Transesterification of Jatropha Seed Oil Naturally Extracted by Distilled Water on Highly Stabilized Structure of Zeolite NaX Impregnated with Potassium Buffer Solution

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Abstract—This research aimed at establishing species of active potassium on the 3D-framework of Zeolite NaX that was prepared with a molar ratio of 1Al:O:1.65K:O:2.2SiO:2: 5.5 Na2O:12H2O by hydrothermal synthesis. The potassium acetate/acetic acid buffer solution impregnated catalysts produced the desired K loadings of xKa/NaXs (x values were 9, 10, 11 and 12 wt%). Characterization of catalysts were carried out with FTIR, N2 adsorption-desorption, XRD and CO2-TPD to analyse the respective parameters of the reagents (catalysts) and the product. The 3D-framework of NaX was not destroyed but the surface area and the volume of its micropore were found to have slightly decreased when number of loadings of K was increased. All prepared xKa/NaXs were used to conduct transesterification of Jatropha Curcas Linnaeus (JCL) seed oil and methanol at the molar ratio of 1:16. 12K/NaX emerged as the best catalyst which produced 78.39 wt% yield of biodiesel and found to be the highest yield at a reaction time of 3.5 hours. The basicity increased with K loadings and facilitated a better catalytic performance for biodiesel synthesis from this source.

Index Terms—Biodiesel, Jatropha seed oil, Potassium, Transesterification, Zeolite.

I. INTRODUCTION

The world has seen rising levels of energy consumption lately owing partly to the growth in population as well as rapid industrialization of which the result is an energy crisis because of limited resources of non-renewable energy; fossil fuel [1]. The current rate of global energy consumption is presently perceived as unsustainable due to depleting supplies and immense utilization of these energies which also contaminate the environment. Many opinions are that the change in climate as of now constitutes a pressing worldwide environmental issue [2]. In this point of view, alternative energy resources will form a core of the future of energy consumption and sustainability in the world [1, 3].

Amongst the promising sources of energy is biodiesel, also called mono-alkyl ester, because of its remarkable properties as being biodegradable, renewable and non-toxic [4, 5]. Biodiesel can be produced by treating vegetable oils, tallow and animal fats using alcohols with low molecular weight such as methanol and ethanol in presence of acid or base catalysts produced via a process called transesterification [6]. In several attempts, edible oils such as peanut, rapeseed, palm, soybean, and non-edible oils including sea mango and Jatropha were exploited as renewable resources to produce biodiesel [7, 8]. However, using edible oils as feedstock may affect balance of food supply. Jatropha seed oil, among non-edible resources, has mostly been used [7, 9] because it is found to be fast-growing which is protected from insect attack by toxic compounds such as curcin in its seed kernel [9, 10]. Fortunately, this kind of non-edible oil thrives well in Thailand [11]. Consequently, it is considered as rich source for biodiesel production because the seed is found to contain extractable oils exceeding than 30 wt% [9]. In this research, Jatropha seed oil was utilized as a precursor to synthesize biodiesel with heterogeneous catalysts with the intention of catalyst recovery the from the mixture for possible reuse. Ensuring good health is a concern for all, so organic solvents that may cause carcinogenicity were not used in the oil extraction from the seeds. Instead, a screw press is used for the extraction and distilled-water wash to ensure a safer procedure for energy sustainability and environmental conservation.

Amongst the various catalysts for Jatropha seed oil biodiesel synthesis are heterogeneous catalysts such as alkaline earth metal oxides, basic zeolites, as well as
hydrotalcites which have been modified and practically used [12]. The main advantage in using these catalysts is the ease of separating from the mixture and its suitability for treating feedstocks of low quality, that is TG with higher percentage of water and free fatty acids [13]. Besides esterification of oil in heterogeneous system, using heterogeneous basic catalysts do not easily undergo reverse reaction to generate soap through the neutralization of the FFA and saponification of the triglyceride [14]. In addition, heterogeneous catalysts can be used relatively over in subsequent reaction since they can easily be separated from the reaction mixture at the end of the process. These catalysts are found to be less corrosive than other acidic ones guaranteeing safer handling and adequate safety for the environment [15, 16].

