Synthesis and Characterization of Lithium Silicate and Potassium Silicate from Rice Hsk Ash by Hydrothermal-Microwave Method and Application for Biodiesel Catalyst

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SYNTHESIS AND CHARACTERIZATION OF LITHIUM SILICATE AND POTASSIUM SILICATE FROM RICE HUSK ASH BY HYDROTHERMAL-MICROWAVE METHOD AND APPLICATION FOR BIODIESEL CATALYST

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Abstract

Rice husk ash (RHA) is an agricultural by-product material. When analyzed, it was found that the silica content comprised a high component of 92-95% by weight. Silica could be converted to silicate compounds of lithium and potassium. This research studied the synthesis of silicate compounds of lithium and potassium by the hydrothermal microwave method. Lithium and potassium silicate were used as a heterogeneous biodiesel catalyst. The synthesis studies were of varying types and concentrations of alkali hydroxide (Li and K), including the wattage of the microwave oven and time of reaction. The alkali metal silicate (Li and K) were characterized by Fourier Transform Infrared (FTIR) spectrometer, X-ray diffractometer, and Raman spectrometer. The concentration of 10M of alkali hydroxide at 800 watts for 10 min. was under optimized conditions for the formation of lithium and potassium silicate. Scanning electron microscopy was used to analyze the crystalline characterization of lithium and potassium silicate that influenced the catalyst. These silicate compounds were then utilized as a biodiesel catalyst and provided methyl ester at 22.20% and 88.40% by weight, respectively, determined by gas chromatography-mass spectrometry.

Keywords: Alkali metal silicate, Rice husk ash, Hydrothermal-microwave method, Biodiesel catalyst

Introduction

Intensive research has been conducted to explore alternatives to petroleum derivatives due to the rapid decline of easily accessible oil reserves (Semwal *et al*., 2011). From the beginning of the 19th century, biodiesel has been considered to be one of the best alternative fuels produced by transesterification of lipids (Alrobaian *et al*., 2019). Biodiesel has been seen recently as a potential low-carbon fuel that would lead to sustainable energy (Zabeti *et al*., 2009). Biodiesel can diminish emissions of carbon monoxide, particulates, hydrocarbons, and sulfates. Biodiesel also has favorable fuel properties, such as enhanced lubricity, safe handling and storage, chemically bound oxygen, and comparable energy content (Ghiaci *et al*., 2011).

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The biodiesel industry has grown steadily since 1999 with commercial production facilities constructed worldwide (Luna *et al*., 2014; Cherikkallinmel *et al*., 2016). Furthermore, the oxygen content of 11-15% in the molecular structure speeds up the combustion process in compression ignition engines and decreases pollutants, such as soot, fine particles, and carbon monoxide (CO) (Atadashi *et al*., 2013).

The common feedstock for biodiesel is vegetable oil and animal fat. Fatty acid methyl esters (FAME) have found favor for use as a blend component of fuel due to its lack of ester components. Biodiesel can be used in conventional compression ignition engines which need no modification (Wang *et al*., 2012).

The widely used industrial method for commercial production of biodiesel from vegetable oils/fats uses a base catalyzed transesterification process, such as sodium hydroxide (NaOH), potassium hydroxide (KOH) as the homogeneous catalyst, and methanol (Dai *et al*., 2020). Homogeneous-based alkali hydroxide catalysts are the most common used in biodiesel production, but NaOH and KOH catalysts lead to soap formation and slow down reaction rate. Additionally, problems, such as a complicated separation downstream process and unwanted soap formation, have encouraged researchers to discover the application of heterogenous catalysts in biodiesel production. Other than solving the problems mentioned above, heterogeneous catalysts have a higher adaptability to cheaper feedstocks (Nomanbhay and Ong, 2017).

Rice husk is generally considered as agricultural waste, mainly used for energy generation, and is used as raw material to develop technological products, such as high purity silica ash. Rice husk could be an efficient solid fuel in various industries for burning in a boiler to produce steam, thus conserving both energy and resources (Della *et al*., 2002; Kalapathy *et al*., 2002). Burning rice husk in air always produces rice husk ash (RHA) which is typically viewed as agricultural waste (Saceda and Leon, 2011; Chen *et al*., 2013). RHA is largely composed of silica (87-99%) with small amounts of inorganic compounds, and has been widely used to adsorb organic dye, such as malachite green, and inorganic metal such as Pd^{2+} and Cu^{2+} metal ions due to their well-ordered structure and high surface area (Vinu and Binitha, 2020).

