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Electrosynthesis of $Al(OH)_3$ by Al(s)|KCl(aq)||KCl(s)|C(s) system

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Abstract. Electrosynthesis of $Al(OH)_3$ by using Al(s)|KCl(aq)||KC(aq)||C(s) system had been done.The presence of electrolyte was much influenced to the electrolysis result. The purpose of this work was to investigate the purity of $Al(OH)_3$ compound with the presence of KCl concentration using XRD characterization. Electrolysis process was done in 2-compartment system, by using aluminium plate electrode as anode, carbon electrode as cathode, and electrolyte solutions of KCl were varied at the concentrations of 0.25; 0.30; 0.35; 0.40; 0.45 M. The electrolysis process was done at room temperature and potential of 12 V for 6 h.The electrolysis products were characterized using XRD and the the best electrolysis result was determined its thermal property by using TGA-DSC.The result showed that electrolysis of Al(s)|KCl(aq)||KC(aq)||C(s) system with the variation of electrolyte concentration solutions gave white precipitations with the masses of 50.6; 51.8; 56.1; 64.9; 97.2 mg, respectively. The XRD characterization of the electrolysis product precipitates gave the best product as $Al(OH)_3$ when using KCl concentration of 0.3 M, while at KCl concentration of 0.45 M was resulted $KAlOCl_2$, and another variation KCl concentration gave a mixture product of $Al(OH)_3$and $KAlOCl_2$. Thermal analysis result of the best electrolysis product using TGA-DSC showed the reduced total sample mass of 45.47 % and two endothermic peaks at 270.08°C as the transformation of $Al(OH)_3$ to AlOOH (boehmite) with the absorbed energy of 2.47 kJ/mol. At temperature of 660.28°C was the phase change of AlOOH (boehmite) to γ-Al_2O_3 with absorbed energy of 0.23 kJ/mol. Then, at temperature above 800°C there was no mass reduction, that meant it formed stable phase of Al_2O_3. To sum up, the purity of $Al(OH)_3$ compound was much influenced by KCl concentration and the increase of temperature transformed the $Al(OH)_3$ sample to AlOOH (boehmite) and γ-Al_2O_3.

1. Introduction

$Al(OH)_3$ is metal hydroxide that is much used as flame retardant additives for polymer due to its property in thermal degradation that absorbs heat (endothermic), releases water, and forms oxide film of Al_2O_3 at the surface of polymer [1]. Electrosynthesis is a part of electrolysis methods that uses electricity to produce chemical reactions, in this case that involving redox reaction, such as electrosynthesis of $Al(OH)_3$. The method has advantages such as using small amount of reactant, the process is simple and fast [2].

Synthesis of $Al(OH)_3$ was done by Tchomgui-Kamga *et al.* [3] with electrolysis method using 1-compartment system. An aluminium metal electrode with successive electrolyte solutions of $Al_2(SO_4)_3$, $Al(NO_3)_3$, $AlCl_3$, $(NH_4)_2SO_4$, NH_4Cl, $(NH_4)HCO_2$, Na_2SO_4, $NaNO_3$, NaCl, $NaClO_4$, $Na_2C_2O_4$, $NaCH_3CO_2$ were used in the system. The result of bayerite, $Al(OH)_3$,was found in all solutions, except ammonium salts. While boehmine, AlOOH, was resulted from ammonium salts. Shalaby *et al*. [4] did an electrolysis in 1-compartment system using aluminium anode in electrolyte solutions of NaCl, KCl, $NaNO_3$, and $NaNO_2$ with concentration variations of 0.5 to 5 g/L. The result was $Al(OH)_3$ and the higher electrolyte concentration increased the efficiency of the produced coagulation. In this work, we used different system as previous researches, since by using 1-comparment the main and side products will mix together and makes the product characterization difficult therefore, 2-compartment system was used[5, 6]. It contained aluminium as anode in one compartment and graphite as cathode in another compartment that was connected by salt bridge.Yan *et al.* [7] had done a synthesis by using 2-compartment system namely by separation of $Al(OH)_3$ and NaOH from $NaAlOH_4$ solution with titanium coated with ruthenium as the electrodes. Graphite has an advantage of its inertness, therefore it is not easy to be oxidized or reduced, while alumilum anode as Al^{3+} ion sources from its oxidation reaction. Electrolyte solution of KCl was used as in 1-compartment since it has a good conductivity, in this case the KCl concentrations were varied (0.25, 0.30, 0.35, 0.40, 0.45 M). The purpose of this work was to get $Al(OH)_3$ by electrosynthesis and to characterize the purity of the products using XRD as well as to get the thermal property of pure $Al(OH)_3$ using TGA-DSC.