In several investigations, basic catalysts are supported for use in transesterification of various oils into ethyl esters. One variety of those, zeolites such as zeolite NaX and NaY are widely utilized as supporting materials because their structures are found to be porous, giving them their unique properties of enhanced surface area capable of improving more active phase distribution than other materials [17] and supercage cavities in 3D-framework. Therefore, applications of zeolite NaX and NaY are found to be useful in many research fields including molecular sieve, functioned-surface adsorption, pressure and chemical tolerance [18]. Zeolite NaX is hereby selected as a heterogeneous catalyst for this research of biodiesel synthesis because zeolite X possesses enhanced basicity compared to zeolite Y. Literature shows that it is more active and the yields in biodiesel production is higher [19, 20]. A loading of alkali or alkaline earth metal onto the zeolite cavities is found to improve the basic strength and the basicity of zeolite NaX. Manadee et al [21] loaded zeolite NaX with KOH for methanolysis of soybean oil in a research. Results of the research showed that the zeolite NaX had increased in basic strength from less than 9.3 to more than 15 when KOH loadings were increased. It was observed then that, the modification of KOH marginally collapsed the framework of the zeolite structure because of the high loading of KOH, leading to agglomeration of the active species of the KOH.

Nonetheless, the entire pore structure was not significantly destroyed, and 10% loading of KOH yielded the best catalyst. This caused the yield of biodiesel to be reduced because of the reduced active basic sites. [22] demonstrated the correlelation between basicity and biodiesel yield with the transesterification of sunflower oil using KNO3/NaX as the catalyst in the presence of methanol. The prepared catalyst of 35 wt% of KNO3 carried highest catalytic activity and provided highest biodiesel yield. Although the catalyst was at its best performance, a longer time (6h) for the reaction and sizable volume of powder catalyst (10g) was consumed. Sunflower oil transesterification was also conducted by Luz Martinez et al., [14] with methanol and using CuONano/NaX Zeolite as the catalyst species. The basicity was also found to have highly influenced biodiesel yield and the catalyst with 16wt% CuONano showing the highest catalytic activity. Although the catalyst provided high biodiesel yield, plenty of powder catalyst (5g) was consumed and long reaction time (6h) had also been spent. Though such catalysts provided the high biodiesel yield, reaction times to complete transesterification has also been prolonged because of the instability of zeolite framework structures.

The objectives of this research are to extract Jatropha seed oil by a compressed machine and naturally wash by distilled water. This kind of oil will be used as a precursor for transesterification with methanol in a molar ratio of 1:16 as done by Manadee [21] on xK/NaXs. The 9-12 wt% of K species on NaX zeolite synthesized rice husk silica will be prepared by hydrothermal synthesis to establish active species on the functioned framework. Characterizations will elucidate catalytic properties of potassium species immobilized on zeolite NaX through several techniques including Fourier transform infrared spectroscopy (FTIR), CO2 temperature programmed desorption X-ray diffraction, (XRD), and N2 adsorption-desorption. The Correlation between catalytic performance characteristics with the yield in Jatropha seed oil synthesis is to be investigated as well.

II. METHODOLOGY

A. Synthesis of Zeolite NaX

Zeolite NaX was synthesized from silica, obtained from a waste product, rice husk from rice mills following the method adopted by Manadeet al., [21]. The molar ratio of the silica gel was 1Al2O3:1.65K2O: 2.2SiO2: 5.5Na2O:122H2O which was prepared from Na2SiO3 and NaAlO2 was kept in paraffin film sealed polypropylene bottle. It was crystalized by hydrothermal treatment in oil bath at 60 °C for 48 hours. The mixture was then hydrated with deionized water after filtering at a ph of 9, thereafter it was dried overnight at a temperature of 60°C.

B. Catalyst Preparation

The xNa/NaX and xK/NaX were prepared by impregnation procedure adapted from Manadee [21] and Ramos [23]. The adsorbed water in zeolite NaX was removed by drying for 2 hours in an oven at a temperature of 100 °C. It was then, doped with Potassium acetate buffer solution (99% CH3COOK, Unilab and glacial acetic acid 100% CH3COOH, BDH). Various potassium loading of 9, 10, 11 and 12 wt% were prepared as the catalysts for transesterification and dried at room temperature and later at 80°C for 12 hours. The samples were then calcined for 5 hours at a temperature of 550°C [24].