RHA has been used to synthesize lithium silicate by solid-state reaction as a biodiesel catalyst (Chen *et al*., 2013), producing Li_2SiO_3 and Li_4SiO_4. The product Li_2SiO_3 can better perform as a catalyst by transesterification in biodiesel than Li_4SiO_4 by providing a higher yield percentage of fatty acid methyl ester (FAME) (Dai *et al*., 2016). Ca_2SiO_4 has been synthesized using silica from RHA with a hydrothermal-thermal conversion reaction (Mansha *et al*., 2011), and the product shows efficient performance as a biodiesel catalyst. It is suggested that silica from RHA could be a precursor to synthesize an effective biodiesel catalyst. Three alkali metals (Li, Na, and K), supported by rice husk silica as a biodiesel catalyst, were stirred and heated, and then dehydrated and calcined at 500°C for 3 hrs. All the alkali metal silicate under the process was an efficient biodiesel catalyst with a very high methyl ester content. (Hindryawati *et al*., 2014). However, the process consumed reaction and heating times for calcination.

In this study, we attempted to adopt a method to reduce time and electricity to synthesis lithium and potassium silicates. The hydrothermal-microwave method resulted in quality and quantity advantages over the conventional heating technique. The RHA and alkali hydroxides of lithium and potassium were the precursors to synthesize lithium and potassium silicates by the hydrothermal-microwave method. Both alkali silicates were used as a catalyst in the transesterification reaction to prepare biodiesel. The catalysts were characterized by Fourier Transform Infrared Spectroscopy (FTIR), Resonance Raman (RR) spectroscopy, X-ray diffractometry (XRD), and scanning electron microscopy (SEM). FAME was determined by gas chromatography (GC).

Materials and Methods

Materials

Lithium hydroxide, potassium hydroxide, and methanol were AR grade and palm oil was of commercial quality. RHA was the residue from the biomass stove at Maejo University, Chiang Mai, Thailand.

RHA Analysis

RHA was sieved using a 150 micron mesh and the chemical evaluated by composition by X-ray fluorescence (PANalytical) which showed contents of silica (SiO_2)-88.6, calcium oxide (CaO)-3.05%, aluminum oxide (Al_2O_3)-2.71%, potassium oxide (K_2O)-2.62%, zirconium dioxide (ZrO_2)-1.41%, ferric oxide (Fe_2O_3)-0.78%, magnesium oxide (MgO)-0.15%, titanium dioxide (TiO_2)-0.10%, and a loss of ignition -0.58%.

Preparation of Alkali Metal Silicate

10 g of RHA were mixed with 300 mL of 10 M lithium hydroxide (LiOH) and potassium hydroxide (KOH) in a hydrothermal tube. The mixtures were heated by microwave at 600 and 800

watts for 5 and 10 min. The suspension was filtered through filter paper #1 and dried at 180°C in a hot air oven.

Preparation of Transesterification in Palm Oil

100 g of palm oil was added to a three-necked, round bottom flask equipped with a reflux condenser on a heating mantle. 1.50 g of catalyst and 25 mL of methanol were stirred for 30 min. prior to mixing with palm oil. The mixture was then refluxed at 65-70°C for 3 h. The percentage of yield of biodiesel was calculated following (Birla *et al*., 2012; Maneerung *et al*., 2015) and the percentage of yield of fatty acid methyl ester (FAME) following (Dodds *et al*., 2005). See the following equation.

$$\text{Yield (\%)} = \frac{\text{weight of biodiesel}}{\text{weight of palm oil}} \text{x } 100.$$

$$\text{Yield of FAME (\%)} = \frac{\text{total peak area of methyl ester}}{\text{total peak area of fatty acid}} \text{ x } 100.$$

Characterization

Fourier Transform Infrared Spectrometer (Perkin Elmer, Spectrum RXI) was the tool used to investigate the formation of lithium and potassium silicates. The spectrum was measured over a range of 4,000-400 cm^{-1} with 4 cm^{-1} resolution and 16 scans. Resonance Raman (RR) spectrometry (Jobin Yvon Horiba T64000) was carried out using a triple monochromator at 50× microscope with a 532 nm exciting laser source in the range of 500-150 cm^{-1}. X-ray diffractometry (Rigaku mini flex II) confirmed the phase of lithium and potassium silicates. Scanning electron microscopy (JEOL, JSM 6335 F) was performed on all powder samples to study the shape of the products.