2. Experiment methods

The solution of 200 mL KCl (concentrations of (0.25, 0.30, 0.35, 0.40, 0.45 M) was soaked to 2-compartment system. Aluminium electrode (9 cm x 4 cm) in anode compartment and 8 bar of graphite electrodes (from bateray) in cathode compartment with the distance among the electrodes was 2.5 cm. The two compartments were connected with salt bridge. The electrolysis was done for 6 h with potential of 12 V and the electrolysis obtained product was $Al(OH)_3$. Then, the product was filtered, dried and characterized using XRD (Shimadzu Maxima 7000) to know crystal products in $Al(OH)_3$ sample. TGA-DSC (Perkin Elmer 6000) was used to investigate the decomposition temperature and thermal property of the product.

3. Results and Discussion

In the electrolysis process, aluminium metal anode dissolve to form Al^{3+} [4], while graphite is inert and remains stable, therefore in cathode compartment water will reduce to hydrogen gas. The reduction and oxidation reactions that occur in electrolysis process as the following [4]:

$$Al^{3+}(aq) + H_2O\,(aq) \longrightarrow Al(OH)^{2+}(aq) + H^+(aq) \quad (1)$$

$$Al(OH)^{2+}\,(aq) + H_2O(aq) \longrightarrow Al(OH)_2^+\,(aq) + H^+(aq) \quad (2)$$

$$Al(OH)_2^+\,(aq) + H_2O\,(aq) \longrightarrow Al(OH)_3\,(s) + H^+(aq) \quad (3)$$

Overall reaction:

$$Al^{3+}\,(aq) + 3H_2O(aq) \longrightarrow Al(OH)_3(s) + 3H^+(aq) \quad (4)$$

The cation of K^+ flows to cathode and reacts with OH^- from water reduction reaction to form KOH. The reaction as follow [8]:

$$K^+(aq) + OH^-(aq) \longrightarrow 2KOH(aq) \quad (5)$$

The formation of KOH was proven by the increase of pH solution at catode compartment.

3.1. *Effect of KCl concentration to electrolysis products*

The increase in KCl concentration will improve the current that flows through the electrolysis cell [9], as consequence it will improve the precipitate resulted. as in shown in Fig. 1.

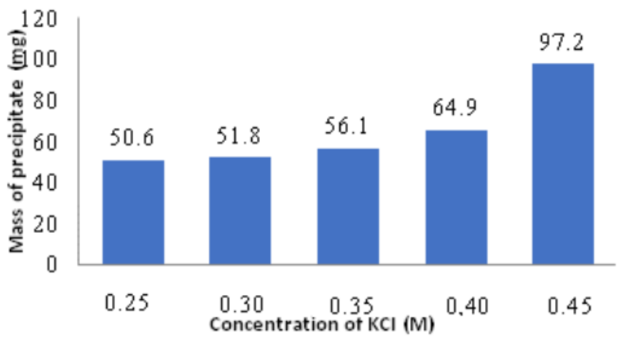

Figure 1. The correlation between electrolyte concentration (M) with electrolysis mass of precipitate products.

