weak, medium and strong basic strength), the number of strong basic sites that are considered to be the main parameter in yielding a high biodiesel yield [31]. The total basicity and basic site distributions of synthesized solid base catalysts were measured by temperature programmed desorption of CO₂ (Table 2 and Figure 2). Figure 2 showed the different desorption peaks of catalysts, which indicated the presence of basic sites with different basic strengths. The CO₂ desorption band at 100–500°C are assigned as interaction of CO₂ with sites of weak and medium basic strengths. Whereas, the CO₂ desorbed at temperature of ~ 600°C can be attributed to the much stronger basic site corresponding to unbounded O²⁻ anions [32,33]. The results showed that calcium-based mixed metal oxides (CaMgO and CaZnO) and alkali supported alumina (NaOH/Al₂O₃) possess similar number of basic sites (~400 μ mol of CO₂/g). This indicated that catalyst with multi-metal ion interaction showed synergy effect by enhancing the basicity on active site of catalyst [34].

Transesterification of edible palm and non-edible Jatropha oils

The transesterification of edible (palm) and non-edible (Jatropha) oils in the presence of different solid base catalysts (NaOH/Al₂O₃, MgO, CaO, SrO, CaMgO and CaZnO) were studied thoroughly by varying the transesterification conditions. The transesterification reaction was performed at different reaction time (2–6 h) and catalyst amount (1–5 wt.%) under constant reflux temperature (65°C) and methanol to oil ratio of 15:1. The catalyst amount is a crucial factor to improve the rate of transesterification reaction. As reported by most of the studies, the increase of catalyst loading shall increase surface active sites of catalyst. This indirectly improves transesterification process by increase the accessibility of triglyceride and methanol to the catalyst surface. Other than that, reaction time for transesterification is another

Table 2 TPD-CO₂ profile of synthesized base catalysts

| Catalyst            | Amount of basic site (μ mol of CO₂/g) | Total basicity (μ mol of CO₂/g) |
|---------------------|--------------------------------------|---------------------------------|
|                     | T<sub>max</sub> (<300°C) | T<sub>max</sub> (300–500°C) | T<sub>max</sub> (>500°C) |
| Alkali supported alumina | - | - | 467.68 |
| NaOH/Al₂O₃        | - | - | 467.68 |
| Alkaline-earth metal oxide | | | |
| MgO                | 31.36 | - | 418.95 |
| CaO                | - | - | 290.42 |
| SrO                | - | - | 396.56 |
| Calcium-based mixed oxide | | | |
| CaMgO              | 2.68 | - | 449.70 |
| CaZnO              | - | - | 412.70 |
important criterion to determine the equilibrium point of reaction in order to avoid any reverse process [6,37]. The biodiesel profile revealed that edible palm oil underwent transesterification relatively at a higher reaction rate compared to the non-edible Jatropha oil. Results showed that the transesterification of palm oil yielded more than 90% of biodiesel under mild condition of 3 wt.% catalyst, 3 h reaction time and 15:1 methanol/oil molar ratio. Jatropha biodiesel production required higher amount of catalyst (4 wt.%) (Figure 3) and longer reaction time (4 h) (Figure 4) in order to improve the biodiesel yield to more than 80% compared to palm biodiesel.

The present study imply that the nature of edible and non-edible oil significantly influence the transesterification activity of catalysts [38]. Although non-edible Jatropha oil has tremendous potential for biodiesel production but it contains high free fatty acids (FFAs) as compared to palm oil. The FFAs content is key parameter for determining the

Figure 2 TPD-CO$_2$ spectra of synthesized solid base catalysts.

Figure 3 Effect of catalyst amount for solid base catalyzed transesterification reaction of palm and Jatropha oil (methanol: oil ratio of 15:1, 65°C and 3 h).
viability of the vegetable oil in transesterification process. Higher FFAs content of the oil will lower the biodiesel selectivity. This happened to NaOH/Al₂O₃ catalyzed transesterification of high acid Jatropha oil. Jatropha biodiesel yield is 58%, while the low acid palm oil achieved conversion of 99% (Figure 5). The presence of FFAs in Jatropha oil triggered saponification with sodium base catalyst resulting soap formation which could lead to formation of gel and emulsion. This is highly undesirable and complicates the purification process of biodiesel [10]. The NaOH/Al₂O₃ was deactivated as the active sodium was loss to formation of soap and in turn reduced transesterification activity. FFAs content directly influences biodiesel yield in biodiesel production.