C. Characterization Techniques

An X-ray diffractometer, (Bruker axs D5005) was used to acquire the powder patterns of the catalyst. A current and voltage of 40mA and 40kV respectively were set to generate the Cu Kα X-ray (at λ = 1.54 Å). The angle of scanning, 20, were varied in steps of 0.05 degree/min from 5 to 50, using 0.2 slit and all scanning done at the same time to assure samples peak intensity comparability.

The characteristic functional groups on the catalysts were investigated using FTIR employing the KBr pellet technique. The KBr and samples of catalyst were dried in an oven for 3 hours at the temperature of 110 °C to dehydrate them and then mixed together at a ratio of 50:1 (KBr to sample). The mixture is then kept in a holder and compressed, and the spectra measured and recorded using spectrometer, Perkin Elmer Spectrum GX with the wavenumber range from 400 cm⁻1 - 4,000 cm⁻1.
Micromeritics ASAP 2010 was used to determine the catalysts surface area for N2 adsorption-desorption analysis. Before the measurements of the adsorption, catalyst of mass 0.1 to 0.2 g was degassed for 8 hours at a temperature of 300 °C. The range of 0.01 to 0.2 relative pressure (P/P0) was used to calculate the BET surface area, employing the Barrett-Joyner-Halenda (BJH) method to calculate pore sizes from the isotherms CO2 adsorption-desorption branches.

Basic site characteristics were obtained with BELCAT-B instrument for CO2-TPD analysis. Number of basic sites and strength of the catalyst’s samples were obtained by the maximum CO2 absorbed as well as the temperature of desorption respectively. Quartz wool is used to cover an amount of 0.05g of the catalyst sample packed into a holder. Under a flow of helium (He) gas, the samples were heated to 500 °C from ambient temperature at 10 °C per minute and maintained for 1 hr. CO2 mixed with helium (He 99%) gas was supplied after it had cooled to 50 °C and at a rate of 30 ml/min for 30 min. Physisorbed species were later purged from the sample with He for 1 h. The temperature was ramped at 10 °C/min from 100 to 810 °C and then, peaks at low and high temperatures were obtained from CO2-TPD. The area under the curve was integrated to get the basic site density [23].

D. Compression of Triglyceride from Jatropha Seeds

Oil rich seeds of Jatropha Curcas L. were obtained for this experiment from field crops in Nong Ya Plong District, Petchaburi, Thailand. The seeds were washed from adhering dust with distilled water and crushed with a 40-horsepower mechanical compressor for 30 minutes. The crushed mixture was washed by distilled water and filtered to separate solid waste from Jatropha seed oil without extraction. The remaining 2 layers between Jatropha seed oil (top layer) and distilled water (bottom layer) was left at room temperature to separate from each other. The bottom layer of distilled water was drained out and the top layer of Jatropha seed oil was finished off by warming at 70 °C to evaporate the remained water. Finally, Jatropha seed oil was held in a 1000-liter chamber (Oxygen-free) to precipitate small particles for 3 months. After that, a yellowish brown-liquid oil (Triglyceride) on the top layer of the chamber was separated to filter and transfer to a stock-solution chamber for transesterification.

