Removal of Lead From Aqueous Stream Using Aluminium Oxide (synthesized by simple precipitation method) as an Adsorbent

by

Mohamad Saiful Bin Hamid

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

____

(Professor Dr. Khairun Azizi bt Azizli)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHAMAD SAIFUL BIN HAMID

ABSTRACT

Lead is one of the heavy metals that have high bad effect to the human health which can be found in waste water due to human activities related with heavy metal industry. Lead can be removed using adsorption process using aluminium oxide as adsorbent. Commercial aluminium oxide was used as the controlled variable to compare the effectiveness as an adsorbent with the synthesized aluminium oxide. Both of the adsorbents characterized using FESEM and XRD showed that they were in y-phase with low crystallization. Synthesized aluminium oxide has mesopores with pores ranging from 2- 50 nm. Surface area for synthesized aluminium oxide (161.4m^2/g) was higher than commercial aluminium oxide which 90-100 m^2/.g. Study on effect of contact time showed that both of the adsorbents seemed to achieve equilibrium after 30 minutes. However lead uptake using synthesized aluminium oxide was higher than commercial aluminium oxide. Adsorption at different pH using both of synthesized and commercial aluminium oxide was highest at pH11. The adsorption study carried out showed that the synthesized aluminium oxide potentially gave better results than commercial aluminium oxide.

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

Lead is needed for various purposes in life. However, excess of lead and mismanagement of lead can result in damages to the environment and human. Contamination of lead in water maybe caused by waste water discharge from the battery industry, corrosion of lead service lines and natural existence. Lead is toxic at certain level of concentration which is harmful to human health [1-2].

The study for removal of lead has been carried out continuously to find the effective way of removing it. Adsorption is one of the methods that have been used for this purpose. Researchers focus on finding good adsorbent that can efficiently remove lead. Several studies on the adsorption of lead using different types of adsorbents have been carried out since last few years.

In finding the best adsorbent, several factors have been taken into consideration that may affect the whole process. The kinetic of adsorption is the main factor to investigate the rate of the lead adsorbed. However other factors that may influence its application in industry are cost, availability, durability of the adsorbent and serviceability.

S.H.Abdel-Halim *et al.*[3] used different types of natural materials such as animal bone powder, Nile rose plant powder, commercial carbon and ceramic as adsorbent. The study was to learn and understand the ranking of these adsorbents with respect to the removal of lead. Factors that can affect the adsorption process were also studied i.e. contact time, pH, and the concentration of lead solution. The sequence of good adsorbent at different condition is in order of: bone powder > active carbon > plant powder > commercial carbon.

Amarasinghe and Williams [4] used tea waste as adsorbent. Tea waste was introduced in this study because of low cost. In this study, lead and cooper were used to compare the effectiveness of tea waste as adsorbent. The adsorption increases as the pH increases.

1.2 Problem statement

1.2.1 Problem identification

Many industrial waste water, particularly those associated with metal manufacturing and processing, contain high concentration of heavy metal such as lead. Lead causes health problem to the human and disturbs the food chain of the environment as it is poisonous. The amount of lead in the river will increase if the waste water discharged from the related industries does not undergo proper treatment process. Lead dissolves in water as lead hydroxide or lead sulfate [2]. Lead sulfate dissolves at higher concentration at pH of 8.7 and above while the concentration of lead hydroxide is inversely proportional to the pH of water.

Recent industrial practice for lead removal is by precipitation as lead carbonate [2]. The other possible method is using adsorption to remove lead. Literature review shows that different approaches have been used such as activated carbon and few other adsorbents. In this project, the potential of aluminium oxide as an adsorbent will be explored for the removal of lead from aqueous stream.

1.2.2 Significant of the study

Adsorption is an alternative way for lead removal and any other materials. The usage of adsorption had been proven to have a high efficiency and easier to be implemented. It is also more economical compared to any other process.

The use of activated carbon is widely used for the removal of heavy metal. However aluminium oxide has the potential as an adsorbent and has been used to remove other chemicals. The porosity of aluminium oxide is high and this is an important characteristic as an adsorbent. Aluminium oxide prepare using simple precipitation method is cheaper than preparing it using sol-gel technique.

This project will explore the potential of aluminium oxide prepared using simple precipitation method. The characteristics of the aluminium oxide prepared by this process will be compared to the characteristics of commercialize aluminium oxide available in the market.

1.3 Objective of study

The objective of the project is to study the characteristics of the aluminium oxide prepared using simple precipitation technique with the commercialize aluminium oxide. Both of the adsorbents have different characteristics because the processing routes are different.

Experiments were conducted to find the optimum parameters for adsorption of lead. The parameters include pH, contact time, concentration of lead and amount of adsorbent used.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of lead

The history of lead started around 6500 B.C. during the Roman Empire and this metal was well-known for its usage. The main use of lead was for utensil, food storage container lining, water and sewer pipes, ink and paint.

Lead, a bluish-white metal is soft, malleable, ductile and a poor electric conductor. Lead actually is resistant to corrosion but can tarnish upon exposure to the air. This soft and malleable metal is widely used because of its properties and that the shape of the metal can be transformed easily. The symbol for lead in the periodic table is simply “Pb” or known as plumbum. The atomic number is 82 and the atomic mass is 207.2 $g.mol^{-1}$. At 20 ^{o}C, the density of this metal is 11.34 $g.cm^{-3}$ and can vary as the temperature changed. Compared to the other hard metals, lead melts at 327 ^{o}C and the boiling point is 1755 ^{o}C, which is quite low [5].

Pewter and solder are types of lead alloys. The application of lead is mostly found in the lead acid battery and for vehicles. One of the special property of lead is it can shield radiation. A few millimeters of lead layer can protect humans from any radiation that can be harmful. Therefore, lead is used in the manufacturing of lead based protection dressed for those who are exposed to radiation. This is also applied for computer and television glass to protect users from radiation [5].

Lead can be found in air, water and soil either naturally or due to human activity. In air, lead exists as vapor, fine particles of organic halogens such as Pb bromide and Pb chloride [6]. Naturally lead is discovered in mineral called Galena in the form of PbS, widely mined in Australia other country [5]. Most of the lead found is due to heavy metal industry such as mining, electro-plating, lead smelting and metal finishing industries [3]. The concentration of lead in water rises to dangerous level that can become a threat to humans.

The issue of the lead amount in water has become an important topic to be discussed due to its toxicity. Many health effects are due to the excess amount of lead in body. This element enters into human body through the consumption of the polluted water.

