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Low- Calcination Temperature to Synthesize α -Alumina From Aluminium Waste Can Using Sol-Gel Method

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Abstract. Many countries around the world are facing issues in managing solid waste materials; most of these wastes such as aluminium can are deposited to the landfills, leading to environmental pollution. Recycling is considered as an effective technique to manage the aluminium can waste since it can provide benefits in terms of energy savings, reduce volumes of waste and cost-effectiveness. In this article, it was desired to turn the aluminium can waste into α -Alumina using sol-gel method. Alumina exists in many crystalline structures which degenerate to the most stable hexagonal α -phase at high temperatures. α -Alumina (α -Al₂O₃) is the most stable crystalline structure widely used and studied as electronic packaging, corrosion resistance ceramics, high-temperature structural material, and translucent ceramics. FTIR, XRD, SEM-EDX, TGA, and BET were employed to investigate the properties of α -alumina. The experimental results obtained from this study demonstrates the possibility of producing alumina from an aluminium can waste with the exact surface area of 5.2105 m²/g, crystallite size at 132.50 nm and total weight loss of 2.71% at 900 °C calcination temperature.

1. Introduction

One of the most common challenges facing many countries around the world is the way solid materials can be controlled [1]. Recent trends in solid waste management have revealed novel and/or re-engineered processes, resulting in decreased waste emissions, innovative methods to minimize the present levels of disposable waste materials in the environment, and utilizing the waste in developing high-value products [2]. Nowadays, recycling aluminium can waste is considered as one of the best common practice to reduce production cost and minimize negative environmental effects [3]. The aluminium cans consist of 97.12% of Aluminium, 1.08% of Manganese, 0.863% of Magnesium, 0.589% Ferrous, 0.18% of Silicone, and 0.153% of other chemicals [3]. Besides, aluminium recycling has useful advantages such as requiring only 5% of energy to produce aluminium as well as its utilization in producing alumina powder [4]. Alumina is a very central ceramic oxide that is utilised in homes and industries. Moreover, it is utilized in high-temperature applications and microelectronics. Alumina is obtainable in many different crystalline structures such as η , γ , δ , θ , β , κ , χ , and α -Alumina due to the increasing temperature in calcination process of aluminium hydroxide (Al(OH)₃) [1]. α -Alumina is the steadiest form of alumina. It is the toughest and stiffest phase of oxide ceramics. Its excellent dielectric qualities, high hardness, good thermal properties, and refractoriness make it the desirable material for huge applications. The high purity alumina is highly preferable in both reducing and refractoriness atmospheres at 1925 °C [2]. It is used as a structural ceramic due to its mechanical and excellent thermal properties since alumina is produced at high calcination temperature [5]. Likewise, it has a high thermal shock resistance because of high average grain size which is about 200 μ m [6]. α -Alumina has a particle size distribution ranging from 30 to 32 nm which can be used in bio-ceramics applications [7]. In recent



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time, bio-ceramic is considered as one of the most interesting and promising implanting applications widely used. High purity alumina bio-ceramics were introduced as a substitute for surgical metal alloys for tooth implants and total hip prosthesis. Additionally, it is used in biocompatibility with other components in bio-glass-metal fibre composite [8]. Thus, this study focused on synthesizing α -Alumina (α -Al₂O₃) from an aluminium can waste using the sol-gel method at 900 °C calcination temperature.

2. Experimental Work

2.1. Materials and Methods

Waste of aluminium cans was retrieved from Mercu Resolution Sdn Bhd landfill site in Sungai Ikan, Terengganu. The required chemicals such as Sulfuric Acid (H₂SO₄) ($\geq 98\%$), and Potassium Hydroxide (KOH) ($\geq 85\%$) were procured from Sigma-Aldrich. Meanwhile, deionized water was obtained from Laboratory of the Faculty of Chemical & Natural Resources Engineering, Universiti Malaysia Pahang as dilution solvent and for preparation of stock solutions.