Biodiesel Analysis

The primary analysis was pH and viscosity at 40°C. The constant of capillary (Cannon) was 0.958 cSt/s. The Methyl ester group was analyzed by Fourier Transform Infrared Spectrometer. The free fatty acid methyl ester was carried out by gas chromatography with standard carbon C17.

GC Parameter

- Column HP5-MS 30 m × 0.250 mm × 0.25 μm,
- Max. temperature 350°C,
- Flow 73.9 mm/sec,
- Heat 230°C,
- MS detector,
- Flow 1.5 mm/min, and
- Make-up flow 20 mL/min.

Results and Discussion

Fourier Transform Infrared Spectrometer (FTIR) was used to investigate the interaction of silica, sodium, and potassium hydroxide to form lithium and potassium silicates (Figures 1 and 2, respectively). The major characteristic absorption bands at 468-632 cm^{-1} are attributed to the vibration of M^{+}-O bond.

The symmetric stretching of (Li)-O-Si-O-(Li) was at 468 cm^{-1} and (K)-O-Si-O-(K) at 461 cm^{-1} (Halasz *et al*., 2010). The stretching of O-Si-O shows at 1,006-1,012 cm^{-1} (Hindryawati *et al*., 2014). The asymmetric stretching of (H)-O-Si-O-(K) at 705-706 cm^{-1} was the vibration of the silanol

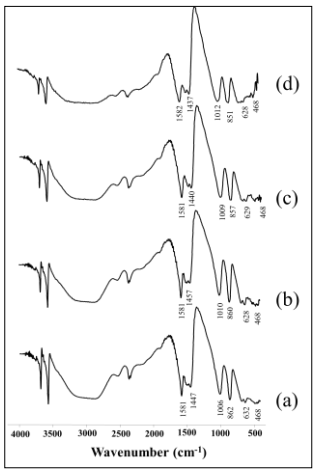

Figure 1. FTIR spectra of products synthesized from LiOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800W-10 min

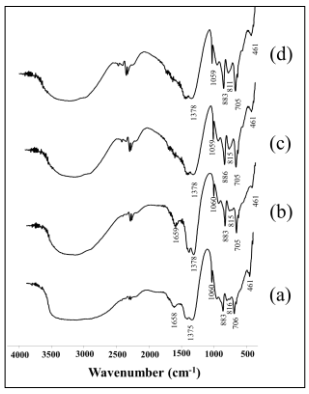

Figure 2. FTIR spectra of products synthesized from KOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

group in the silicate compound (Halasz *et al*., 2010; Coyle *et al*., 2020).

The appearance of the vibration of (M)O-Si-O(M) suggested that an interaction occurred between silica, lithium, and potassium hydroxide. The heating power at 600 watts showed a stronger band than 400 watts.

The products were then synthesized by lithium and potassium hydroxide, and heating power at 600 and 800 watts for 10 min. was characterized by Resonance Raman Spectrometer for studying the metal-ligand mode, as shown in Figures 3 and 4.

The three low-frequency regions, and RR bands at approximately 390-392 cm^{-1} were assigned to symmetric stretching of Si-O-Si. The bands at 212-367 were Li-O stretching of lithium silicate bonds (Richet *et al*., 1996). In the alkali and silicate in Figure 4, only K-O-Si was seen at 226 cm^{-1} band (Richet *et al*., 1996; Calahoo *et al*., 2016). The RR results supported the formation of potassium and silicate which corresponded with the FTIR results. The RR vibration of Li-O-Si occurred at Raman shift 296-297 cm^{-1} (Richet *et al*., 1996).

An X-ray diffractometer was used to confirm the phase of lithium and potassium silicates, as shown in the results in Figures 5 and 6. The lithium silicate was JCPDS no.00-030-0766. At 800 watts for 10 min. the intensity was higher and the peak sharper than 5 min. An X-ray diffractometer of the results from potassium hydroxide at a heating power of 800 watts was assigned to potassium silicate with JCPDS no.00-031-1076.