Lead in water is found as lead hydroxide. However, lead does not simply react with water. The reactivity of lead to react with water increases mainly due to the existence of oxygen as shown in Equation 2.1.

$$\boxed{2Pb(s) + O_2(g) + 2H_2O(l) \longrightarrow 2\,Pb(OH)_2(s)} \qquad \text{Equation 2.1}$$

Lead is one of four metals that have most damaging health effect. Lead can accumulate in the bloodstream, bone and soft tissues. Excessive amount of lead in the body can cause anemia, damages the kidney, reproductive disorder, mental disorder and behavior disorder. Woman and children uptake lead more than man. It can cause spontaneous abortion, menstrual disorder and infertility. Lead can affect the children by slower down growth. Most extreme effect is it can cause death if the amount of lead exceeds 100 µg/dL of blood [5-6].

World Health Organization (WHO) has set the limit of lead in water as 10 ppb in 2010 [5]. Therefore, new methods of removing lead from water are studied to reduce the concentration of lead in water and to improve health quality.

2.2 Adsorbent

Literature review from a few journals show that the effort of finding the best method to remove lead from water is continuously carried out by researchers around the world. Different methods used have advantages and disadvantages that can be further improved. Most of the methods used are adsorption of lead using different types of adsorbents. The selection of the adsorbent depends on its characteristic and properties that can adsorb lead at certain rate and certain capacity. Other factors are also taken into consideration for efficiency.

The general attributes of adsorbent for any purposes are capacity, selectivity, regenerability, kinetics compatibility and cost [7]. An adsorbent will not fulfill all the attributes as a perfect adsorbent. Adsorbent must has the attributes since that is the characteristics of adsorbent.

Capacity is the loading which mean the amount of the adsorbate that can be adsorbed by an adsorbent. This is the important characteristic of an adsorbent. The capacity depends on the factors that influence the adsorption such as temperature, pressure, pH fluid condition and others. Capacity is related to cost in a way that if the capacity of adsorbent is high, then the amount of the adsorbent needed is lesser. Capacity is affected by the structure of the particle. Particle with high surface area and high porosity generally has high capacity.

Selectivity is about the interaction that exists between the adsorbent and fluid. Different adsorbent has different selectivity on a certain element. It is related to the force that exists on the surface of adsorbent. Adsorbent must be regenerability. The application of adsorbent required the adsorbent to be regenerated once it is saturated.

Time for the adsorption to reach breakthrough curve in a fixed bed is much depending on the kinetics of the adsorbent. The faster the kinetic the faster the adsorption can reach breakthrough curve. It mainly related to the rate of the adsorption process. Compatibility covers all aspect of chemical and physical attack toward the adsorbent. Chemicals can damage the adsorbent if it is not compatible.

Cost is one of the factors that influence the selection of the adsorbent. An adsorbent should be cost effective regardless for what type of application it being used for. High capacity adsorbent with high cost will not be favorable for application. Adsorbent that can fulfill most of the attribute can be categorized as an effective adsorbent.

Natural materials were used by Abdel-Halim [3] to study the kinetic adsorption of lead. The materials used were animal bone powder, active carbon, Nile rose plant powder, commercial carbon and ceramics. Lead uptakes for bone powder at any parameters was highest compared to others. The uptake by ceramics is almost negligible meaning that ceramics was not a good adsorbent to remove lead. The equilibrium state for bone powder, active carbon, Nile rose powder plant and commercial carbon were achieved at 15, 30, 45 and 120 minutes respectively. The adsorption isotherm of lead for all of the adsorbent favors the pH of the solution reaching neutral condition which is pH of 6.

The adsorption of lead using tea waste follows the Langmuir isotherm [4]. The equilibrium time for lead uptakes is 20 minutes. The same condition was studied by Abdel-Halim [3], where the adsorption of lead using tea waste as adsorbent was maximum in the range of pH 5-6.

Various studies conducted using different types of adsorbents have shown the optimum parameters for lead adsorption. These parameters can be used as reference for this study. The optimized parameters may change depending on the adsorbent used. Further steps in this study will identify the best parameters for the lead adsorption.

2.3 Aluminium oxide as adsorbent

This study is focusing on aluminium oxide (Al_2O_3) as the adsorbent for lead removal from aqueous stream. Aluminium oxide is also known as alumina and is very important for ceramic industry. The main property that suits this adsorption process is because aluminium oxide is not soluble in water. The density of this adsorbent is 4.0 g/cm^3. It is poor electricity conductor but has good thermal conductivity. The boiling point and melting point is very high at 2980^{o}C and 2000^{o}C respectively. It resists acid and alkaline attack and possess very high strength and stiffness.

Seong-Geun (2002) [8] compared the aluminium oxide prepared using different precipitating agent which are ammonium hydroxide and ammonium hydroxide. The solid obtained using ammonium hydroxide as precipitating agent was transformed to aluminium oxide using calcinations process. Both of the solids obtained by different precipitating agent are in the y phase, a phase that has high porosity.

The lead adsorption by aluminium oxide occurs due to physical bond form at the surface of aluminium with lead. The adsorption also occurs in the pores of adsorbent. The higher the porosity the better the adsorbent is.

Porosity can be categorized into three different types; micropores (0.3-2 nm diameters pore), mesopores (2-50 nm diameters pore) and macropore (diameter larger than 50 nm) [9]. The pore categories were classified by International Union of Pure and Applied Chemistry (IUPAC).

2.4 Basic theory of adsorption

The adsorption theory was developed in 1881 by Kayser that described the increasing concentration of the gas or liquid in the neighbouring solid surface. Roque-Malherbe [9] defined adsorption as increase in concentration of gas molecules in a solid surface or increase in the concentration of a dissolved substance at the interface of a solid in a liquid phase.

The adsorption occurs as the molecule of the adsorbate is adsorbed onto the surface of the adsorbent. The equilibrium state is when the adsorbent is fully saturated with adsorbate. The adsorption process on the solid surface may happened either at one layer of adsorbate or more depending on the Van der Waals force created between the adsorbate and adsorbent [10-12].

The equilibrium between the concentration of the solute and the adsorbent can be illustrated as in Figure 2.2.