2.2. Synthesis of α -alumina

The procedure for conducting the synthesis of α -alumina was adapted from the Sol-gel method with some modifications according to the need of this study and prevailing environmental conditions [9]. Aluminium can waste were cut into minuscule sizes and then liquefied in 3 M potassium hydroxide (KOH) solution. Thereafter, the mixture was subjected to filtration, and pH of the available aluminate filtrate solution was attuned to 9 using 1 M of sulfuric acid (H₂SO₄). The solution obtained was then subjected to continuous stirring to form a gel-like mixture. Aging of the mixture was achieved by fixing the aging time at one day and adjusting the reaction temperature at 40°C and room temperature. The aluminium hydroxide gel was separated by centrifugation and washed severally using deionized water. Thereafter, the dried aluminium hydroxide was processed by placing it inside an oven at 80 °C for 12 h. The dried product was placed inside a furnace at 900 °C for 2 h calcination time.

2.3. Characterization of α -alumina

The physicochemical characteristics of synthesized α -alumina were investigated using different characterization techniques. Characterization of α -alumina was achieved using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), Energy-Dispersive X-ray (EDX), Brunauer-Emmett-Teller (BET), and Thermo-Gravimetric Analysis (TGA). The functional groups of alumina powders were determined using FTIR (Nicolet iS5 Model Spectrum100). The XRD (RIGAKU MINI FLEX II X-ray diffractometer) was conducted to verify the crystalline structure of the synthesized powder. SEM (Benchtop Scanning Electron Microscope TM3030Plus) was utilized to examine the surface morphology and particle size; whereas, EDX (SwiftED3000) was employed to determine the elemental compositions and atomic weights of the catalyst. BET (Micromeritics ASAP 2020) was done to figure out the pore volume, surface area, and pore size of the catalyst. The thermal resistance of alumina decomposition was determined using TGA (5E-MAC IV Proximate Analyze).

3. Results and Discussion

3.1. Synthesis of Alumina

α -Alumina was synthesized at the constant aging time (24 h) using different reaction temperatures (room temperature and 40 °C); calcination time of 2 h and calcination temperature of 900 °C. The synthesized alumina was denoted as AL-24-x, where x represents the reaction temperature. Alumina synthesized at 24 h aging time and reaction temperatures (room temperature and 40°C) was presented as AL-24-30 and AL-24-40, respectively.

3.2. Reaction Temperature Effect

FTIR spectra show the variations of reaction temperature on synthesised alumina AL-24-30 and AL-24-40 as presented in Figure 1. Both synthesized alumina have similar bands. The main peaks at 614 cm^{-1} indicate the complex Al-O stretching mode absorption (Xu et al., 2016) whereas the broad bands around 1120 cm^{-1} caused by the symmetrical deformation vibration of Al-O-H modes [10]. The spectrum of the synthesized alumina displayed sharp peaks at 614 cm^{-1} and 1120 cm^{-1} , these can be allotted to the existence of nano-structure alumina functional groups [11-12]. Hence, the increase the reaction temperature, the longer and sharper intensity of absorption peaks that attributed to the stretching and deformation vibration bands that showed the relationship of the alumina functional groups [12].