Figure 7 showed the SEM images of the products Li_2SiO_3 at 600 and 800 watts for 5 and 10

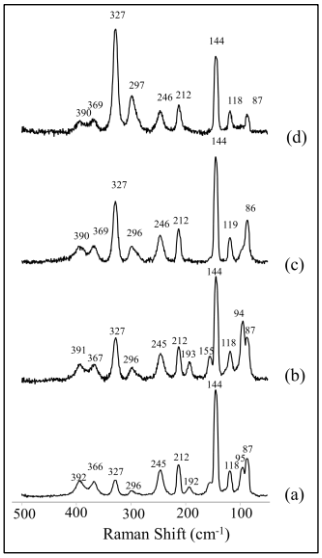

Figure 3. RR spectra of products synthesized from LiOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

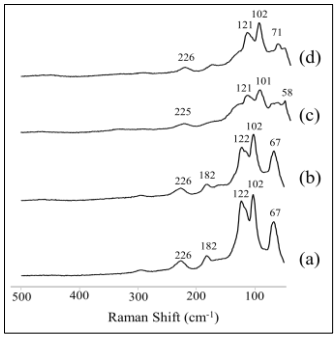

Figure 4. RR spectra of products synthesized from KOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

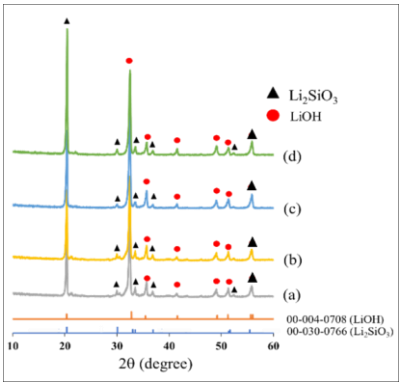

Figure 5. X-ray diffraction of products synthesized from LiOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

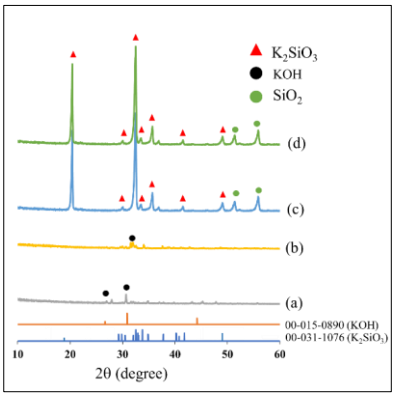

Figure 6. X-ray diffraction of products synthesized from KOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

min. The Figures 7(a) and 7(c) shows the small particles of Li_2SiO_3 catalyst. Figures 7(b) and 7(d), Li_2SiO_3 was synthesized for 10 min showed larger crystals than the product synthesized for 5 min.

The SEM images of the K_2SiO_3 synthesis using 600 and 800 watts for 5 and 10 min. are shown in Figure 8, while Figures 8(a) and 8(b) show melted crystal and observable clear products. The products synthesized for 10 min. were higher crystalline than for 5 min. All the products were synthesized using a microwave heater for 5 and 10 min. which can produce small size crystals from the effect of microwave heating (Rao *et al*.,1999).

Biodiesel analysis using FTIR, the methyl ester group of biodiesels, is shown in Figures 9 and 10.

The characteristic vibrations of the products from lithium and potassium silicate catalysts found C=O stretching of ester at 1742-1744 cm^{-1} (Ault and Pomeroy, 2012) and the asymmetric stretching of C-O-C in ester bond at 1,168-1,170 cm^{-1} (Zhang *et al*., 2013) was present in both palm oil and biodiesel. The splitting of methyl band of palm oil was 1,434-1,438 cm^{-1} of the asymmetric stretching of methyl ester of biodiesel (Gog *et al*., 2012).

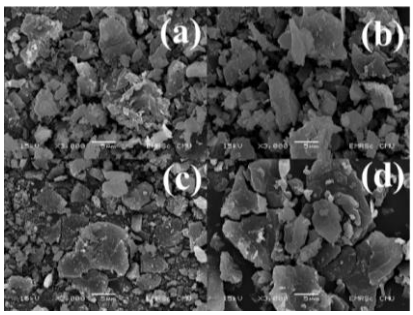

Figure 7. SEM of products synthesized from LiOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

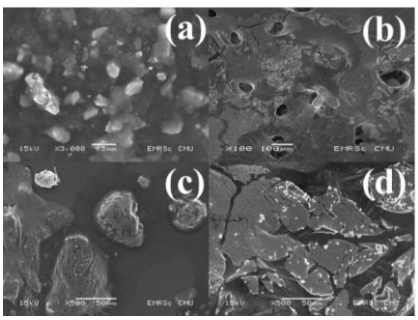

Figure 8. SEM of products synthesized from KOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

In this research, analysis using GC was carried out to identify the methyl ester which resulted in biodiesel production. The results of GC analysis on the produced methyl ester indicated that there is a substance of methyl hexadecanoate in methyl ester with a retention time of 35.36, as followed by Velloso *et al*. (2013) and Maulidiyah *et al*. (2017).

