Biodiesel Synthesis from Used Cooking Oil Using Red Mud as Heterogeneous Catalyst

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Biodiesel Synthesis from Used Cooking Oil Using Red Mud as Heterogeneous Catalyst

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Abstract. The problem associated with biodiesel production is economic feasibility. The biodiesel cost will reduce when the low cost feedstock was used as feedstock. Used Cooking Oil (UCO) is a promising candidate as raw material for biodiesel synthesis. In this study, the investigation of biodiesel synthesis from UCO was studied using red mud as heterogeneous catalysts. The catalyst was prepared by impregnating Potassium metals on red mud. The catalyst physico-characteristics were determined using Nitrogen gas adsorption, FT-IR, XRD, and XRF. The catalyst was tested to synthesize biodiesel from UCO. The reaction temperatures, methanol to oil mass ratio, and amount of catalyst were varied to examine their effects on biodiesel synthesis. The optimum reaction conditions were obtained at 60°C of reaction temperature, 10:1 of methanol to oil mass ratio, and 10% of catalyst amount. The highest biodiesel yield of 94.4% was obtained.

Introduction

Several factors that lead to the search for alternative energy sources are increasing energy demand, global warming due to greenhouse gas emissions, environmental pollution, and reduced fossil fuels supply [2]. Biodiesel has been proven to replace diesel fuel. Biodiesel contains of monoalkyl fatty acid esters with long chain of hydrocarbons. Biodiesel has several benefits such as emit less pollutant subtances, biodegradable, generates from renewable sources and reduce greenhouse gases production [14]. Conventionally, transesterification of triglycerides and esterification of free fatty acids were carried out to yield biodiesel using assistment of acid or base catalysis . However, the problems encountered with the biodiesel synthesis are the expensive of feedstocks cost and the emersion of competition between energy and food supply [10]. Currently, more than 95% of biodiesel synthesis has been using various type of edible oil, such as canola, palm, corn, sunflower and soybean [4]. Nevertheless, the use of edible oil lead to the uneconomically viable of biodiesel price compared to petrodiesel. Reducing the feedstocks cost can be achieved by using non-edible oils and waste cooking oils as raw materials. The non-edible oils have low price and easy cultivating in land-poor. Energy crops such as jatropha [15], mahua [13], karanja [11], Ceiba pentandra [16], Calophyllum inophyllum [3], and rubber seed [8] represent some of non-edible oils plant. Used cooking oils (UCO's) are oils or animal fats that have been used for cooking or frying in the food processing industry, restaurants, and households. UCO's have a low-cost which available abundantly and sustainable. Utilization of UCO's as a feedstock will increase the economic viability in term of reducing of biodiesel price.

In the last decades, many researchers have been studied different heterogeneous catalysts for biodiesel synthesis [1, 5-7, 9, 12]. Many advantages was gained such as easily separated from the product mixture, produce less wash water and can be reused when applying the heterogeneous catalysts for biodiesel synthesis compared to the homogeneous catalysts. From the literature study, it has been summarized that most of the heterogeneous catalysts preparation have time consuming preparation, complex synthesis routes, and expensive. To address these drawbacks, a new type of heterogeneous catalyst from tailing residue needs to develop. Red mud is a residue from extraction process of bauxite mineral to produce alumina in the Bayer process. The pH of red mud is high with in the range between 10–13 due to the usage of sodium hydroxide during alumina extraction from bauxite. Red mud contains several metals oxide such as Al_2O_3, SiO_2, Fe_2O_3, TiO_2 and more which is regarded as a hazardous waste. Red mud which has high alkalinity is potential to utilize as a heterogeneous basic catalyst for biodiesel production. The red mud utilization as a heterogeneous catalyst will emerge several benefits i.e. low cost, environmentally benign, and exhibits high activity during biodiesel synthesis.

This study focused on the biodiesel synthesis from UCO using red mud as catalyst. The incipient wet impregnation procedure was applied to synthesize the catalysts. The catalyst physico-characteristics were determined using Nitrogen gas adsorption, FT-IR, XRD, and XRF. The investigation of several operating conditions in term of the reaction temperature (40 – 60°C), the methanol to UCO mass ratio (1:2 – 2:1), and the amount of catalyst to oil (1 – 10% wt. of UCO) were varied to obtain the optimum reaction conditions.