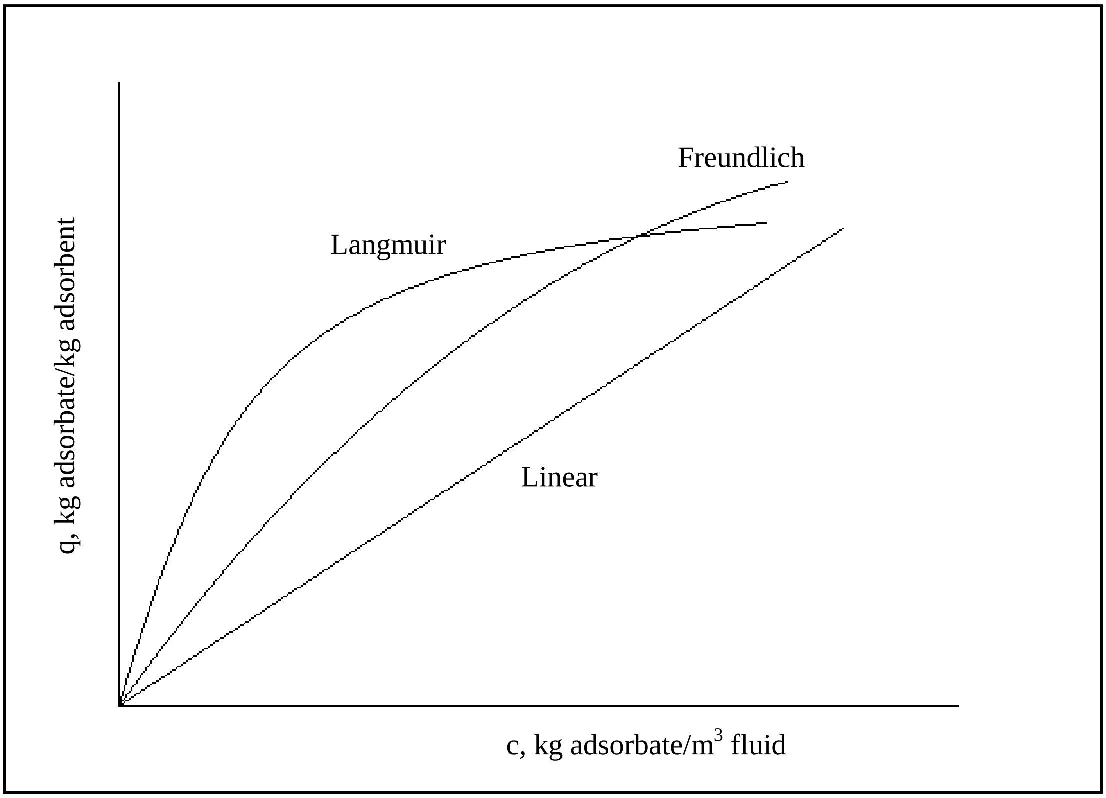

Figure 2.2: Some common types of isotherm (Geankoplis, 2003)

The adsorbent will reach the equilibrium where the surface is saturated with adsorbate. At this point, no more adsorption process will take place. Therefore, the adsorbent need to be regenerated to remove all the adsobate and to be used back for the adsorption process [10-12].

Some factors that can influence the adsorption process are pH of the solution, temperature, concentration of the solution and surface area. In this experiment, the aluminium oxide is prepared in solid powder form to maximize the contact surface. The adsorption of heavy metal occurs mainly due to the physicochemical interaction between the metal ion and the surface of the adsorbent [4].

CHAPTER 3

METHODOLOGY

3.1 Research methodology

The adsorption study using aluminium oxide as an adsorbent is illustrated in Figure 3.1. Commercial aluminium oxide was used as the controlled experiment. The results obtained for synthesized aluminium oxide were compared to the commercial aluminium oxide.