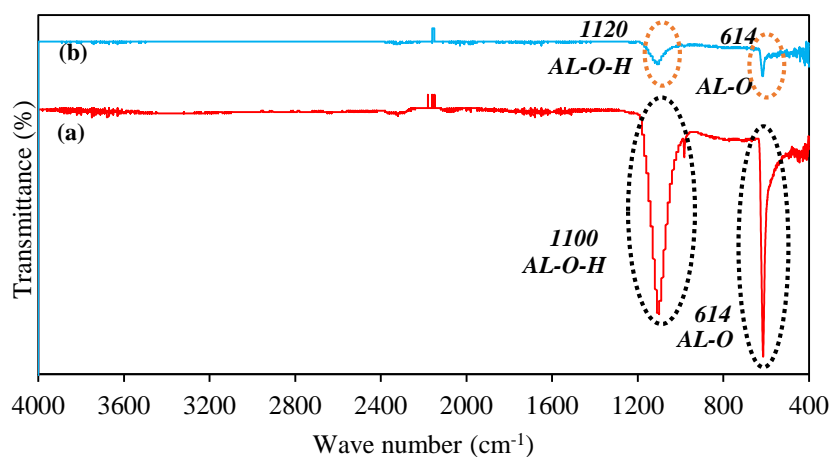


Figure 1. FTIR spectra comparison of the effect of reaction temperature on the formation of Alumina; (a) AL-24-30 and (b) AL-24-40.

The X-ray diffraction patterning of the formed alumina AL-24-30, AL-24-40 and AL-commercial are presented in Figure 2. The observed peaks were identified as diaspore ($\alpha\text{-Al}(\text{OH})_3$), aluminium oxide ($\alpha\text{-Al}_2\text{O}_3$) crystalline structure and observed peaks of corundum which is a structurally crystalline variant of aluminium oxide; they commonly have titanium, iron, chromium, and vanadium traces [13]. The XRD forms of alumina in wide-angle region recommended the phase of $\alpha\text{-Al}_2\text{O}_3$ at $2\theta^\circ$, it possesses broad peaks such as 30.06° , 31.06° , 35.98° , 43.73° , and 54.95° for AL-24-30; whereas, peaks of AL-24-40 were 30.06° , 31.058° , 35.98° , and 43.73° that followed a small peak at 54.95° [14]. However, AL-commercial has an XRD pattern with only one phase of alumina phase types which was corundum that is commonly known as α -aluminium oxide. All the appeared peaks of Al-commercial pattern were similar to corundum. For instance, $2\theta^\circ$ comprises 25.82° , 35.40° , 38.02° , 43.60° , 54.74° , and 60.42° . The alumina synthesized at room temperature were structurally more crystalline with characteristic broad peaks compared to the alumina synthesized at 40°C due to the increased reaction temperature that affects the formation of gel-like alumina; hence, influencing the extent to which its structure was crystallized.

The size of crystallite in the synthesized alumina released from X-ray uttermost broadening using Scherrer formula as depicted in Table 3.1. XRD graph demonstrates the formation of the crystalline phases of alumina passing throughout the following series of polymorphic phase transformation due to thermal treatment before its final conversion to the thermodynamically stable phase $\alpha\text{-Al}_2\text{O}_3$ [15]. The run of transformations is $\text{Al}(\text{OH})_3 \rightarrow \gamma\text{-AlOOH} \rightarrow \gamma\text{-Al}_2\text{O}_3 \rightarrow \delta\text{-Al}_2\text{O}_3 \rightarrow \theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ [16-17]. Alumina synthesized at room temperature showed sharper and longer intensity as well as more crystalline structure, it was almost compatible with FTIR results.