Experimental

Catalyst Preparation. The Ca/RM catalysts were synthesized by the incipient wet impregnation using metal salts solution of Calcium Nitrate ($CaNO_3$). Then, the red mud as catalyst support was mixed with a Calcium Nitrate solution and stirred vigorously while the temperature was heated up to dry the mixture. When the slurry of mixture was formed, the mixture was then calcined at 600 °C in a furnace for 2 hours. The textural characteristics of red mud and Ca/RM catalysts were analyzed using the Nitrogen adsorption isotherm at 77 K using Quantachrome ASAP 2010 instrument. The structure and crystallinity of materials were identified by using a Rigaku Multiflex X-ray diffractometer, using radiation of Cu Kα, the high voltage source of 40 kV and 20 mA. Datas were recorded with scanning angle between 5 to 80° (2θ) at a scanning rate of 1◦/min. The X-Ray Fluorescence (XRF) spectrophotometer analysis was applied to determine elemental composition of the materials. A series of Hammett indicators i.e. phenonpthalein ($H\_$ = 8.2), 2,4-dinitroaniline ($H\_$ = 15) and 4-nitroaniline ($H\_$ = 18.4) was used to determine the basic strength of Ca/RM catalyst.

Results and Discussion

Catalyst Characterization. The structure and crystallinity of the raw red mud and Ca/RM catalysts was determined by applying XRD analysis. The results of diffraction pattern peaks analysis from XRD measurements were depicted in Fig. 1. As illustrated in Fig. 1, the XRD diffractogram exhibits several metal oxides on red mud such as Fe_2O_3 (hematite), Al_2O_3 (boehmite), and TiO_2 (anatase titania) as major mineral phases. Meanwhile, on the Ca/RM catalysts the characteristics peaks of CaO were shown at 2θ =19, 23, 29, 32 and 34°. In addition, small peak of CaO was displayed at 2θ =31°. It is noteworthy that, the incorporated of potassium metals on red mud was succeed by the presence of the presence of characteristics peaks CaO of well as of on XRD Diffractogram. The findings in line with XRF results, in which potassium content in the Ca/RM catalyst increased after impregnation. The surface area was determined by N_2 adsorption isotherm analysis. The surface area of Ca/RM catalysts was 20.2 m^2/g, while that of raw red mud was 24.8 m^2/g. The reducing of surface area was attributed to the successful incorporating of potassium oxide to pores as confirmed in the results from XRD analysis. The strong base of Ca/RM catalysts were analyzed using Hammet indicators. The results showed that the phenolphthalein colour ($H\_$ = 8.2) was changed from colourless to pink as well as the 2,4-dinitroaniline colour ($H\_$ = 15) from yellow to mauve. However, the 4-nitroaniline colour ($H\_$ = 18.4) has not changed. It concluded that, the Ca/RM catalyst's is considered as a strong base catalyst due to the designated of basic strength was in the between $15 < H\_ < 18.4$.

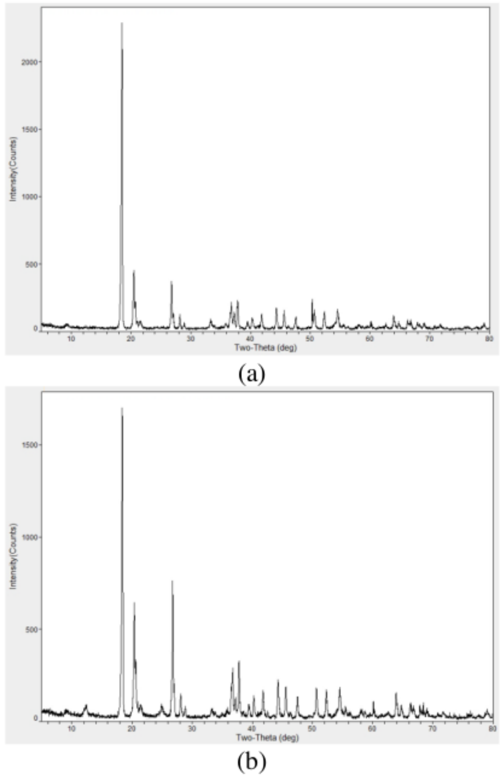

Fig. 1. XRD measurements: (a) raw red mud; and (b) Ca/RM catalysts

Catalyst Activity. The transesterification reaction is reversible, thus it needs to employ a methanol excess to keep the reaction in the forward direction. The effects of methanol to KSO mass ratio on the biodiesel yield in the transesterification reaction of UCO were showed in Fig. 2. As illustrated in Fig. 2, the yield of biodiesel was 74% when methanol to UCO mass ratio was applied at the value of 1:1. Increasing of methanol to UCO mass ratio to 2:1 increased the biodiesel yield to 89%. The further increasing of methanol to UCO mass ratio from 3:1 to 4:1 did not significantly enhance the biodiesel yield. Therefore, the maximum biodiesel yield was achieved at a 2:1 methanol to UCO mass ratio.