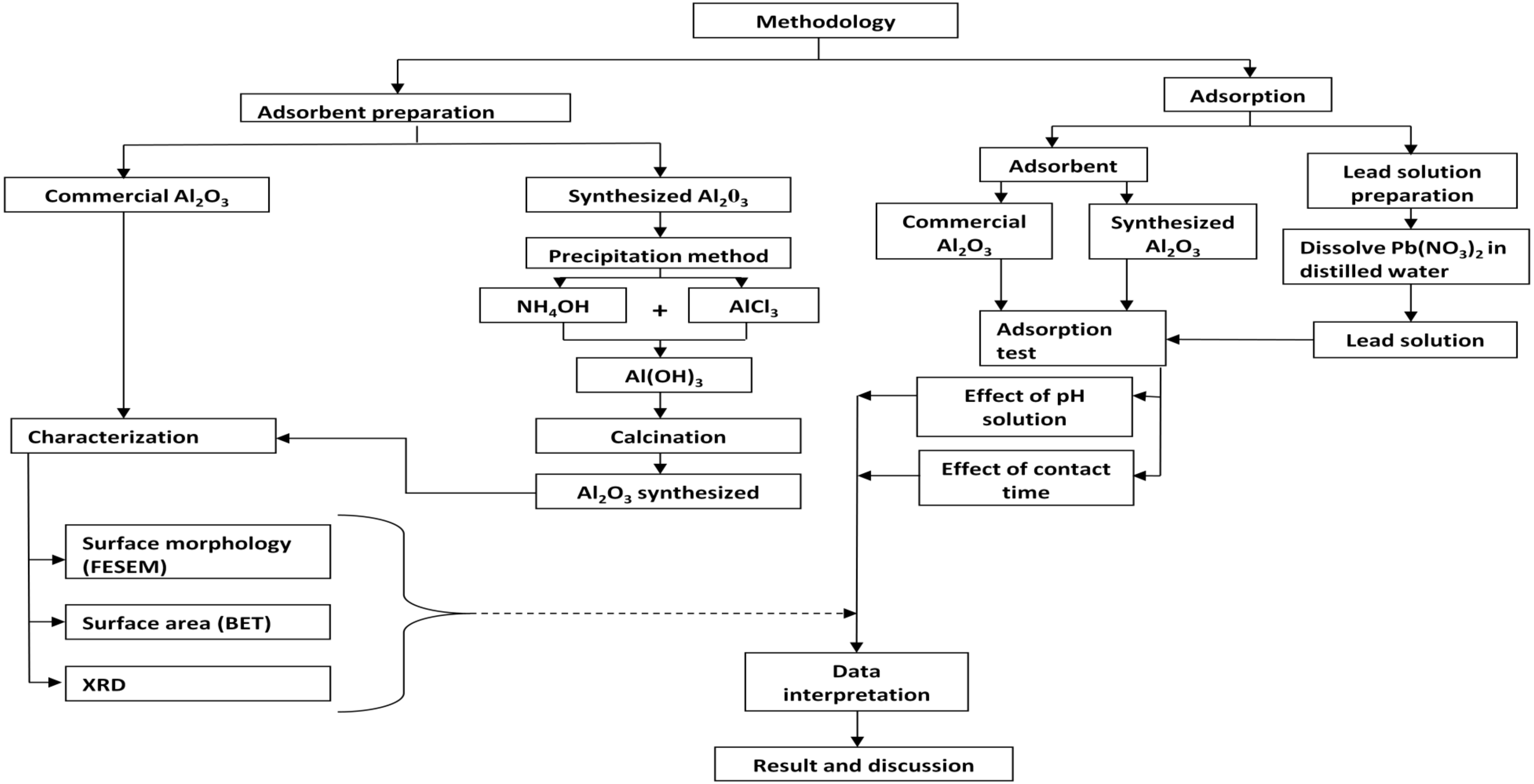

Figure 3.1: Research methodology

3.2 Preparation of aluminium oxide

Aluminium oxide was prepared using simple precipitation method using aluminium chloride and ammonium hydroxide as the reactant. This experiment used aluminium chloride suppled by Merck. 2 molar of the solution was titrated with ammonium hydroxide solution 30% (Merk) until it forms almost gel form. The gel is then washed with distilled water to remove any unreacted chemical. The precipitate was dried in the oven for 72 hours at120^{o}C to completely dry the aluminium oxide. It was then ground with a grinder to obtain small particles of aluminium oxide. The solid was then calcined for 2 hours at 700 ^{O}C to activate the adsorbent and increase the porosity. The adsorbent is then characterized using FESEM, BET, XRD, and TGA.

3.3 Preparation of lead solution

Lead solution was prepared using lead nitrare ($Pb(NO_3)_2$) as this is the only lead substance that can dissolve in water besides lead acetate. In general, to prepare 1000 ppm of lead solution, 1.598 g of lead nitrate is to be dissolve in 1L of distilled water. Further dilution process can be done to get the desired concentration.

3.4 Adsorption Test

3.4.1 Effect of pH of solution

1. 100 ml of 40 ppm lead nitrate solution were placed in 5 conical flasks.
2. pH values of solution adjusted with 0.5M HCl and NaOH solutions :
   Varied pH: pH 3, pH 5, pH 7, pH 11 and pH 13
3. The solutions were placed in a water bath shaker at 190 rpm at 27ºC
4. At time zero, 1.0 g of adsorbent was added into each of the conical flasks.

4. Samples of solution were taken from the conical flasks at interval time of 5, 10, 15, 30, 60, and 120 minutes. The solutions were filtered and analysed.

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3.4.2 Effect of contact time

1. 250 mL of 40 ppm lead solution was prepared using the standard solution (1000 ppm) in a conical flask.
2. The solutions was set at pH 5 and was placed in a water bath shaker at 190 rpm at 27ºC. At time zero, 1.0 g of adsorbent was added to the conical flask.
3. Samples were taken at 5, 10, 15, 30, 60, 120 and 180 minutes and sampling was continued until the systems attained the equilibrium.
4. Solutions were filtered and analyzed.

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Adsorbent Characterization

Both of the adsorbents, commercial and synthesized aluminium oxide were characterized using FESEM, TGA and XRD. The characterization was to obtain the properties of the adsorbent. The synthesized aluminium oxide was characterized using nitrogen gas adsorption method (BET) to determine the surface area. Characterization of adsorbent is important to understand the adsorption behavior based on its properties.

4.1.1. Field Emission Scanning Electron Microscope (FESEM)

Adsorbents were characterized using FESEM (model ZEISS SUPRA$^{\text{TM}}$ 55VP) to get the particles morphology of the adsorbents at 30 000 magnification. From the image, the structure of its pores can be analyzed. The commercial aluminium oxide structure is lamellar of coarse particle as shown in Figure 4.1 (a) and (b). There is less pore can be seen clearly from the image. The surface of the adsorbent is almost flat which contain small amount of pores and resulting in low surface area.

