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Synthesis of alumina from aluminium can waste to be applied as catalyst support for biodiesel production

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Abstract. Abundant of aluminum beverage cans are normally discarded after use have caused considerable land pollution and environmental problems. This research is therefore aimed to synthesize alumina from aluminum can waste which is one of the most common kind of waste. The objective of this research is to synthesize and characterize alumina produced from aluminum can waste, and to be applied as catalyst support in the biodiesel production. In this study, the alumina from aluminum can waste was produced via Sol-gel method by varying the aging time. Characterization of alumina was performed by using FTIR, XRD, BET, and SEM-EDX. The synthesized alumina was used as catalyst support for potassium nitrate catalyst to be applied in biodiesel production by using transesterification reaction of cooking oil. The biodiesel produced was analyzed by using gas chromatography-mass spectrometry (GCMS) and FTIR. The experimental results revealed that the alumina powder synthesized at room temperature have high surface area which are suitable to be used as catalyst support of producing biodiesel. In conclusion, it has been demonstrated that it is possible to produce alumina from aluminum can waste that can be used as catalyst support for biodiesel production. From the GCMS and FTIR results, it was proven that biodiesel is produced.

1. Introduction

Waste management in Malaysia has become a challenging undertaking in recent years due to rapid economic development and population growth, land scarcity and poor infrastructure and expertise. Some of the current trends in managing solid waste include reengineered process which helps to reduce quantities of emissions, strategies to maximize current levels of waste utilization and development of high value products [1]. Aluminum found in solid waste includes can, wrapping foil, car engines and body parts, while food packaging and automobile production are being two of many possible sources in municipal solid wastes. Recycling aluminium can waste save precious natural resources, energy, time and money [2].

Alumina is one of the most important ceramic oxides which have vast technological and industrial applications. It is used in many applications such as to contain molten metal, as insulators in spark plugs, and others applications such as dental and medical use [3]. Alumina has also been applied as catalyst, catalyst support and adsorbent. For example, alumina is used to support calcium oxide (CaO) base catalyst for the transesterification of palm oil in biodiesel production [4]. Activated alumina also act as adsorbent in removal of nickel ions from industrial plating effluents.



Various sources can be used to produce biodiesel such as from waste cooking oil, plastics, and vegetable oil, animal fats. For example, by using waste cooking oil that has many disposal problems like water and soil pollution, human health concern; it can be used as an effective and cost efficient feedstock for biodiesel production [5]. Preparation of biodiesel from cooking oil and methanol by transesterification reaction using the heterogeneous catalyst will increase the reaction yield and minimize the methanol molar ratio because huge amount methanol required for reaction will involve an additional cost for the separation of biodiesel [6]. Thus, this research is aimed to utilize the aluminum can waste from the municipal solid waste (MSW) to produce alumina and to be applied as catalyst support for biodiesel production.

2. Materials and Method

2.1. Materials

Aluminum can waste was collected from Mercu Resolution Sdn Bhd landfill site located in Sungai Ikan, Terengganu. The chemicals potassium hydroxide (KOH) ($\geq 85\%$) and sulfuric acid (H_2SO_4) ($\geq 98\%$) were purchased from Sigma-Aldrich. Deionized water employed in this study was from Millipore Elix 5, USA. Materials needed for the production of biodiesel were cooking oil that was obtained from local market; methanol ($\geq 99.8\%$) and potassium nitrate ($\geq 99.0\%$) were purchased from Sigma-Aldrich.

2.2. Methods

Methods used in this study were divided into three stages where the first stage is synthesizing γ -alumina from can waste, second stage involved on the incorporation of the synthesized alumina as catalyst support in KNO_3 and the third stage is the application of the synthesized catalyst $\text{KNO}_3/\text{Al}_2\text{O}_3$ in the biodiesel production.

2.2.1 Synthesis of mesoporous γ -alumina

The mesoporous γ -alumina was synthesized via sol-gel method. Aluminum can waste was cut into small pieces and dissolved in potassium hydroxide solution. Then, the mixture was filtered and adjusted the pH at 9.0 using sulfuric acid. The solution was stirred until it became gel-like. The mixture was aged by fixing the reaction temperature at room temperature and varying the aging time at 6 hours and 24 hours. After that, the mixture was separated using centrifugation. Finally, the aluminum hydroxide gel was put into the oven at 80°C for half day for drying process and calcined into furnace at 500°C . Alumina synthesized at reaction temperature (room temperature) and aging times (6 – 24) hours was denoted as AL-6-30 and AL-24-30 respectively. The physicochemical properties of the synthesized γ -alumina were investigated by using different characterization techniques. γ -alumina was characterized by using X-Ray Diffraction (XRD) (RIGAKU MINIFLEX II X-ray diffractometer) to determine the crystalline size, structure and phase identification, Fourier Transform Infrared Spectroscopy (FTIR) (Nicolet iS5 Model Spectrum100) to obtain the functional group of sample powders while Brunauer-Emmett-Teller (BET) to measure the specific surface area, pore size, pore volume and pore size distribution of the sample powders while Scanning Electron Microscopy (SEM) (Benchtop Scanning Electron Microscope TM3030Plus) was carried out to inspect the surface morphology whereas Energy-dispersive X-ray (EDX) (SwiftED3000) was used to determine the elemental compositions that present in the catalyst.