3.2. Characterization of electrolysis products

3.2.1. X-ray Diffraction (XRD). The difractograms of electrolysis precipitate products with the varied concentrations of KCl can be seen in Fig. 2. The XRD characterization of the high peaks of the sample found was compared with JCPDS database number 20-0011 and 73-0631 for $Al(OH)_3$(bayerite) and $KAlOCl_2$compounds, respectively.

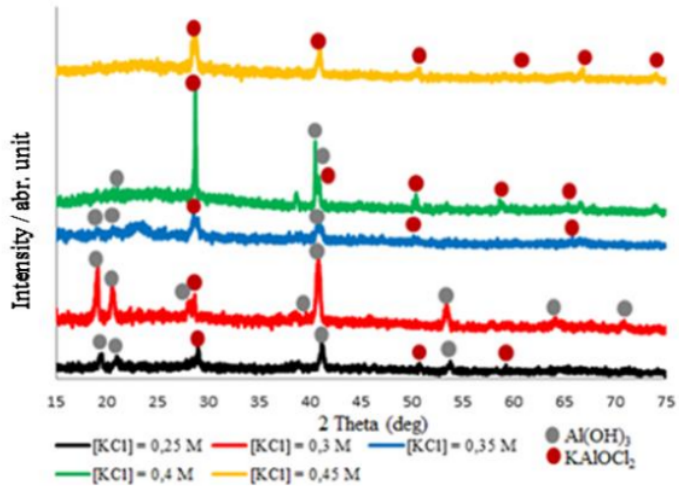

Figure 2. Difractogram of electrolysis products with varied concentration of electrolytes.

The product resulted from electrolysis process with KCl concentration of 0.25 M was a mixture of $Al(OH)_3$ and $KAlOCl_2$ as shown by the peaks at 2θ of 41.16, 19.43, 21.98. JCPDS database number 20-0011 and 73-0631 for $Al(OH)_3$ and $KAlOCl_2$ have 2θ of 28.87, 50.77, 59.19 and 28.42, 40.63, 50.32, respectively.The product resulted from electrolysis in KCl 0.3 M was $Al(OH)_3$ shown by 8 peaks with 3 high peaks at 2θ of 40.79, 19.07, 20.60 as the peaks of $Al(OH)_3$. Electrolysis with KCl 0.35M produces a mixture of $Al(OH)_3$ and $KAlOCl_2$ confirmed by the peaks at 2θ of 40.86, 19.10, 20.71 as $Al(OH)_3$ and the presence of peaks at 2θ of 28.64, 66.57, 50.40 as $KAlOCl_2$. while the electrolysis with KCl 0.40M also resulted a mixture of Al(OH)$_3$ and KAlOCl$_2$ supported by the peaks at 2θ of 40.52, 40.76, 20.63 as Al(OH)$_3$. The presence of KAlOCl$_2$ is shown at 2θ of 28.69, 40.85, 50.39. Finally, the compound resulted from the elecrolysis with KCl 0.45M is a compound of KAlOCl$_2$. The presence of KAlOCl$_2$ is shown by 6 peaks with 3 high peaks at 2θ of 28.67, 40.88, 50.57 as KAlOCl$_2$. Therefore the electrolysis result in KCl 0.30 M gave Al(OH)$_3$ product as shown by 8 peaks of Al(OH)$_3$. In KCl 0.45 M as the highest concentration of KCl used is identified 6 peaks of KAlOCl$_2$. Meanwhile for other variation of KCl concentrations result a mixture of Al(OH)$_3$ and KAlOCl$_2$. The presence of KAlOCl$_2$ can be possibly explain as follow. The KCl ion reacts with Al^{3+} ion released from electrolysis of aluminium anode to form KAlCl$_4$. The presence of Al$_2$O$_3$ as the result of aluminium anodation [9] will react with KalCl$_4$ to form KAlOCl$_2$ [10].