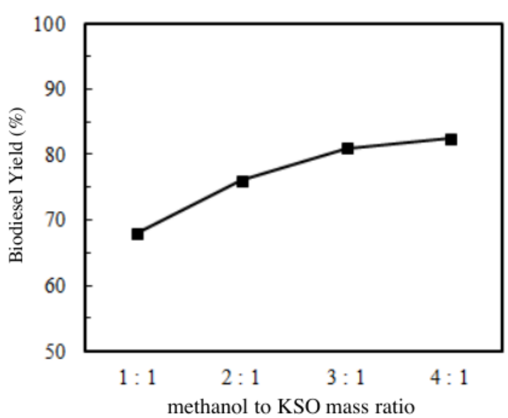

Fig. 2. Influence of Methanol to UCO Mass Ratio on Biodiesel Yield

The one of important parameters that affects the biodiesel yield is catalyst amount. The value of catalyst amount was varied from 1 to 10 wt. % UCO. As presented in Fig. 3, the biodiesel yield was enhanced when catalyst amount from increased from 2.5 to 5 wt. % UCO. The maximum biodiesel yield of 86% was attained at a catalyst amount to 10 wt. % UCO. The increasing of catalyst amount lead to more total number of available active sites resulted in faster reaction rate to achieve reaction equilibrium and generated more FAME.

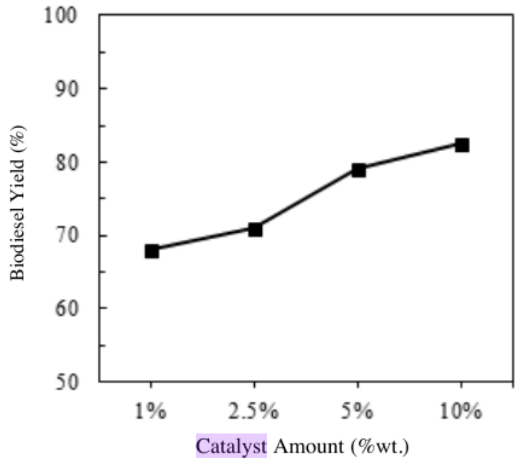

Fig. 3. Influence of Catalyst Amount on Biodiesel Yield

The variations in reaction temperatures are applied to investigate their influence of on the biodiesel yield as shown in Fig. 4. The different reaction temperatures were applied from 40 to 60 °C min to obtain the optimum reaction temperature for reaction. The increasing of reaction temperature from 40 to 50 °C resulted the enhancing of biodiesel yield from 76 to 83%. It was observed that at a reaction temperature of 60 °C, the biodiesel yield reached maximum value at 88%. At high temperature, the limitation between mixture of reactant and catalyst would reduce and the reactant molecules also gained more kinetic energy. It caused an accelerating of reaction rate between reactants that resulted to produce more FAME.

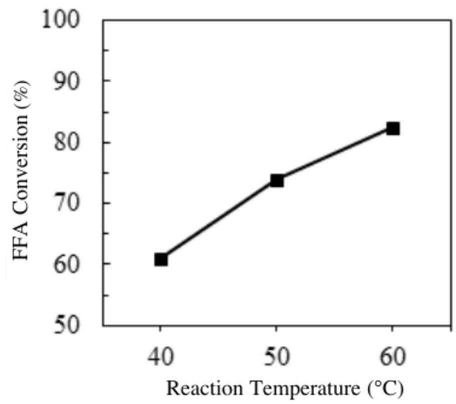

Fig. 4. Influence of Reaction Temperature on Biodiesel Yield

Conclusions

Red mud which a residue from bauxite processing was employed as catalyst support for biodiesel production from Used Cooking Oil. The catalyst was prepared by impregnating the red mud with Potassium metal. The optimum reaction conditions were obtained at 60°C of reaction temperature, 10:1 of methanol to oil mass ratio, and 10% of catalyst amount. The highest biodiesel yield of 83% was obtained.

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