2.2.2 Synthesis of catalyst for biodiesel production

The catalyst that was synthesized in this study was KNO_3 on alumina ($\text{KNO}_3/\text{Al}_2\text{O}_3$) which is also known as base heterogeneous catalyst. The aqueous potassium nitrate loaded on alumina and stirred for 4 hours at 85°C and 250 rpm by using impregnation method. The purpose of stirring is to make sure the catalyst well distributed on support. The catalyst was further dried for 16 hours at 120°C . Prior to each reaction, the catalyst was calcined at 500°C for 5 hours in furnace. The catalyst prepared to KNO_3 loading amounts of 35 wt%, was designated as 35% $\text{KNO}_3/\text{Al}_2\text{O}_3$. The characterization of catalyst was investigated by using FTIR, XRD, SEM, EDX, and BET.

2.3. Application of alumina as catalyst support in biodiesel production

The method of conducting the production of biodiesel was based on Zabeti et al. [4] with some modifications [4]. Transesterification reaction was performed in three neck glass batch type reactor. 150 mL of cooking oil and 56.80 mL of methanol were poured into the flask. Then, the reaction was initiated by adding catalyst with a desired amount. The mixture was heated at reaction temperature of 65 °C and stirred with an optimum speed of 600 rpm for 5 hours. After the reaction completed, the catalyst was separated by centrifuged for 10 min. The liquid was put into separating funnel and left it overnight until the liquid separated into two phases. Biodiesel was analyzed using the result by Gas Chromatography-Mass Spectrometry (GC-MS) and Fourier Transform Infrared Spectroscopy (FTIR).

3. Results and discussion

3.1. Characterization of synthesized alumina

Fourier transform infrared (FTIR) analysis of the alumina was done in the wavenumber ranging from 400 to 4000 cm^{-1} . The FTIR spectra of AL-6-30 and AL-24-30 are as indicated in figure 1. In this section, the effect of aging time on the physicochemical of synthesized alumina is studied through the characterization of powder. FTIR spectra of the synthesized samples have been shown in Figure 4.1. The appearance of peaks indicates the presence of a better crystallized phase. The bands of AL-6-30 were around 613 cm^{-1} and 1102 cm^{-1} while the bands of AL-24-30 were around 614 cm^{-1} and 1104 cm^{-1} . The band around 613 cm^{-1} for sample AL-6-30 and the band at 614 cm^{-1} for sample AL-24-30 were assigned to $\nu\text{-AlO}_6$. While the band around 1102 cm^{-1} for sample AL-6-30 and 1104 cm^{-1} for sample AL-24-30 were assigned to Al-O-Al. The peak corresponding to 1101 cm^{-1} is assigned to Al-O-Al symmetric bending stretching vibrations. The peaks in the region of 500–750 cm^{-1} are assigned to $\nu\text{-AlO}_6$, whereas the shoulder at 890 and the line at 800 cm^{-1} are assigned to $\nu\text{-AlO}_4$ [7]. There is no peak around 3500 cm^{-1} and 1600 cm^{-1} which shows that both samples of synthesized $\gamma\text{-Al}_2\text{O}_3$ are fully dried from water. This is supported by Rajaeiyan and Bagheri-Mohagheghi (2013) where intense bands centered around 3500 cm^{-1} and 1600 cm^{-1} are assigned to O–H stretching and bending modes of adsorbed water due to high tendency of $\gamma\text{-Al}_2\text{O}_3$ for adsorbing water [8]. Based on the graph, 6 h and 24 hours aging time gave same intensity of Al-OH at peak of 614 cm^{-1} . Both of aging time FTIR analysis shows that larger intensity of aluminum hydroxide and aluminum oxide in the sample.