Biodiesel products from Li_2SiO_3 catalyst at 800 watts for 10 min. were the highest percentage by weight of methyl ester at 88.91% and percentage by weight of fatty acid methyl ester (FAME) at 22.20%. The biodiesel products from K_2SiO_3 catalyst were at 800 watts for 5 min.

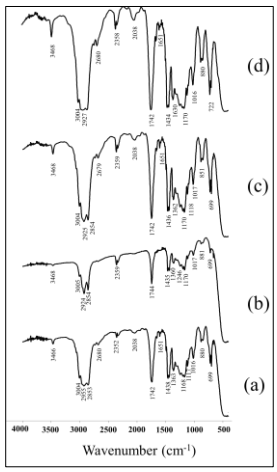

Figure 9. FTIR of biodiesel products synthesized from LiOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

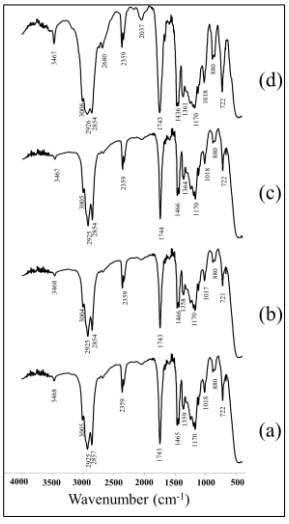

Figure 10. FTIR of biodiesel products synthesized from KOH by microwave for (a) 600 W-5 min (b) 600 W-10 min (c) 800 W-5 min (d) 800 W-10 min

Table 1. **Percentage of FAME yield and primary analysis of biodiesel**

Amount of catalyst	% Yield biodiesel (W/W)	% Yield FAME (W/W)	Viscosity (cSt)
1.5 g of Li_2SiO_3-600W-5min	35.34	21.82	12.88
1.5 g of Li_2SiO_3-600W-10min	42.28	19.04	12.60
1.5 g of Li_2SiO_3-800W5min	44.01	18.17	13.94
1.5 g of Li_2SiO_3-800W-10min	44.68	22.20	13.19
1.5 g of K_2SiO_3-600W-5min	37.31	69.30	4.92
1.5 g of K_2SiO_3-600W-10min	36.09	74.50	4.75
1.5 g of K_2SiO_3-800W-5min	37.59	88.40	4.74
1.5 g of K_2SiO_3-800W-10min	58.76	85.30	4.458

The primary analysis of biodiesel products was pH and viscosity at 40°C, as shown in Table 1 above. The pH and viscosity of each biodiesel were in the range of the European biodiesel standard (EN) 14214 specifications.

Lithium and potassium silicate catalysts could provide a biodiesel yield of 44.68% and 58.76%, respectively. The maximum yield was at 800 watts of microwave heat.

The types of alkali hydroxide and watts of microwave heating were affected by parameters in the role of the yield of the biodiesel catalyst. The fatty acid methyl ester (FAME) content by GC with Li_2SiO_3 and K_2SiO_3 catalyst was 22.20% and 88.20%, respectively. K_2SiO_3 gave a higher FAME than Li_2SiO_3 due to the opportunity contraction between the catalyst and reaction, which directly affected reaction speed.

The Li^+ cation with smaller atomic size (compared with K^+) bonds strongly with SiO_2 and forms a stable compound that makes the catalyst's performance less than that of potassium (Castro *et al*., 2012; Hindryawati *et al*., 2014). The 800-watts heating power of the microwave oven for 10 min. provided the highest percentage of free FAME. The type of alkali was a significant parameter for the percentage of yield of biodiesel and FAME.

The microwave effect of the synthesized products that could microwave interactively with materials has been based on concepts of dielectric heating and the resonance absorption due to the crystal shape and purity of the compound. Therefore, the hydrothermal microwave method was the potential method to synthesize lithium and potassium silicates as biodiesel catalysts.

Conclusions

Lithium and potassium silicates could be synthesized by microwave-assisted method using rice husk ash, lithium hydroxide, and potassium hydroxide as a precursor to heating at 800 watts for 10 min. and 800 watts for 5 min.

The bonding formation of lithium and potassium silicates was confirmed by Fourier Transform Infrared Spectrometer, resonance Raman spectrometer, X-ray diffractometer, and transmission electron microscope.

Lithium and potassium silicates could be used as a catalyst in biodiesel production from palm oil. The percentage yields FAME for lithium silicate catalyst and potassium silicate were 22.20% and 88.40%, respectively.

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