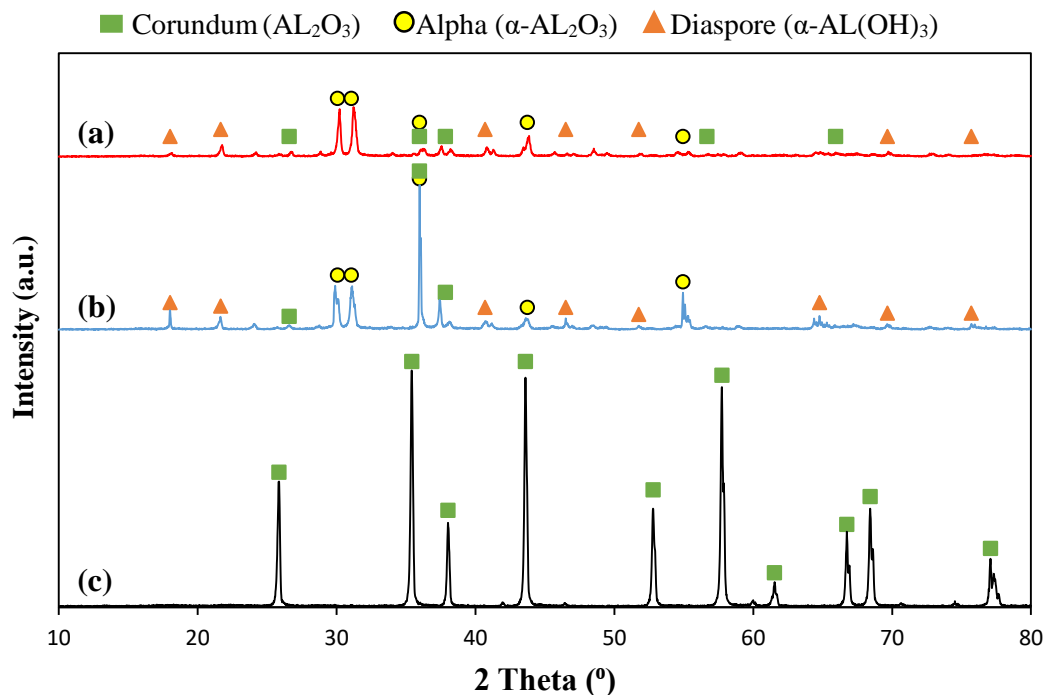


Figure 2. XRD spectra comparison of the influence of reaction temperature on the formation of Alumina; (a) AL-24-40, (b) AL-24-30 and (c) AL-commercial.

Figure 3 represents the SEM-EDX spectra of AL-24-30, AL-24-40 and AL-Commercial with mean weight and elemental atomic percentage. From the figures (Figures 3.4a and 3.4b), it is observed that the solidified morphological appearances for spectra AL-24-30 and AL-24-40 are comparatively identical, with heterogeneous particle sizes. After formation, both alumina have characteristic shapes that were not spherical. It was also observed that linking very minute crystallites during the calcination process resulted in particles with relatively disordered shapes [3]. Comparatively, both alumina exhibits a more morphologically disordered surface appearance than the AL-commercial. In the figures, the red circle indicates the particular spot in which EDX samples were extracted. From the EDX result, it revealed that the powders AL-24-30 and AL-24-40 were made up of sulphur, oxygen, aluminium, and potassium. The presence of potassium in the synthesized alumina was due to its utilization in dissolving the aluminium can wastes and pH adjusting, respectively. Importantly, removing the traces of potassium and sulphur were easily achievable through adequate washing using deionized water for the duration in the synthesis of aluminium hydroxide gel. The formed alumina at room temperature showed the highest elemental composition and percentage atomic weight of aluminium. The results of this finding might be compatible with the previous studies [6, 16].

Table 1 shows the distinct surface area, pore volume and pore diameter of synthesized AL-24-30, AL-24-40 and AL-commercial. One of the suitable approaches for the nano-structure alumina preparation without collapse of the nano-structure phase at calcination temperature 900 °C is the sol-gel technique [14]. The surface area of the synthesized alumina at room temperature increased from 5.2105 to 10.2506 m²/g along with the increasing reaction temperature. Comparatively, the AL-commercial possesses a higher surface area than the synthesized alumina at 15.3875 m²/g. Additionally, the alumina formed at room temperature had higher distinct surface area than the alumina synthesized at 40 °C. Furthermore, the results of surface area analyses are consistent with the results of XRD analyses. This means the alumina at room temperature appeared to be more crystalline structurally in XRD with higher intensity of the observed peaks and lower surface area. This emphasized that an increase in crystallinity lead to a corresponding decrease in the surface area [18]. The highest crystalline size of synthesized

alumina was recorded for alumina synthesized at room temperature to be 132.50 nm. The effect of reaction temperature was further supported by the fewer number of aluminium hydroxide grains formed during aging at higher temperatures. This process allowed a well-defined particles growth and led to the formation of the oxides having bigger pores and lesser surface areas [6].