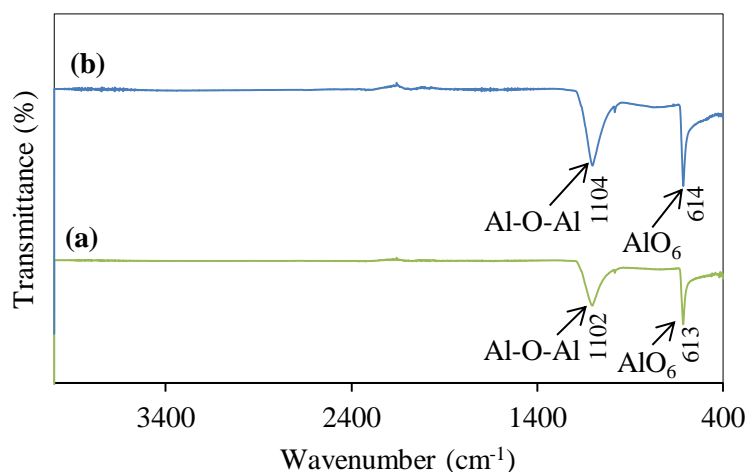


Figure 1. FTIR spectra comparing the effect of aging time on synthesis of alumina; (a) AL-6-30 (b) AL-24-30

Figure 2 shows the X-ray diffraction patterns of the synthesized alumina AL-6-30, AL-24-30 and AL-commercial. For both AL-6-30 and AL-24-30 all the observed peaks were identified as aluminum oxide, or alumina (Al_2O_3) crystalline structure. The XRD patterns of mesoporous alumina in wide angle region

suggested the phase of γ - Al_2O_3 at 2θ are 30.2° , 31.2° with broad peaks, 43.0° , and 67.0° [JCPDS card 10-0425] [9]. In the XRD results, there were boehmite, corundum and gamma alumina identified in the synthesis of alumina. Boehmite is an aluminum oxide hydroxide (γ - $\text{AlO}(\text{OH})$) mineral, a component of the aluminum ore bauxite. It is dimorphous with diaspore. The structural collapse of boehmite occurs, after hydrogen transfers and water extraction [10]. Then, through an aluminum migration process, the γ -alumina characteristics appear. Corundum is a crystalline form of aluminum oxide, typically containing traces of iron, titanium, vanadium and chromium [11]. The XRD patterns of synthesized alumina AL-6-30 and AL-24-30 in small angle, which is 1 – 10° have no XRD peaks. This implies that this mesoporous alumina has no long-range order pore structure. For alumina synthesized at room temperature, as indicated in Figure 2, they appeared to be less crystalline in structure with broader peaks. AL-6-30 had higher intensity for the observed peaks compared to AL-24-30 which showed that AL-6-30 had more crystalline structure. For AL-commercial, the observed peaks were identified as an aluminum oxide, or alumina (Al_2O_3) crystalline structure. The peaks of Al_2O_3 were at 37.2° , 45.92° , 60.48° , and 66.7° .

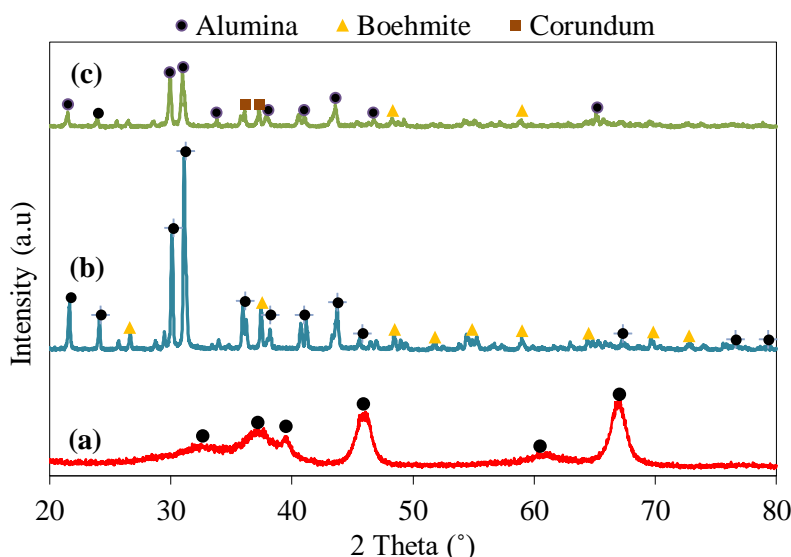


Figure 2. XRD pattern comparing the effect of reaction time on synthesis of alumina; (a) AL-commercial (b) AL-6-30 and (c) AL-24-30