E. Performance of Catalyst in Transesterification

5.0g of Jatropha seed oil is initially preheated to a temperature of 65 °C with Heto Lab Equipment, (AT110, Denmark) water bath. 2.9g of methanol (99.8% CH3OH, Merck) was mixed with a catalyst of 0.2g. A water-cooled condense fitted to a 50-mL round-bottom flask was used for the stirring of the mixture (of oil to methanol ratio of 1:16) at 400 rpm using a magnetic stirrer. A Centrifuge, (Heraeus SEPATECH, Labofuge 200 Germany) was used to separate the mixture after the desired reaction time, at a speed of 3000 rpm. The upper layer of the reaction mixture was decanted into a separation funnel and later washed to remove methanol and glycerol. The lower layer of catalyst was washed with DI water for possible reuse. Rotary evaporator was used to remove the excess and entrained methanol. TLC plate (Silica Gel 60 F254, Merck) is used preliminarily to identify the product of the mixture (biodiesel) by spotting on the plates and then developed by immersing in a solvent mixture of diethyl ether (C4H10O, AR grade)/ glacial acetic acid (100% CH3COOH, BDH) and petroleum ether (AR grade, JT Baker) of ratio (15:1:85, v/v/v) and exposed to iodine vapor (UNICHROM) to estimate the conversion. A gas chromatograph (GC-HP6890-Series) is used to determine the compositions of biodiesel from the reaction with the internal standard method. The GC equipped with 30-meter HP-INNOWAX polyethylene glycol capillary column, 0.32 mm id and 0.15 µm film thicknesses and with a flame ionization detector (FID) had its column temperature raised and held at 140 °C for 3 min initially before increasing at a rate of 10 °C/min to its final temperature of 240 °C and held constant for 8 minutes. The methyl ester content analyzed by the GC is used to calculate the yield of biodiesel from the transesterification process adapting the equation of Supamathanon, [11]. The internal standard method is used to calculate the biodiesel yield as obtained from a separate calibration curve [21]. The biodiesel yield, yield, was calculated using Eq 1, with the methyl ester mass concentration Cester, the density of sample of methyl ester (g/L), equation 1 gives;

\[
\text{yield} = \frac{(\text{Cester} \times n)}{\rho_{\text{oil}}} \times 100% \\
\text{Eq. 1}[21]
\]

III. RESULT AND DISCUSSION

A. XRD Characterization of Catalysts

XRD patterns of the calcined zeolite NaX, xK/NaX are shown in Fig. 1 (that is NaX doped with potassium acetate buffer solution, with loadings K, x = 9, 10, 11 and 12 wt%). The intensities of characteristic peaks of NaX were observed to be decreasing on all catalysts [25] with increasing K loadings. Peaks indicating the presence of new phases metals and alkali acetate at 2-Theta of 9° (ICPDS 29-1160) or K2O at 2-Theta of 31°, 39°, 51°, 55° and 62° were absent [21]. The absences of the new phases and large clusters of K2O on the zeolite framework indicated a good depressiveness. The occlusion of excess K in the cavities of NaX structures impregnated with potassium acetate solution might be responsible for the partial collapse of the structures [21].

![Figure 1. X-ray Diffraction patterns of metal oxide phases in zeolite NaX (9–12 wt% K).](image)

The temperature of calcination (550 °C) was found to be enough to have transformed all potassium acetate into K2O. Furthermore, secondary scattering of X-ray caused by species of K might have been responsible for the decrease
The symmetric vibration bands of COO− from CH3COOK were absent at bands 1577 cm−1 and 1641 cm−1. Furthermore, carbonate species of K2CO3 showed strong bands at 1457 cm−1 and 1385 cm−1. The T−O bond is suspected to have taken part in the reaction between K2CO3 and the support resulting in the shift to 1119 cm−1 because of weaker bond [28] and the stretching vibrations of Al-O-K or Si-O-K groups is partially responsible for the broad band seen at 3432 cm−1 [29]. Active basic species of this catalyst for the transesterification could come from the protons of OH groups on the support replaced by K+ ions form Al-O-K or Si-O-K groups during activation [24].

In peaks observed. It is also likely that the scattering might have been caused by the K species on the external surface of the structure resulting in lower reflection from the plane of the zeolite. Notwithstanding, the preserved structure as indicated by the characteristic peaks at high loadings of potassium resulted from the alkali metal preventing the hydrolysis of the bond of Si-O-Al during the zeolite thermal treatment [26] [27]. Accordingly, characterization manifested that the active sites on the catalyst had K2O derived from CH3COOK, surface hydroxyl groups, and Al-O-K (or Si-O-K) groups.

**B. FTIR Spectroscopy Characterization of Catalysts**

Figure 2 shows the FTIR spectra of zeolite NaX at the various levels of potassium loading. The COO−asymmetric stretching was seen to be responsible for the characteristic band at 1407 cm−1.