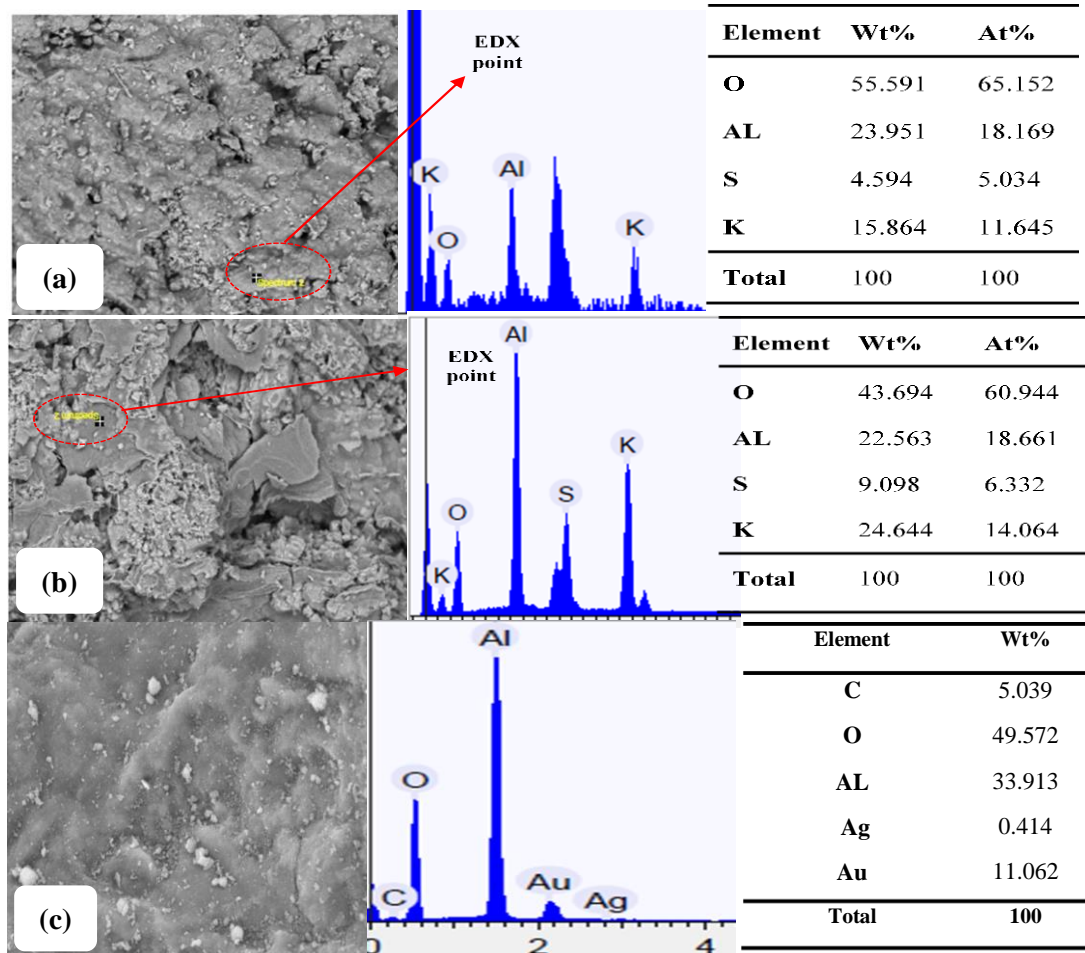


Figure 3. SEM-EDX spectra comparison of the influence of reaction temperature on formation of Alumina; (a) AL-24-30, (b) AL-24-40 and (c) AL-commercial

Table 1. Results of distinct surface area, pore volume and mean adsorption pore diameter of AL-24-30, AL-24-40 and AL-commercial using BET comparison of the effect of reaction temperature on formation of alumina.