Figure 3 shows the SEM-EDX spectra of AL-6-30, AL-24-30 and AL-commercial with average weight and atomic percentage of elements that present on the samples. From Figures 3(a) and 3(b), it can be observed that the solid morphologies for both AL-6-30 and AL-24-30 were similar, with heterogeneous particle sizes. Both of the synthesized alumina had non-spherical shape. As it is observed, some particles with disordered shape were obtained by joining very small crystallites during the calcination [12]. AL-6-30 and AL-24-30 appeared to be more disordered compared to the surface morphology of AL-commercial. The red circle in the figures represent the spot where EDX sample were taken. Analyzing the spectra for AL-6-30 and AL-24-30 in (a) and (b) it was noted that the powders are mainly composed of oxygen, aluminum, sulphur and potassium. Oxygen has the highest weight and atomic percentage in all samples followed by aluminum. In Figure 3(b) for AL-24-30 shows that aluminum was produced higher when used 24 h aging time. The presence of potassium in AL-6-30 and AL-24-30 was due to the usage of potassium hydroxide solution in dissolving the aluminum waste cans. There were traces of sulphur in the spectra of Figure 3 as sulphuric acid was used for pH adjusting. These traces of potassium and sulphur can be eliminated by having sufficient rinsing with deionized water during the formation of aluminum hydroxide gel. Regardless the reaction time, all synthesized aluminas had similar trend in the composition for each element.

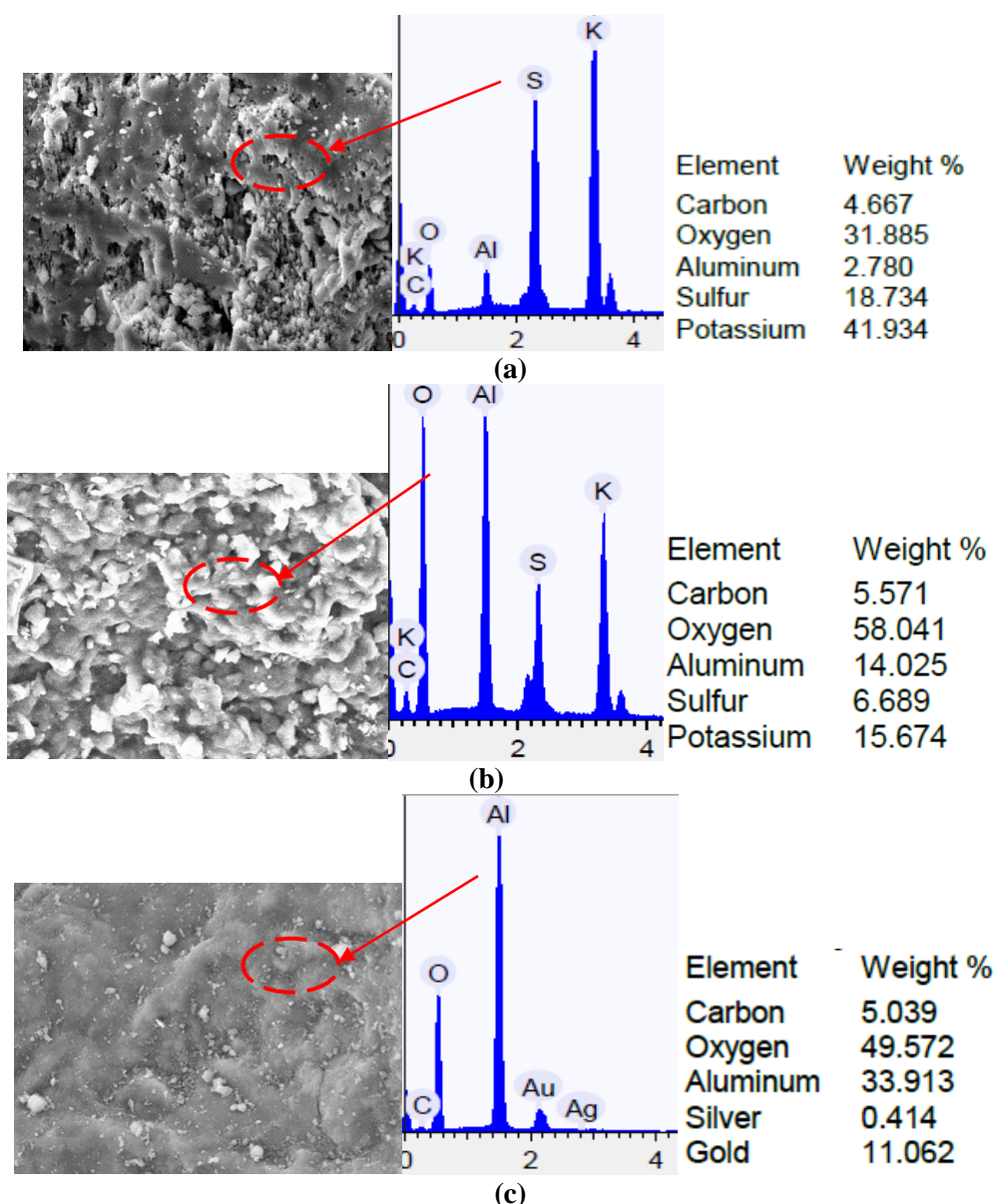


Figure 3. EDX comparing the effect of reaction time on synthesis of Alumina; (a) AL-6-30 (b) AL-24-30 (c) AL-commercial