Other than oil characteristics, the rate of transesterification is greatly depending on catalyst’s basicity. Among the solid base catalysts, the catalytic activity of MgO for both palm and Jatropha-based transesterification are low, which is 52% and 5%, respectively. The results suggested that, under reflux condition, the optimum content of biodiesel synthesis using edible and non-edible oil greatly depend on the type of the solid base catalyst used. According to Di Serio and his co-researcher [33], basicity of catalyst is directly proportional to the biodiesel yield. MgO catalyst with
lower amount of basicity is less active than other catalysts by producing less yield of biodiesel under mild condition. This fact has been proven by the correlation effect between transesterification activities and basicity of different catalysts (Figure 5).

**Durability of catalyst**

Under optimized conditions (reaction temperature of 65°C, 3.0 wt.% of catalyst, 15:1 methanol/oil molar ratio and 3 h reaction time), the solid base catalysts (NaOH/Al2O3, CaO, SrO, CaMgO and CaZnO) were reutilized for another two runs for both edible palm and non-edible Jatropha oil (Figure 6). Among these catalysts, CaMgO and CaZnO demonstrated high reusability. However, MgO catalyst was not selected for the reusability test as it is low in catalytic activity. Surprisingly, only calcium based mixed metal oxides catalysts are capable to maintain high yield > 80% of palm-biodiesel and > 70% of Jatropha-biodiesel at third run. From the results, NaOH/Al2O3 catalyzed reaction drop drastically throughout the three runs. This apparently demonstrated that the catalyst was unstable during reaction in which Na-O-Al was decomposed in the present of methanol in the first run reaction medium. Therefore, the catalytic activities of second and third runs were reduced as the active Na species was reduced. For alkaline-earth metal oxides catalysts, the catalytic activities were reduced gradually in every cycle. The metal oxides are poisoned by absorption of H2O, CO2 and reaction medium (e.g. glycerol, oil, methyl ester). Extra washing step and thermal activation is required prior to each cycle to maintain high reusability. In order to evaluate the leaching of the catalyst, elemental analysis was performed to study the leaching of Ca, Mg and Zn phases for CaMgO and CaZnO catalysts. The result for the fresh and used catalyst was depicted in Table 3. It is showed that the only minor amount of calcium, magnesium and zinc leached out during reaction and thus it is negligible. This result is concordance with the catalytic activity of CaMgO and CaZnO which capable of maintaining its activity after the third cycles.

**Experimental**

**Preparation of solid base catalysts**

**Supported alkaline metal catalysts: NaOH/Al2O3**

The catalyst was prepared by incipient wetness impregnation of γ-Al2O3 powder with an aqueous solution of NaOH compounds. Al2O3 support was impregnated with 15 ml sodium hydroxide (50 wt.%) solution. The impregnate was dried in oven at 100°C overnight and undergone thermal treatment at 500°C.

**Alkaline-earth metal oxide: MgO, CaO and SrO**

MgO, CaO and SrO were obtained after calcination of pulverized magnesium carbonate (MgCO3), lime stone (CaCO3) and strontium carbonate (SrCO3).

**Mixed metal oxide: CaMgO and CaZnO**

The mixed metal oxide catalysts were prepared by using co-precipitation method, and subsequent calcinations of the precursors. These catalysts were obtained by slowly adding a 2 M aqueous solution of the corresponding metal nitrates to an aqueous solution containing...
Na$_2$CO$_3$ and NaOH. The precipitation was performed under vigorous stirring at 65°C, for 1 day. Finally, the solids were filtered, washed with deionized water and dried at 100°C. The synthesized precursors were then undergone thermal treatment (800°C) in the air to produce mixed metal oxides.

Characterization

The crystalline phases of synthesized catalysts were analyzed by powder X-ray diffraction analysis using a Shimadzu diffractometer model XRD 6000 with employing Cu–K$_\alpha$ radiation to generate diffraction patterns from powder crystalline samples at ambient temperature. Specific surface area of the catalysts was obtained by the BET (Brunauer–Emmer–Teller) method using Thermo Finnigan Sorptomatic 1900 series nitrogen adsorption/desorption analyzer. The total basicity and basic strength of the catalysts were measured by temperature programmed desorption of carbon dioxide (CO$_2$-TPD) using a Thermo Finnigan TPDRO 1100 apparatus provided with a thermal conductivity detector. The elemental composition of fresh and used catalysts was determined by using inductively coupled plasma-atomic emission spectrometer (ICP-AES) analysis that conducted by using Perkin Elmer Emission Spectrometer Model Plasma 1000.