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**C. N2 Adsorption-Desorption Characterization of Catalysts**

Table 1 shows the BET surface area and pore volume of the catalysts and zeolite NaX.

This work found the results for the surface area and the micropore volumes of the zeolite NaX to be in similar range as literature. [21]. These characteristics were seen decreasing with increasing loadings of K, indicating that the surface of the structure was dispersed with potassium oxide and were occluded in the cavities as well. The change of the zeolite structure to amorphous phase might be due to the large surface area decrease. BET surface area decreased from 711 to 186 m2/g and pore volume because sodium oxide resided in the zeolite cavities when the zeolite NaX was doped with 4.2 wt% sodium acetate solution.

Furthermore, calcination might have caused the collapse of the structure or amorphization resulting in the lower loading of K catalysts possessing higher surface area than higher ones and in the order.

**TABLE I. PHYSICAL PROPERTIES OF THE CATALYSTS AND ZEOLITE NAX AND BIODIESEL YIELD**

<table>
<thead>
<tr>
<th>Sample</th>
<th>BET surface area (m²/g)</th>
<th>Micropore volume (cm³/g)</th>
<th>External surface area (m²/g)</th>
<th>Internal surface area (m²/g)</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zeolite NaX</td>
<td>591</td>
<td>0.27</td>
<td>-</td>
<td>-</td>
<td>Suppes et al. 2004</td>
</tr>
<tr>
<td>Zeolite NaX</td>
<td>650</td>
<td>0.27</td>
<td>72</td>
<td>578</td>
<td>Manadee et al. 2017</td>
</tr>
<tr>
<td>Zeolite NaX</td>
<td>711</td>
<td>0.29</td>
<td>90</td>
<td>621</td>
<td>This work</td>
</tr>
<tr>
<td>9K/NaX</td>
<td>637</td>
<td>0.27</td>
<td>79</td>
<td>558</td>
<td>This work</td>
</tr>
<tr>
<td>10K/NaX</td>
<td>567</td>
<td>0.24</td>
<td>72</td>
<td>495</td>
<td>This work</td>
</tr>
<tr>
<td>11K/NaX</td>
<td>549</td>
<td>0.22</td>
<td>69</td>
<td>480</td>
<td>This work</td>
</tr>
<tr>
<td>12K/NaX</td>
<td>537</td>
<td>0.20</td>
<td>65</td>
<td>472</td>
<td>This work</td>
</tr>
</tbody>
</table>

In addition, BET surface area of zeolite Beta with alkaline loadings decreased when the metal loadings increased [27]. The results suggest that part of the framework was dissolved during doping and filling of cavities by the potassium oxides. The blockage of micropores and the decrease in surface area of the structure might have induced lesser available base active points for activity but this loss of area is seen to have impacted less on the active species of the catalysts [30]. Transesterification of vegetable oils with heterogeneous catalysts has its chemical activity strongly affected by the support type as seen from the results of this study and also suggests that the potassium species is strongly bound to the surface of the NaX zeolite structure. The catalytic activity is also enhanced by the buffer solution which prevented the framework of the zeolite from collapsing.

**D. Catalysts Characterization by CO2-TPD**
biodiesel was found to have increased when K loadings increased, whereas the spots of crude Jatropha seed oil could still be seen with 9-11% K loadings indicating an incomplete conversion for the reaction time after 3 hours. This meant that conversion of crude oil to biodiesel increased at higher loadings of K as indicated from the spot size on the TLC plates. [33]. Transesterification on 12K/NaX obtained a complete conversion. Obviously, this experiment has shown a sole spot of biodiesel as well as the spot of FAME standard reagent at the final stage of a stationary plate.