From Table 1, it is noted that the Specific Surface Area (SSA) of AL-6-30 and AL-24-30 were 6.5264 m²/g and 4.3072 m²/g, showing low surface areas. The pore volume for AL-6-30 was 0.0125 cm³/g while the pore volume for AL-24-30 was 0.0090 cm³/g. The AL-commercial has SSA of 176.6364 m²/g and pore volume of 0.3988 cm³/g. The AL-commercial had high surface area compared to synthesized alumina. The surface area analysis is consistent with the XRD analysis. AL-6-30 and AL-24-30 which had less crystalline structure in XRD had lower surface areas. This indicates that aging time or reaction time is an important factor to obtain high surface area of the products. It has been reported that porous materials having a surface area value above 100 m²/g is considered having a high surface area and is a potential material for catalyst support [13]. But with low surface area still can used as a catalyst support for biodiesel production.

Table 1. Summary of specific surface area (SSA), pore volume and adsorption average pore diameter of AL-6-30, AL-24-30 and AL-commercial by BET comparing the effect of reaction time on synthesis of Alumina

Sample	Specific Surface Area (SSA) ^a , m ² /g	Pore Volume ^b , cm ³ /g	Adsorption Pore Diameter (4V/A by BET), nm	Average Pore Diameter (4V/A by BET), nm	Crystalline Size ^c , nm
AL-6-30	6.5264	0.0125	7.4083		65.27
AL-24-30	4.3072	0.0090	9.2205		42.54
AL-commercial	176.6364	0.3988	8.9207		5.38

^a Calculated by Brunauer-Emmett-Teller method

^b Determined by Barret-Joyner-Halenda (BJH) method

^c Determined by X-ray peak in XRD using Scherrer formula

3.2. Characterization of catalyst KNO₃/Al₂O₃

Figure 4 shows the FTIR spectrum of the KNO₃/AL-commercial and KNO₃/AL-24-30 catalyst. All samples showed an absorption band at around 1360 cm⁻¹ to 1390 cm⁻¹ that could be attributed to the vibration of N-O of KNO₃, while this N-O absorption band was not observed on alumina without loaded KNO₃. Further, the intensity of 1384 cm⁻¹ band grew as the amount of loaded KNO₃ rose [14]. Also, physically adsorbed water on the solid base was observed at around 1636 cm⁻¹ assigned to the bending mode of O-H. With the increase of calcination temperature, the O-H absorption band became weaker and, finally disappeared. On the surface of fully hydroxylated alumina K⁺ ion replaced the protons of isolated hydroxyl groups to form Al-O-K groups which were considered to be the active basic sites. There are no bands ascribed to NO₂ (1550, 1320 cm⁻¹), were detected, demonstrating that the resultant of KNO₃ decomposition was K₂O instead of KNO₃.