3.2.2. TGA-DSC. The electrosynthesis result from KCl 0.3 M as the product is Al(OH)$_3$ analyzed by XRD above, was characterized using TGA-DSC instrument with heating temperature from 50°C to 950°C with heating rate of 10°C/min. Thermogram of the sample is depicted in Fig. 3.

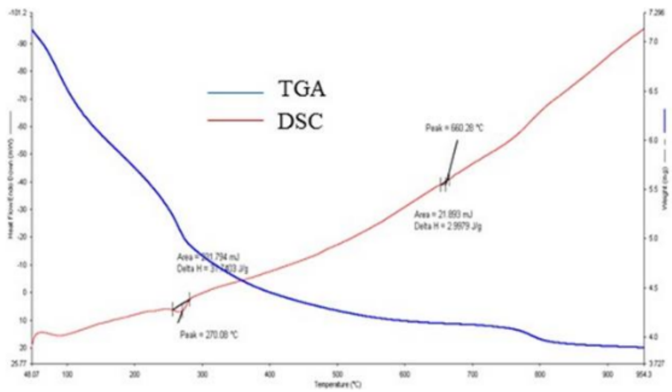

Figure 3. Curve of TGA-DSC of Al(OH)$_3$

DSC curve is formed two endothermic peaks and TGA curve shows the reduced mass of the sample. The sample masss loss is 45.47%. The hydroxyl release process occurs at temperature 100-350°C [10], with first endorthermic peak at 270.08°C with absorbed energy of 31.74 J/g or 2.47 kJ/mol and the total energy absorbed is 231.79 mJ. The peak shows the tansformation from Al(OH)$_3$ (bayerite) to AlOOH (boehmite) [11]. Transformation process is from AlOOH (boehmite) toγ-Al$_2$O$_3$occurs at 550-800°C [11] with second endothermic peak at 660.28°C with absorbed energy of 2.99 J/g or 0.23 kJ/mol and total of absorbed energy is 21.89 mJ. At temperature above 800°Cthere is no mass loss of the sample that shows the stable Al$_2$O$_3$ phase formation.

Theoretically, thermal decomposition process from Al(OH)$_3$ to Al$_2$O$_3$ absorbs energy of 173.61 kJ/mol, while from the characterization is 2.71 kJ/mol. This is due to the impurity that exists in the synthesis result of Al(OH)$_3$. In methane gas combustion, the released energy is 802.36 kJ/mol. Some moles of Al(OH)$_3$ can absorb the energy released and retard the combustion from methane.

Thermal analysis of Al(OH)$_3$ decomposition using TGA-DSC instrument occurs endothermically. The Al(OH)$_3$ decomposition that absorbs energy will increase the fire retardation due to water release from Al(OH)$_3$ and liquidifyes the flammable gas. The decomposition reaction of Al(OH)$_3$ is as the following [12]:

$$Al(OH)_3 \longrightarrow Al_2O_3 + H_2O \qquad (6)$$

Beside that it also forms a thermally stable Al$_2$O$_3$ layer on the the flammable gas.

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4. Conclusion

White precipitates with different masses were formed for electrolysis processes using aluminium anode with varied KCl solutions. From purity analysis using XRD found that precipitate of $Al(OH)_3$ was found in electrolysis using KCl 0.3 M, while $KAlOCl_2$ was formed in the solution of KCl 0.45 M. The mixed compounds of $Al(OH)_3$ and $KAlOCl_2$were formed in other variations of KCl concentrations. Thermal analysis using TGA-DSC for $Al(OH)_3$ from the electrolysis using KCl 0.3 M solution shows reduced mass total of 45.49 % and two endothermic peaks at 270.08°C is the transformation of $Al(OH)_3$ to AlOOH (boehmite) with absorbed energy of 2.47 kJ/mol, while at 660.28°C is the phase change of AlOOH (boehmite) to γ-$Al_2O_3$with absorbed energy of 0.23 kJ/mol. At temperature above 800°C there is no mass loss of $Al(OH)_3$ sample due to the formation of thermally stable phase of Al_2O_3.

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