The foregoing shows that, the 12K/NaX doped with potassium acetate buffer solution were largely active for transesterification because of a good depressiveness of K species through the framework of zeolite NaX. Strong solid base of the catalysts as a result of the impregnation with K loading on Al2O3 precursor was responsible for the high conversion rate experienced during the transesterification of the crude Jatropha oil. Nevertheless, higher yield of biodiesel was achieved when KOH/Al2O3 and KOH/NaY were used as a catalyst for transesterification of palm oil and methanol with lower K weight in KOH/NaY. Transesterification with KOH/NaY resulted in higher biodiesel yield in comparison to KOH/Al2O3 [30]. This indicated that the catalytic efficiency for transesterification of KOH/NaY was more active than that of KOH/Al2O3. Heterogeneous catalytic activity in transesterification was strongly affected by the type of support because of its higher adsorptive surface area and good-tolerance against high corrosive metal oxide with its 3D-framework structure [30, 34]. The strong attachment of potassium to NaY makes zeolites more suitable support than others. Besides, high loadings of K species could cause the collapse of zeolite crystalline structure because the excess K led to its agglomeration at basic sites leading to decreased activity and surface area. As a result, 12K/NaX exhibited the highest conversion for transesterification at 3.5 hours. This investigation focused on the loadings of K species for 12 wt% and consequently used throughout the experiment.

F. Effect of K loadings on zeolite NaX to biodiesel yield and reaction time

The biodiesel yields from xK/NaX (x = 9-12 wt% of K loadings) were determined by GC analysis. As the previous results from TCL indicated, 12K/NaX was the most active
reaction, all processes were focused on its activity. Figure 5 shows results after transesterification with 12K/NaX at the considered times of reaction. Biodiesel yields initially increased with reaction times because a great deal of initially stoichiometric molarity of TG to methanol were high and they were able to force reaction forward to form FAME.

![Graph showing FAME Yield (%)](image)

**Figure 5.** Transesterification of Jatropha seed oil and methanol with a molar ratio of 1:16 on 12K/NaX at 60 °C within 5 hours of reaction time.

Transesterification rate reached maximum yields (highest biodiesel yield of 78.39%) when reaction was 3.5 hours at the molar ratio of TG to methanol of 1:16. Additionally, surface area of the catalytic did not exceed with reagents so that basic sites of K species could dominated reaction to convert TG and methanol molecules to biodiesel [17]. It seemed identical to transesterification of sunflower oil using potassium nitrate loaded zeolite NaX as the catalyst with methanol [22]. An increase in the basicity and several basic sites emanating from the amount of potassium oxide on the support therefore resulted in higher yield of biodiesel. The main active site might have come from the decomposition of KNO3 to give K2O species for soybean oil transesterification. Nevertheless, the reaction rate gradually decreased when reaction time was over 4 hours and biodiesel yield also reduced from 77.74% (4 h.) to 76.00% (5 h.). This might be presumed that adsorption on active sites of TG and methanol was spill-over. Basic site strength of K species was covered and diminished catalytic sites of TG and methanol was spilled.

**IV. CONCLUSIONS**

Highly stable structures of xK/NaX (x = 9 - 12 wt% of K loadings) were successfully synthesized for Jatropha seed oil transesterification with methanol. Characterizations indicated a stability of the 3D-framework of NaX as a support structure for the metal oxide. Increase of the K loadings caused a slight collapse of NaX structure, but surface area sufficiently remained for hosting of K species. Stronger basic sites were established with the increase of K loadings and functioned as the active sites for transesterification. The conversion of TG and methanol increased with K loadings and resulted in a maximal yield of 78.39 wt% at the reaction time of 3.5 hours. The concentration of starting materials and the availability of basic sites has been found to be responsible of transesterification rate and biodiesel yields.

**ACKNOWLEDGEMENTS**

We acknowledge and appreciate the support of the following persons and institutions in their various capacities for this research: P. Maneechot and S. Artkla for the financial support from the National Research Council of Thailand (NRCT), Naresuan University International Scholarship programme for student scholarship for PhD programme at School of Renewable Energy and Smart Grid Technology (SGTech.), Staff Development Programme from Kumasi Technical University (KsTU) for granting Study Leave for Author, Instrument support was from Scientific Equipment Center, Suranaree University of Technology, Nakhon Ratchasima, 30000, Thailand, Research Laboratory Equipment Center, Maha Sarakham University, Maha Sarakham, 44000, Thailand, Faculty of Liberal Arts and Science, Roi-Et Rajabhat University (RERU), 45120, Thailand.

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