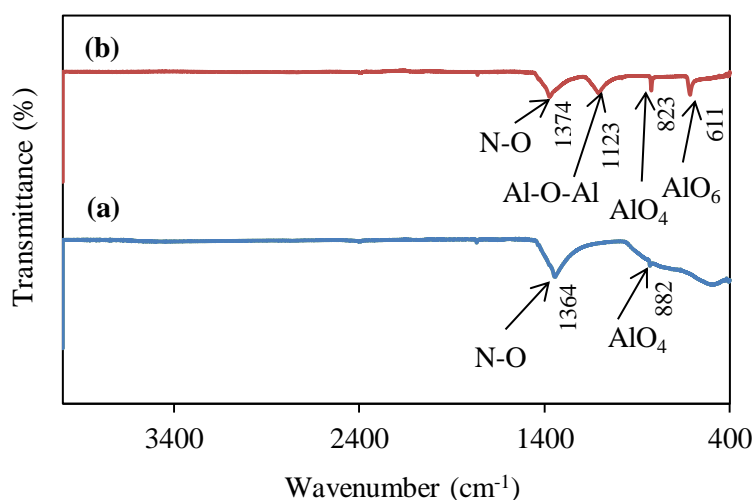


Figure 4. FTIR spectra of (a) KNO₃/AL-commercial (b) KNO₃/AL-24-30

Figure 5 shows the XRD patterns of KNO₃/AL-commercial and KNO₃/AL-24-30 samples. According to Wenlei Xie, 2006 on the patterns of 35% KNO₃/Al₂O₃ sample calcined at 773 K, new diffraction lines appeared with 2 θ of 31°, 39°, 55° and 62°, which were assigned to K₂O. This K₂O phase was probably the cause of the high catalytic activity and basicity of the catalysts because a close relationship was seen between the K₂O phase that appeared and the basicity or the catalytic activity and indicating the good dispersion of KNO₃ on alumina. As the amount of loaded KNO₃ exceeded, the characteristic XRD peaks of KNO₃ appeared on the XRD pattern and their intensities grew with increasing amount of loaded KNO₃, suggesting that a residual phase of KNO₃ remained on the composite. In the XRD results, there

were corundum, potassium, potassium nitrate, potassium dinitramide and niter identified in the synthesis of $\text{KNO}_3/\text{Al}_2\text{O}_3$. The present of corundum which is a crystalline form of aluminum oxide means there has alumina in the catalyst. Potassium dinitramide (KN_3O_4) contains excess oxygen and, therefore, may be also used as an oxidizer in various compositions [15]. Niter is the mineral form of potassium nitrate, KNO_3 also known as saltpeter.

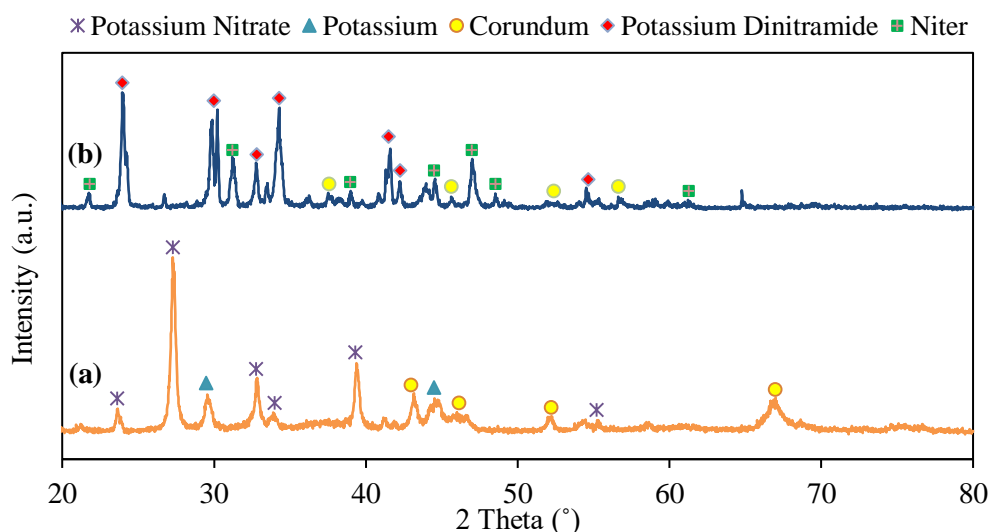


Figure 5. XRD pattern of (a) KNO_3/AL -commercial (b) KNO_3/AL -24-30

3.3. Characterization of catalyst $\text{KNO}_3/\text{Al}_2\text{O}_3$

Figure 6 shows the FTIR analysis for the comparisons of biodiesel by using catalyst KNO_3/AL -commercial and KNO_3/AL -24-30. Both spectra show similar bands. Complete depletion of the band near 2900 cm^{-1} , assigned to the alkene C-H stretch is observed for the deposits. IR bands in the region of $1160\text{--}1200\text{ cm}^{-1}$ for O- CH_3 stretching, clearly demonstrated the transformation of cooking oils into biodiesel [16]. Bands were around 1137 cm^{-1} and 1457 cm^{-1} were assigned to (C-O vibrations) which is the additional ester bands. From the spectra, the band was around 1734 cm^{-1} for both sample were assigned to $\nu(\text{C}=\text{O})$. According to Aryee et al. [17], band observed in the $1750\text{--}1720\text{ cm}^{-1}$ range is attributed to the ester $\nu(\text{C}=\text{O})$ absorption of triacylglycerols and is indicative of the extraction of a small amount of oil into the methanol phase [17]. IR bands in the region of $1370\text{--}1400\text{ cm}^{-1}$ are for O- CH_2 groups in glycerol contain of triglycerides, diglycerides, and monoglycerides [16].

