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Heterogeneous catalysis of transesterification of soybean oil using KI/mesoporous silica

C. Samart [a,*], P. Sreetongkittikul [a], C. Sookman [b]

[a] Department of Chemistry, Faculty of Science and Technology, Thammasat University, Klongluang, Pathumtani 12121, Thailand
[b] Chemical Engineering Division, Faculty of Engineering, Rajamangala University of Technology Krungthep, Sathorn, Bangkok 10120, Thailand

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ABSTRACT

Heterogeneous catalysis of transesterification using KI/mesoporous silica catalyst was utilized to produce biodiesel from soybean oil and methanol. The effects of reaction temperature, percentage of KI loading, reaction time, and amount of catalyst on the conversion to methyl ester were studied. The results showed that increasing reaction temperature, KI loading and reaction time can enhance the conversion. The optimum condition was the reaction temperature at 70 °C, 15 wt.% of KI, a reaction time of 8 h., and a catalyst amount of 5.0% by weight of the oil which yielded 90.09% of the conversion. The fuel properties of biodiesel from the optimum condition were tested and found that only viscosity showed over standard. However, the high viscosity can be reduced by separation of non-reacting soybean oil.

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1. Introduction

In recent years, world energy crisis is reached due to an increasing energy demand and limitation of fossil fuel reserves. Therefore, energy researches have increased directly towards alternative renewable fuel. One of more promising approaches is biodiesel because it is practical and suitable for agricultural countries. Biodiesel is defined as methyl ester produced from vegetable or animal oil which can be used as biofuel. It is non-toxic and bio-degradable [1]. Substitution of petroleum-derived diesel with biodiesel can decrease emission of life-cycle global warming gas as high as 45%. In addition to global warming gas, tailpipe emissions of carbon monoxide, particulate matter (PM) or fine particles, and hydrocarbons are considerably decreased [2]. Utilization of biodiesel can also reduce reliance on oil imports [3]. The conventional transesterification which uses homogeneous catalysis can provide high yield of methyl ester (more than 95%) [1,4]. However, the conventional method has a high cost in operation especially for waste water treatment [4]. The other disadvantage is that the glycerol by-product contains salts resulted from the neutralization of the catalyst. This needs a more costly purification process, if the product is a high-purity, saleable glycerol [5]. For this reason, heterogeneous catalysis is likely to be used replacing the conventional method in the near future.

Presently, there are several catalysts available for using in heterogeneous transesterification. Solid base catalysts have been developed for heterogeneous catalyzed transesterification. In the reaction, MgO, CaO, SrO, and ZnO were are used and they exhibit the conversion efficiency ranging from 90% to 95% [6–10]. The alkali metal on supporter is another catalyst type for heterogeneous transesterification. Superbase catalyst (Eu_2O_3/Al_2O_3), one type of alkali metal catalyst, can catalyze transesterification of soybean oil and give 65% conversion [11]. The other catalyst is potassium supported on larger surface-area material e.g. zeolite and alumina. This catalyst type yields the conversion at 85% which is still lower than that of solid base catalyst [12,13]. However, the advantage of alkali support catalyst is requiring a small amount in catalysis. Thus the aim of this work is to improve the activity of KI support catalyst by using mesoporous silica as a supporter.

2. Experiment

2.1. Catalyst preparation

Two grams of pluronic P123 (Aldrich, USA) were dissolved in 72 ml of 2 M HCl and stirred for 4 h. Pluronic P123 in tetraethoxysilane (Fluka, Italy) solution at the ratio of 3:1 poured in the previous solution and stirred for 4 h. Hydrothermal treatment was applied after the end of reaction time. The mixture was filtered, dried at 100 °C for 6 h., and calcined at 600 °C for 3 h. to obtain the silica powder. The KI solution was impregnated on mesoporous silica by incipient wetness impregnation with concentration of KI at 5, 10, and 15 wt.%. Before being impregnated, the mesoporous silica was evacuated and determined the water adsorption volume. Then, a certain amount of KI solution was loaded in mesoporous silica and left to stand for 24 h. The catalyst was dried at 100 °C for 12 h. followed by calcining under static condition for 3 h. at 600 °C. Surface area, pore size, and pore volume of catalysts were characterized by adsorption desorption

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* Corresponding author. Tel./fax: +662 5644483.
E-mail address: chanatip@tu.ac.th (C. Samart).

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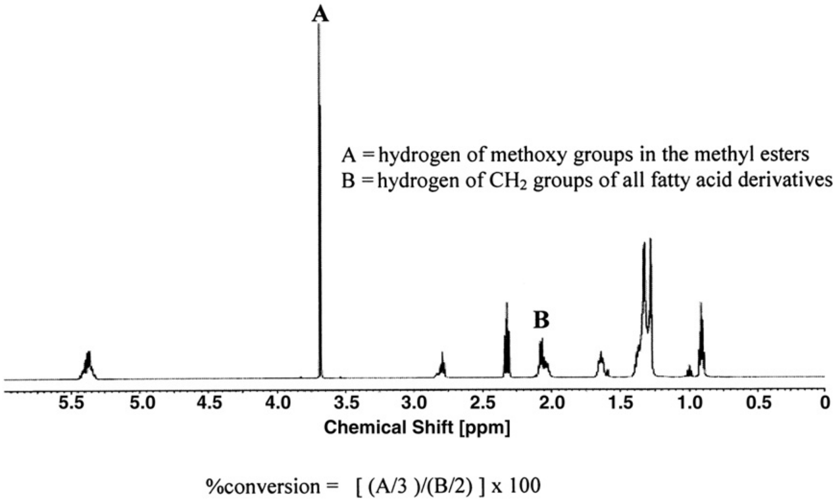