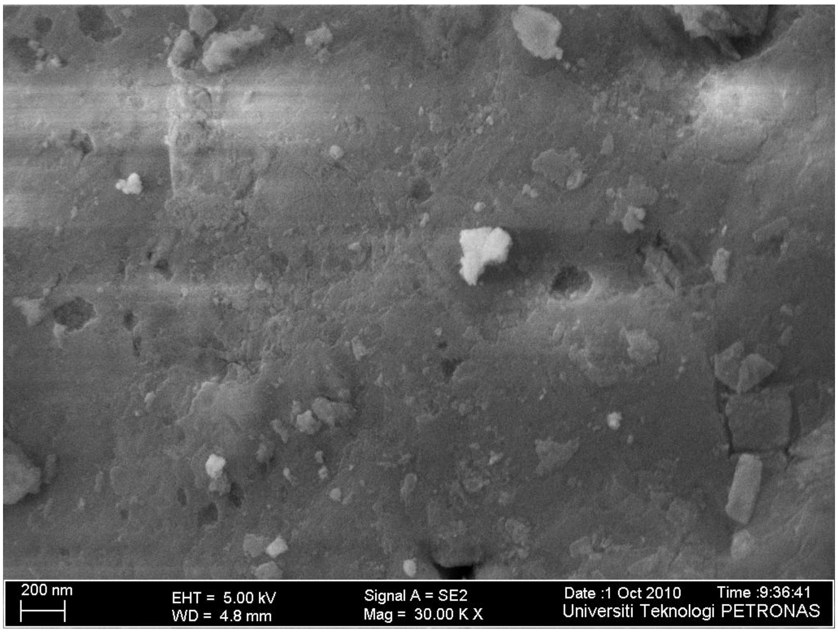

Figure 4.1 (a): Commercial aluminium oxide at 30k magnification

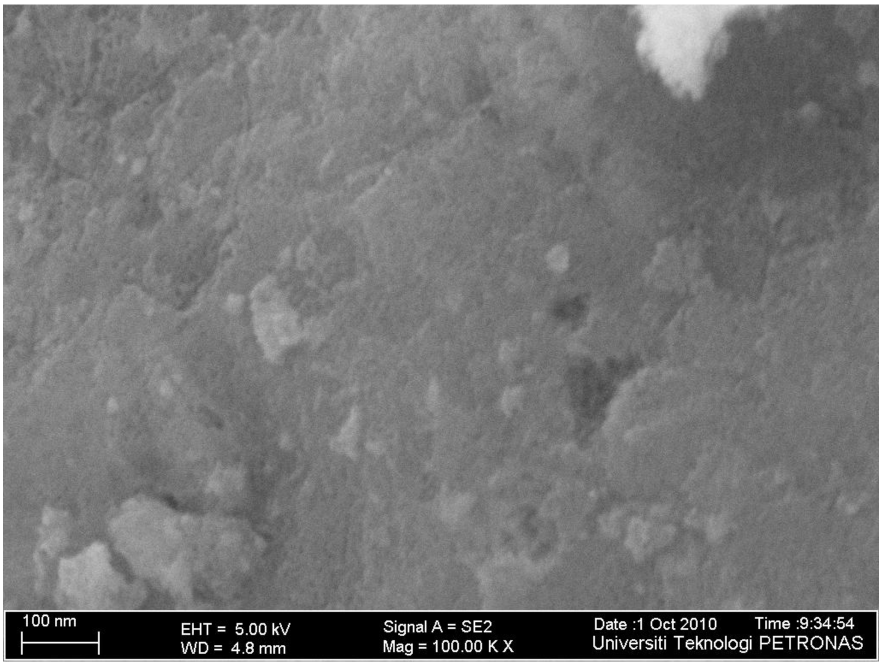

Figure 4.1 (b): Commercial aluminium oxide at 100k magnification

The difference in the microstucture of the two adsorbents is definite. The synthesized aluminium oxide under the same magnification shows the pores of the particle as in Figure 4.1 (c) and (d). The apertures stucture with high porosity can be seen from the images.

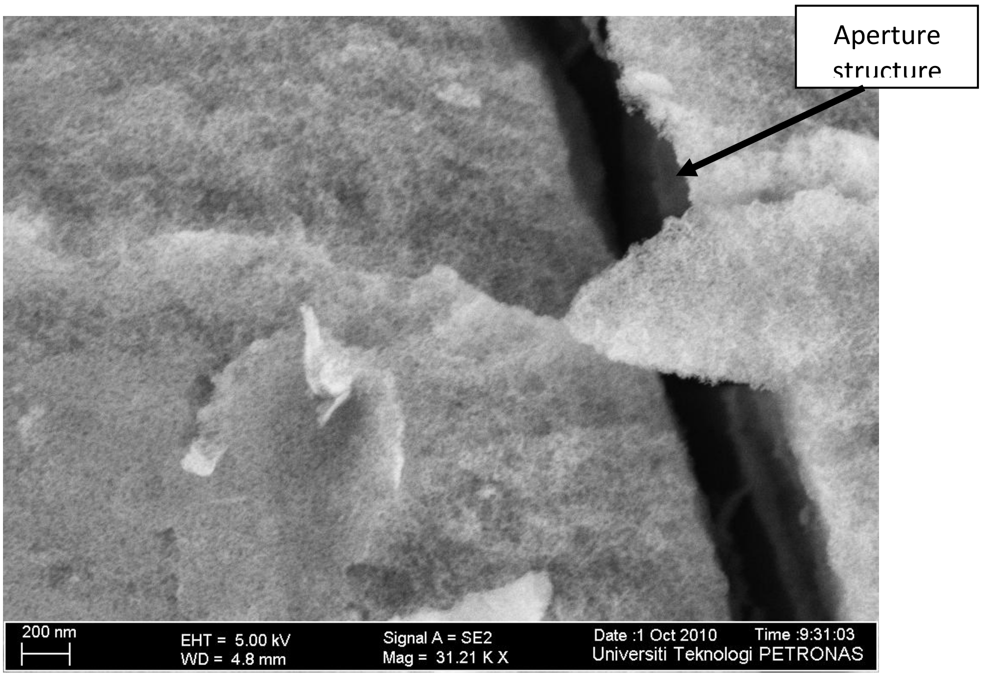

Figure 4.1 (c): synthesized aluminium oxide at 30k magnification

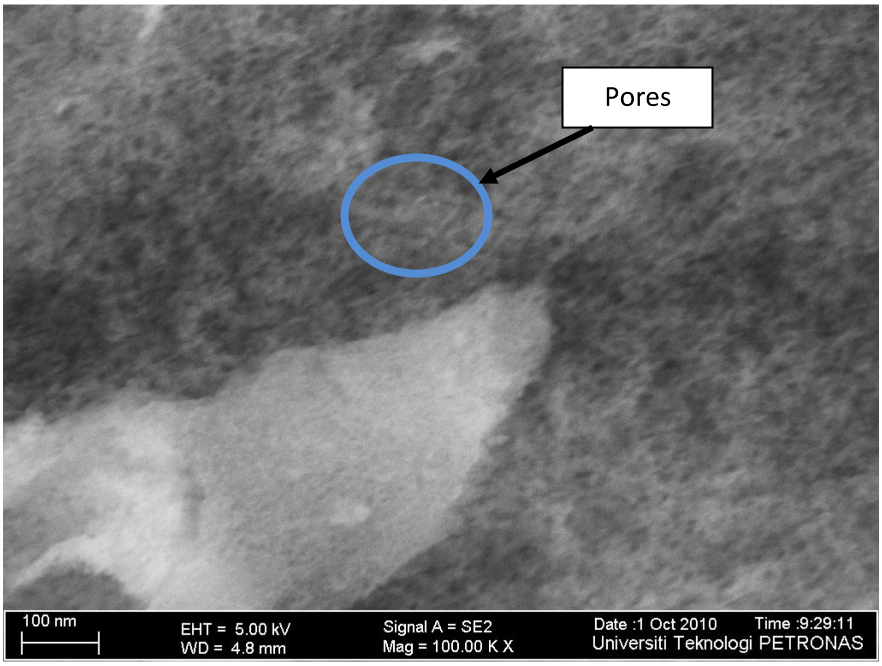

Figure 4.1 (d): synthesized aluminium oxide at 100k magnification

The formation of aluminium oxide particles is in irregular shape with no uniform size distribution. Aluminium oxide prepared by precipitation of ammonia is due to the formation of the particles without any geometrical restriction. The reaction was immediate after the mixing produced spontaneous reaction. It was observed by many author that the reaction cause the nouniform growth of particles that lead to the formation of wide distribution of particle size. The rate of the production of hydrolyzed species is rapid which lead to the increasing of concentration of hydrolyzed species exceeding the concentration that can bring the nucleation. This condition causes the nucleation and the growth happened simultaneously [8].