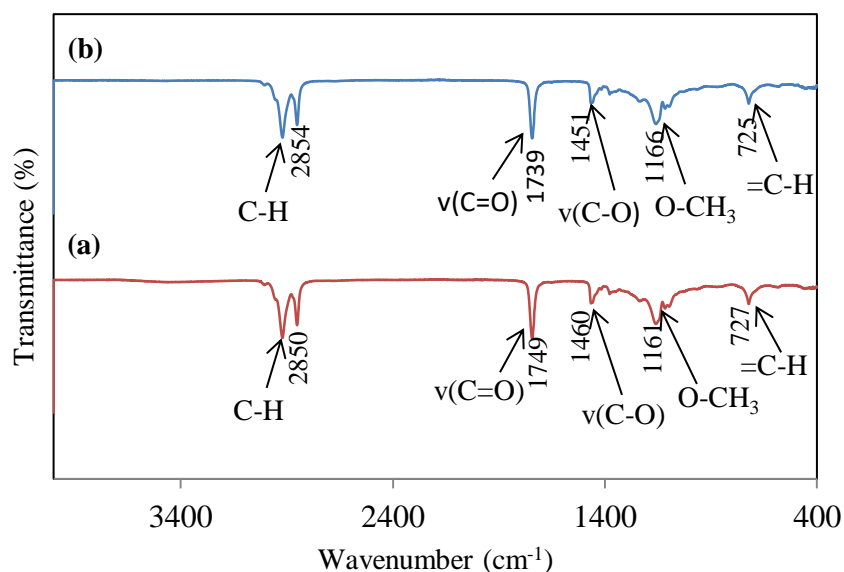
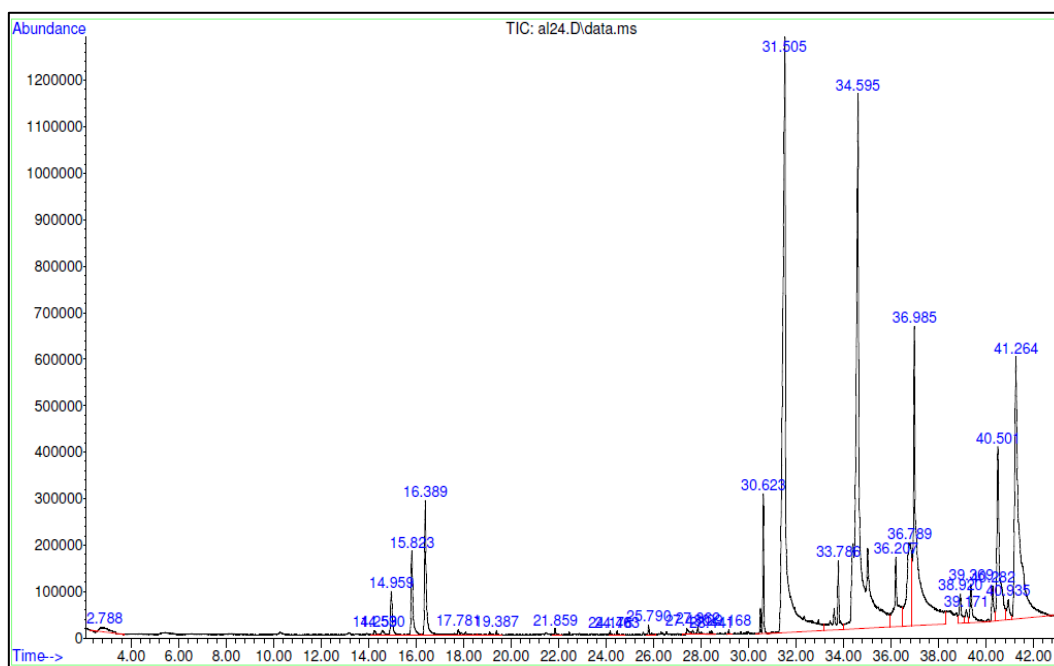
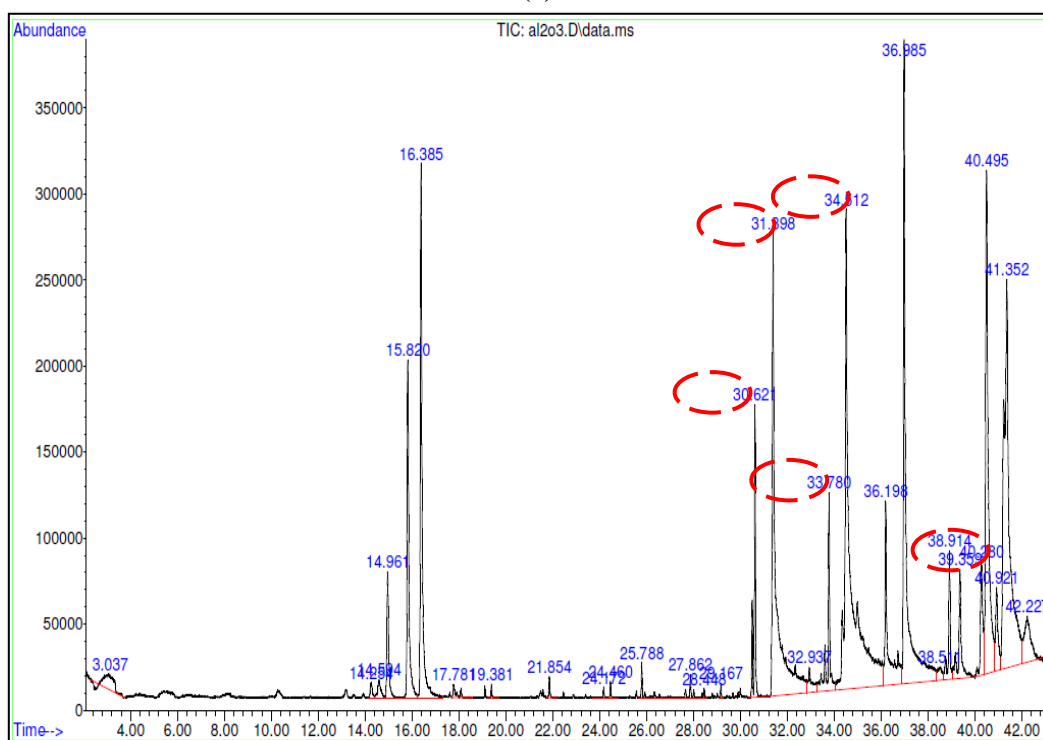