Sample	Distinct Surface Area ^a , m ² /g	Pore Volume ^b , cm ³ /g	Mean Adsorption Pore Diameter (4V/A by BET), nm	Crystallite Size ^c , nm
AL-24-30	5.2105	0.022706	86.7218	132.50
AL-24-40	10.2506	0.005608	78.2302	113.50
AL-commercial	15.3875	0.039882	85.9207	131.65

^a Obtained using Brunauer-Emmett-Teller method

^b Analysed using the Barret-Joyner-Halenda (BJH) method

^c Analysed using the X-ray peak in XRD with Scherrer formula

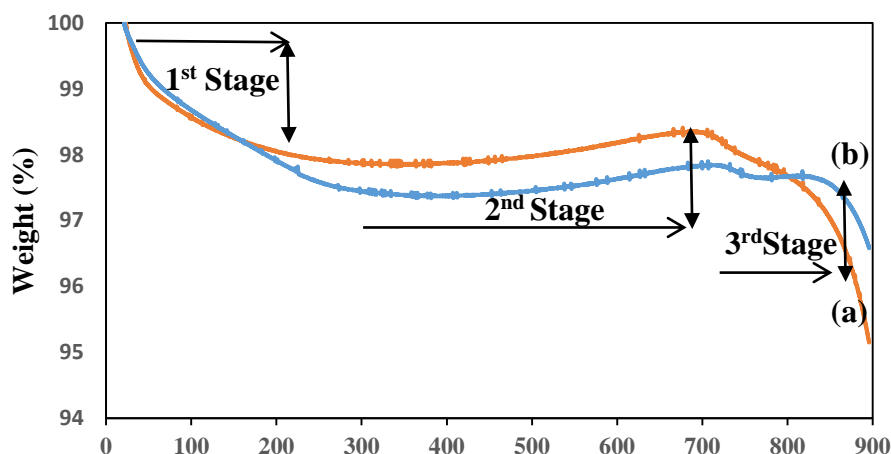


Figure 4. TGA comparison of the effect of reaction temperature on the formation of alumina; (a) AL-24-30 and (b) AL-24-40.

From TGA result, both synthesized alumina had decomposed throughout the three remarkable decomposition stages. Alumina synthesized at 30 °C decomposed at the first stage with the weight loss of about 2.138% at calcination temperature from the beginning to around 150 °C, second stage as the weight loss of around 0.9254% at a calcined temperature between 400 and 690 °C, and final stage with a weight loss percentage of 2.766% at a calcination temperature of around 820°C until it reached the maximum calcination temperature of 900 °C. The alumina synthesized at 40 °C started decomposing with increasing calcination temperature within the time of 100% at the first stage from 21.35 to 100 °C at 2.641% weight loss, the second stage was between 395 and 800 °C with decomposed weight loss of 0.6426%; at the final stage, the decomposition started from 810°C to final calcination temperature of 900°C at 1.079% weight loss. Thus, at calcination of 900 °C, the maximum weight loss of decomposition was about 4.85% and 3.42% for synthesized alumina at aging temperature (for room temperature and 40°C, respectively). This result is in agreement with the study done by Matori et al., who found that the weight loss percentage of α -Al₂O₃ at 900 °C for 3 h was around 5.23% [1]. This implies that the alumina at higher reaction temperature had higher thermal resistance.

4. Conclusion

The goal of this study was achieved in relative to the possibility and visibility of converting aluminium can waste into valuable support catalyst at 900 °C calcination temperature to synthesis α -alumina using sol-gel method. The obtained results from this study showed that α -alumina was successfully synthesized from an aluminium can waste at varying reaction temperatures. The results of this study were in line with the results obtained for the characterization of α -alumina obtained with FTIR, XRD, SEM, EDX, and BET analyses. The synthesized α -alumina had a specific surface area at 10.2105 m²/g which is comparable to the nano-sized alumina. Based on BET and XRD results, alumina synthesized at room temperature was found to have the best conditions at the uppermost precise surface area of 5.2105 m²/g, highest crystallite size of 132.50 nm and total weight loss of 2.71% within higher calcined decomposition temperature at 820 °C.

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