Fig. 1. Analysis of methyl ester produced from the transesterification of soybean oil and methanol using Nuclear Magnetic Resonance Spectroscopy (NMR).

technique (Autosorb 1C, Quantrachome Instruments Co. LTD.). Crystal structure of catalysts was studied from X-ray powder diffraction pattern using Siemens: D8 Advance using Cu Kα radiation in the range of 5° to 80° with a step size of 0.02°.

2.2. Transesterification of soybean oil

The transesterification reaction was carried out in a 250 ml one-necked round-bottom flask equipped with a magnetic stirrer and water-cooled condenser and placed heating oil bath. The ratio of methanol:commercial edible grade soybean oil (Thai Vegetable Oil Public Company Limited, Thailand) at 16:1 was used in the reaction. The effect of reaction temperature (ranging from 50 °C to 70 °C), KI loading percentage (from 5 to 15 wt.%), reaction time (from 6 to 10 h.), and catalyst concentration varied from (2.5% to 7.5%) based on weight of soybean oil on the methyl ester production were investigated. To determine the methyl ester production, the product from the transesterification was filtered and washed with saturated NaCl solution to remove the catalyst and glycerol. After dehydrating the organic phase with anhydrous $MgSO_4$, the treated product was dissolved in $CDCl_3$ and analyzed by Nuclear Magnetic Resonance (NMR) (Bruker, DPX-400) [14]. The conversion of the soybean oil to methyl esters was determined by the ratio of the signals at 3.68 ppm (hydrogen of methoxy groups in the methyl esters) and 2.30 ppm (hydrogen of CH_2 group of all fatty acid derivatives) as described in Fig. 1.

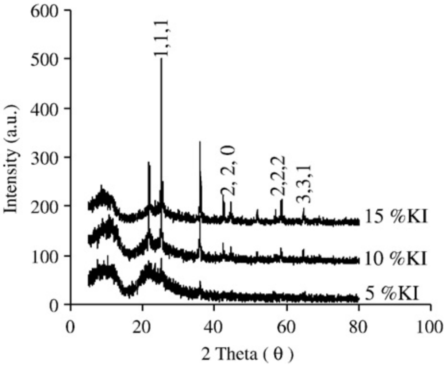

Fig. 2. X-ray diffraction (XRD) pattern of KI/mesoporous silica at KI concentration of 5% wt., 10% wt., and 15% wt.

The product from the selected condition considered basing on the highest yield with good economic were tested fuel properties viscosity, flash point, pour point, and copper strip following the method described in the American Society for Testing and Materials (ASTM).

3. Results and discussion

3.1. Characterization of catalyst

The X-ray diffraction (XRD) patterns of KI/mesoporous silica coated at different KI concentrations with the calcination temperature of 600 °C showed that the characteristic peaks of potassium oxide (K_2O) face-centered cubic crystal at 2 theta equal 25.3°, 41.9°, 51.9°, and 66.9°, while the characteristic peaks of silicate hydrate phase (SiO2 × H2O) is at 21.8° and 35.7° (Fig. 2). There was no characteristic peak of KI observed in the XRD pattern. This means that all of KI phases were transformed into K_2O phase. As can be seen in Fig. 2, the intensities of K_2O peaks were increasing following to the increasing of KI concentration.

Table 1 shows the physical properties of mesoporous silica and KI/mesoporous silica impregnated at various KI concentrations. Compared to KI/Mesoporous silica at various KI concentrations, the mesoporous silica had the largest surface area (BET = 800 m^2/g and Langmuir = 1324 m^2/g) with the pore volume and pore diameter of 1.05 cm^3/g and 52.3 Å, respectively. However, after mesoporous silica was coated with KI, the langmuir surface area was reduced dramatically and BET surface could not determined. This is because pores of silica were covered by metal potassium. According to covering of metal potassium, the porous silica properties were changed to be similar to the nonporous solid properties, which could not precisely determine by the BJH model. One should note that the pore diameter of KI impregnated mesoporous silica was larger (Table 1). This result is in agreement with several reports that reduction of surface area and

Table 1
The physical properties of mesoporous silica and KI/mesoporous silica.

Catalysts	BET surface area (m^2/g)	Langmuir surface area (m^2/g)	Pore volume (cc/g)	Pore size (Å)
Mesoporous silica	801	1,324	1.05	52.3
5%KI/Mesoporous silica	–	10.89	0.0967	560
10% KI/Mesoporous silica	–	0.05	0.00776	836
15% KI/Mesoporous silica	–	3.24	0.0485	910

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