Figure 6. FTIR spectra of biodiesel by using (a) KNO_3/AL -commercial (b) KNO_3/AL -24-30

In this study, GC has been used to identify methyl esters according to their retention time which shows the mass spectrum in Figure 7(a) for biodiesel produced using catalyst KNO_3/AL -24-30 and Figure 7(b) for biodiesel produced using catalyst KNO_3/AL -commercial. Both figures show the similar mass spectrum of methyl ester in producing biodiesel from cooking oil. The red circle in the figures represent the peak of components in biodiesel. The chemical composition of biodiesel carried out using the GC-MS analysis led to the identification of five major components. From Figure 7(a) the identified components of biodiesel are at retention time 33.788 which is 9-Octadecenoic acid (E), (24.93%) represent the main constituent. n-Hexadecanoic acid, (22.67%) was the second major constituent at retention time 31.504 detected followed by 9-Octadecenoic acid (Z) methyl ester (1.96%) at retention time 33.788, Hexadecanoic acid, methyl ester (1.78%) at retention time 30.622 and 9,12-Octadecadienoic acid (Z,Z) (0.73%) at retention time 40.281. From Figure 7(b) shows that Oleic acid, 14-Pentadecenoic acid (17.96%) represent the main constituent at retention time 34.51, n-Hexadecanoic acid (11.35%) was the second major constituent at retention time 31.397 detected followed by Hexadecanoic acid methyl ester (2.78%) at retention time 30.622, 9-Octadecenoic acid (E), methyl ester (2.58%) at retention time 33.783 and 9,12-Octadecadienoic acid (Z,Z) (1.73%) and retention time 40.281.



(a)



(b)

Figure 7. GC-MS analysis on biodiesel by using catalyst; (a) $\text{KNO}_3/\text{AL-24-30}$
(b) $\text{KNO}_3/\text{AL-commercial}$

4. Conclusion

The experimental results show that γ -alumina was successfully prepared from aluminum can waste at different reaction time which are 6 hours and 24 hours. The result was proven through the characterization of alumina by FTIR, XRD, SEM, EDX and BET analyses. According to the mechanism, a coherent skeleton of γ -alumina inherited from the original boehmite (AlOOH) and corundum as the crystallized alumina also have in the alumina sample. Based on XRD and BET analysis results, alumina synthesized at room temperature with reaction time of 24 hours (AL-24-30) was found to have the best condition compared to alumina with reaction time of 6 hours (AL-6-30). From the analysis, we decided to use AL-24-30 as a catalyst support for biodiesel production.

For the biodiesel production, from the FTIR analysis shows that the band and spectra for biodiesel is clearly demonstrated the transformation of cooking oils into biodiesel. In addition, results from GC-MS analysis have proven that biodiesel components have been produced. Overall, this study indicates that the aluminum can waste are able to be converted to alumina and then can be further used as a catalyst support for biodiesel production.

Acknowledgement

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