Catalytic test

The biodiesel feedstock, palm oil and Jatropha oil were purchased from local market and Bionas Sdn. Bhd., respectively. Physicochemical properties of both palm and Jatropha oil were shown in Table 4. Catalytic activity was evaluated by performing transesterification reaction in a batch reflux-reactor, which palm or Jatropha oil was mixed with methanol and synthesized solid base catalyst. The mixture was added into round-bottom flask equipped with a reflux condenser and heated at 65°C under constant stirring speed at 800 rpm. Investigation on effect of reaction time (2–6 h) and catalyst amount (1–5 wt.%) was investigated in order to achieve optimum biodiesel yield. Upon the completion of reaction, the product was cooled to ambient temperature and the catalyst was separated through centrifugation. The excessive methanol was removed using rotary evaporator. Reaction product was then poured into a separation funnel and left overnight for separation with upper layer as biodiesel and glycerin at the bottom layer. The biodiesel product was analyzed by using a gas chromatography (PerkinElmer Autosystem XL, USA) equipped with a flame ionization detector (FID) and connected to a HP-Innovax capillary column (30 m × 0.25 mm × 0.25 μm; J & W Scientific). The content of methyl ester obtained was calculated according to European regulated procedure EN14103.

Conclusions

Heterogeneous base catalysts (NaOH/Al$_2$O$_3$, MgO, CaO, SrO, CaMgO and CaZnO) were used as catalysts for the production of biodiesel from edible palm and non-edible Jatropha oils. All the base catalysts showed the presence of strong basic strength on the active site except for MgO which contained dominant amount of medium basic sites. The transesterification activity of the heterogeneous base catalysts are correlated with basicity and basic strengths of the catalysts. The optimum conditions for solid base catalysts to achieve >90% of palm biodiesel is 3 wt.% of catalyst, 15:1 of methanol to oil molar ratio within 3 h. Jatropha biodiesel yield required higher amount of catalyst (4 wt.%) and longer reaction time (4 h) under reflux temperature to achieve yield > 80%. It is reasonable to conclude that the type of feedstock oil and chemical characteristic of solid base catalyst strongly affects the yield of biodiesel. Despite of good transesterification activity for solid base catalyst, a good candidate of basic catalyst should be able to tolerate free fatty acids and moisture content in feedstock oil. High catalyst’s re-usability without leaching of active component is another desirable characteristic in good candidate catalyst.

Calcium-based mixed metal oxides catalysts (CaMgO and CaZnO after reaction in Jatropha oil.

### Table 3 Leaching tests profile of CaMgO and CaZnO

| Element content$^a$ (ppm) | CaMgO | CaZnO |
|---------------------------|--------|--------|
| Fresh catalyst            | 17.9 ± 0.5 | 36.2 ± 0.3 | 74.3 ± 0.7 |
| 3rd run $^b$              | 15.6 ± 0.7 | 298 ± 0.2 | 86 ± 0.3 | 67.4 ± 0.6 |

$^a$Ca, Mg and Zn content from catalysts were determined by ICP-AES analysis.

$^b$Leaching experiments were conducted in triplicate to determine the content of the CaMgO and CaZnO after reaction in Jatropha oil.

### Table 4 Physicochemical properties and characteristic of palm oil and Jatropha oil

| Properties (unit) | Palm oil | Jatropha oil |
|-------------------|----------|--------------|
| Specific gravity (g cm$^{-3}$) | 0.860–0.90 | 0.914 |
| Viscosity at 40°C (cSt) | 44.2 | 54.8 |
| Sulphated Ash (% mass) | 0.02 | 0.0012 |
| Flash point (°C) | 182 | 235 |
| Cloud point (°C) | 15 | 2 |
| Pour point | 15 | 2 |
| Cetane Number | - | 46.3 |
| Saponification number (mg g$^{-1}$) | 198.9 | 186.48–193.32 |
| Free Fatty acids% (Kg Kg$^{-1}$ × 100) | <1 | 9.0–12.0 |
| Fatty acid composition (%) | Palmitic acid (16:0) | 44.2 | 13.8 |
| Stearic acid (18:0) | 4.5 | 6.8 |
| Oleic acid (18:1) | 39.6 | 41.7 |
| Linoleic acid (18:2) | 9.8 | 35.6 |
and Ca2ZnO) render high durability characteristic in transesterification with low active metal leaching for several cycles. The strong interactions between active metals provide a superior synergism of high basicity and stability effect for transesterification reaction.

Competing interests
The authors declare that they have no competing interests.

Authors’ contributions
LHV conceived of the study, participated in its design and coordination and drafted the manuscript. JIC provided advice on the testing method, analyzed results and helped to draft the manuscript. NFA and RNM performed partial experiments and analyzed results. YHTY contributed to conception and catalyst design, reaction study and provided research advice in data interpretation. All authors read and approved the manuscript.

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