The calcinations process activates the surface of the particles and also contributes to the size of particles. The calcinations at different temperature determine the particles size distribution.

The difference in microstructure determines the adsorption performance. The higher the porosity of the adsorbent, the higher amount of lead uptakes. The porous structure might be because of the usage of ammonium hydroxide as the precipitation agent. Calcination process is also one of the factors that make the structure pores.

4.1.2. Surface measuring using BET

The synthesized aluminium oxide was tested using nitrogen adsorption and desorption test (BET) to measure its surface area. Since the BET test for commercial aluminium oxide was not available, the surface area was taken from the supplier. The BET surface area for commercial aluminium oxide used in this study was in the range of 90- 100m^2/g (from supplier, Merck Chemicals).

Figure 4.2(a) is the result of the adsorption and desorption of nitrogen gas. The result shows that the sample exhibit IV isotherm which is the characteristic of the mesoporous material. The isotherm shows that there are two steps in the hysteresis loop that indicates the presence of mesopores in the sample. From Figure 4.2 (b), the distribution of the mesopores can be seen. The mesopores is in range of 2-50 nm in diameter. From the data obtained, the surface area for synthesized aluminium oxide is 161.4 m^2/g. The average pore diameter of the synthesized aluminium oxide is 8.68nm and the pore volume is 0.360 cm^3/g..

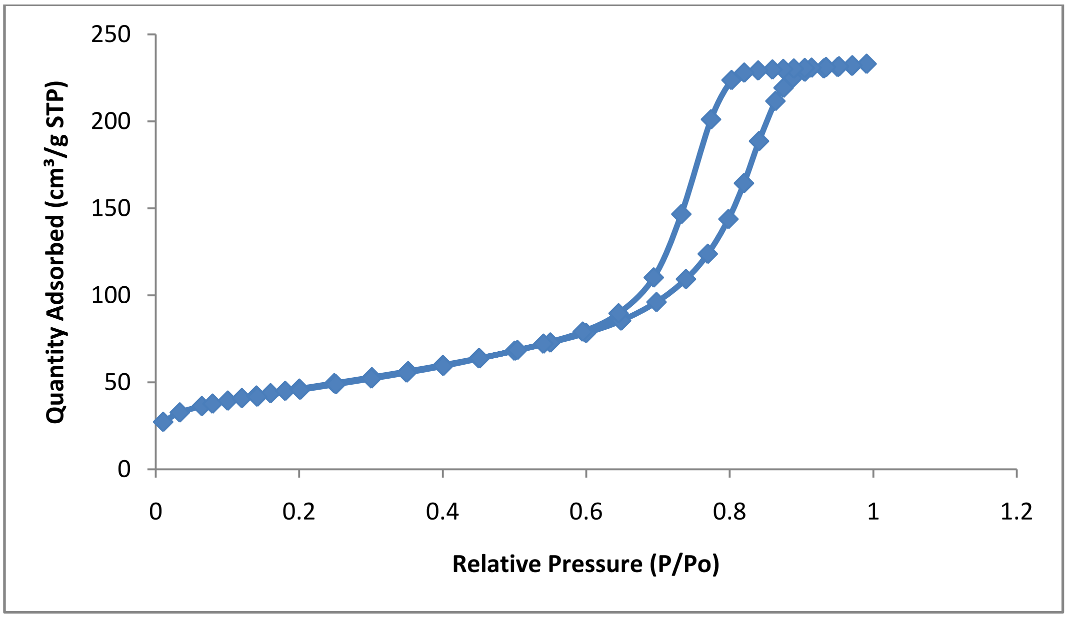

Figure 4.2(a): N_2 adsorption-desorption isotherms of synthesized aluminium oxide

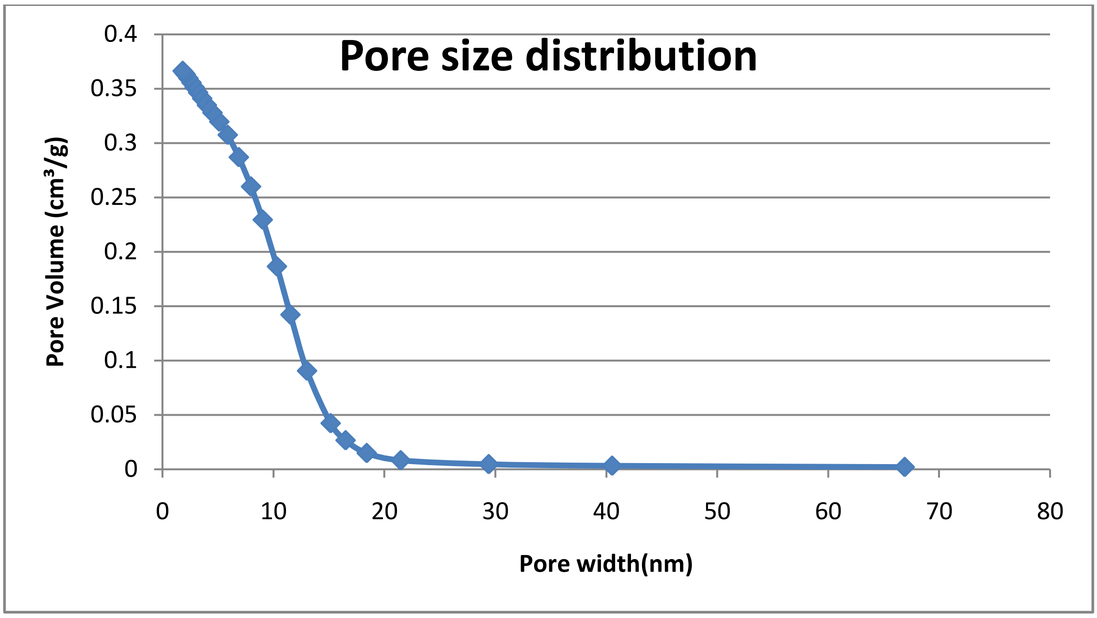

Figure 4.2(b): Pore size distribution for synthesized aluminium oxide

4.1.3. X-ray diffraction (XRD)

The XRD patterns of synthesized aluminium oxide and commercial aluminium oxide are as shown in Figure 4.3. Both of the patterns show the increase in intensity when approaching low angle. The synthesized aluminium oxide shows higher intensity at lower angle compared to the commercial aluminium oxide. This indicates that synthesized aluminium oxide probably has well organized pores distribution as compared to the commercial aluminium oxide [13]. As discussed in 4.1.1, the structure of synthesized aluminium oxide is more porous and can be seen from the FESEM image.

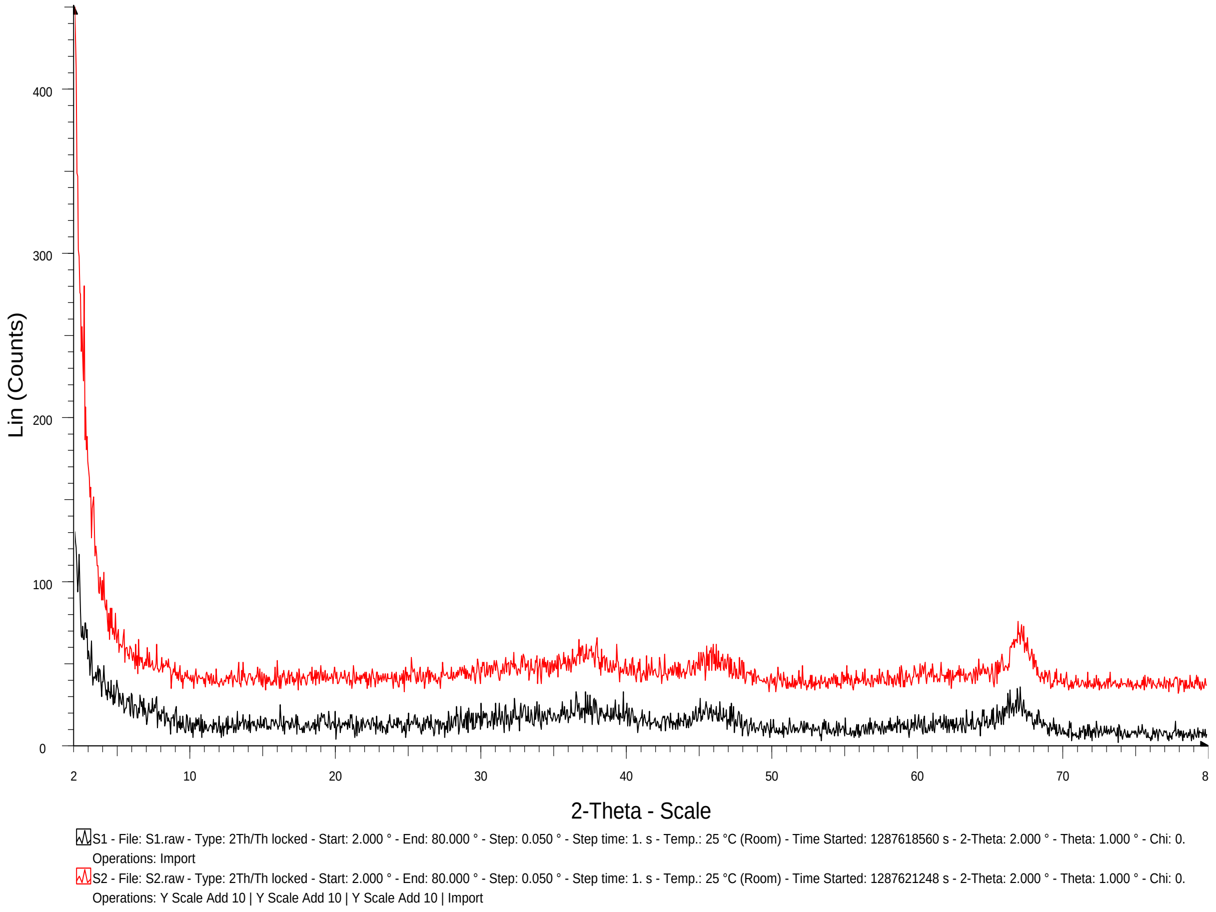

Figure 4.3: XRD pattern for synthesized aluminium oxide and commercial aluminium oxide

Both of the aluminium oxides is in γ- phase. Aluminium oxide in this phase has platy crystal with sharp edge [14]. The crystal has porous structure and can be seen in Figure 4.1(c). However, the porous structure was hardly to be seen from the commercial aluminium oxide. The material might in the γ-phase but has small amount of pores and not well organized. The material might transformed to γ-phase after being calcined at 700^{o}C. Calcination might be the factor that results in γ-phase aluminium oxide.

4.2. Adsorption Test

The adsorption process towards the removal of lead via adsorption process using the synthesized aluminium oxide was evaluated based on the contact time and pH of the solution. The results for each of the parameters were compared with the commercial aluminium oxide.

Equation 4.1 calculates the optimum parameter in each experiment. Percentage of lead uptake at each time interval can be obtained from this equation. The loading of the adsorbate can be assessed by obtaining the percentage of the adsorbate adsorbed.

$$\boxed{\text{(\%) Percentage of Uptake} = \frac{C_o - C_1}{C_o} \times 100} \qquad \text{Equation 4.1}$$

Where

C_o is the initial concentration

C_1 is the final concentration

4.2.1. Effect of contact time

The effect of contact time test was carried out by using 1 g of aluminium oxide in 250 ml of lead solution. The solution was continuously stirred in the water-bath shaker to ensure the uniform distribution of adsorbent. The sample was taken at time interval of 0, 5, 10, 15, 30, 60, 120 and 180 minutes. Based on the literature review, the adsorption of the lead using any adsorbent, the equilibrium was after 30 minutes. The pH of the solution was maintained at pH 5 and the temperature is at 27^oC.

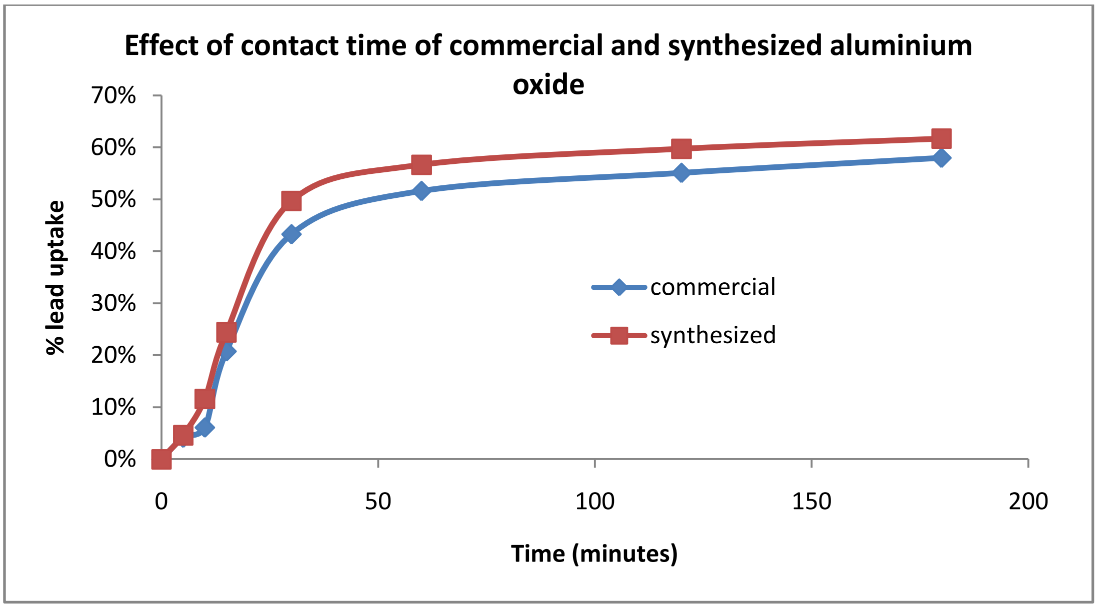

Figure 4.4: Effect of contact time of commercial and synthesized aluminium oxide

Effect of contact time is to study the rate of the adsorption process that an adsorbent can adsorbed. Generally, the rate of adsorption follow the similar trend which the uptake is rapid at the beginning and starting to slower down after the adsorbent is about to reach its equilibrium. Effect of contact time studied of commercial and synthesized aluminium oxide as in Figure 4.2.1 shows that the equilibrium of the adsorption is around 30 minutes. It follows the similar trend of adsorption process which the rapid adsorption activity was at the initial of the process.

The result shown in Figure 4.2.1 describes the adsorption of lead using synthesized aluminium oxide is higher than the commercial aluminium oxide. The rate of the lead uptake by synthesized adsorbent is higher than the commercial adsorbent. The lead uptake for both of the adsorbent was getting slower after 30 minutes. However the lead uptake still occurs up to 180 minutes at slower rate.

The adsorption generally is monolayer on the surface of the adsorbent [10]. Lead uptake was due to the surface force between the lead ion and aluminium oxide surface. This physical adsorption was due to the existence of the Van der Waals force that attracts the lead ion to the surface of the adsorbent. Most lead adsorbed at pores of the adsorbent.

Since the adsorption process occurs monolayer, the adsorption rate will decrease after sometimes as the pores and surface of the adsorbent is filled with the lead ion. The adsorption cannot occur at multilayer adsorption due to the weak force between the surface and the adsorbate.

Furthermore, another driving force for the adsorption process is the ratio between the bulk solution and the solid liquid interface. Initially the bulk lead ion concentration is higher, meaning it enables higher adsorption rate. After 30 minutes, the concentration of lead ion in the solution was getting lesser, giving a smaller adsorption rate.

Lesser lead ion concentration in the solution means that the diffusion of lead ion into the pores of the adsorbent also decreases. This lead to lower adsorption rate because no lead ion can be adsorbed at the pores. If the study was continued after 180 minutes, the percentage lead uptake will maintain at a certain value. At this particular time, the lead ion is no longer adsorbed because the adsorbent is fully saturated. At this particular time, the adsorption has reached equilibrium.

The rate of adsorption and the percentage of lead uptake for synthesized aluminium oxide are higher compared to commercial aluminium oxide. It reflects the characteristic of the adsorbent. The porosity and surface area of the synthesized aluminium oxide are higher than commercial aluminium oxide as discussed in 4.1.

4.2.2. Effect of pH of solution

pH of the solution is one of the parameters that need to be investigated. Adsorption process is influenced by the pH of the solution. Different element with different adsorbent favor different acidity of the solution. pH of the solution determines the activity and the force of the adsorbent.

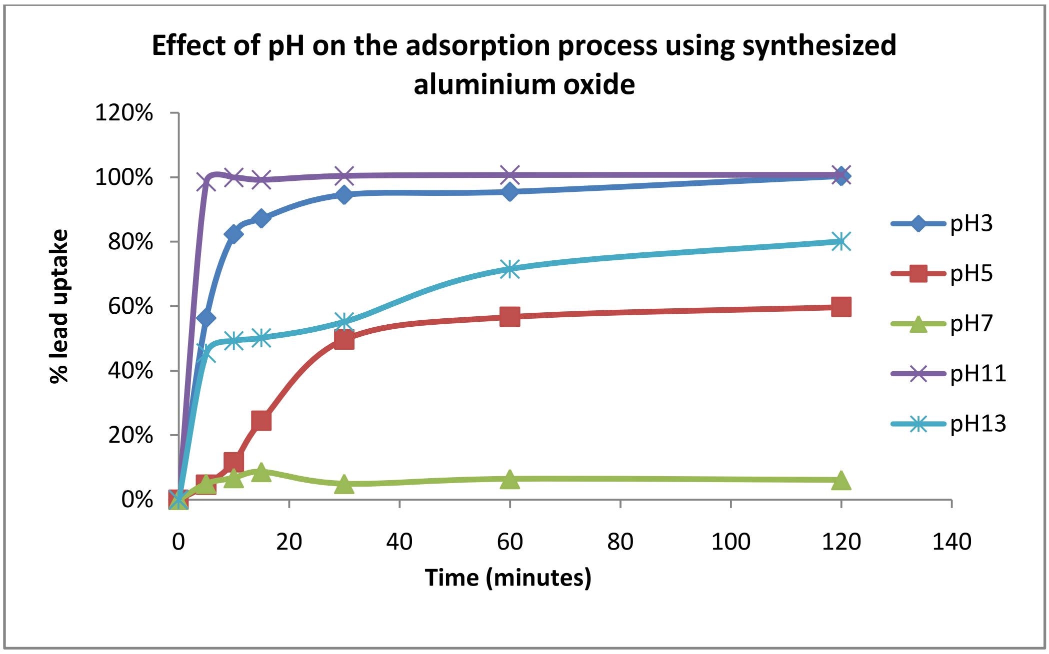

Figure 4.5: Effect of pH on the adsorption of lead by synthesized aluminium oxide

Based on Figure 4.5, the adsorption of lead using synthesized aluminium oxide is highest at pH 11 and lowest at pH7. The adsorption of lead was at highest rate for first 10 minutes at pH 11. The rapid uptake then slow down after that and maintain at 100% uptake meaning that all lead ion have been adsorbed.

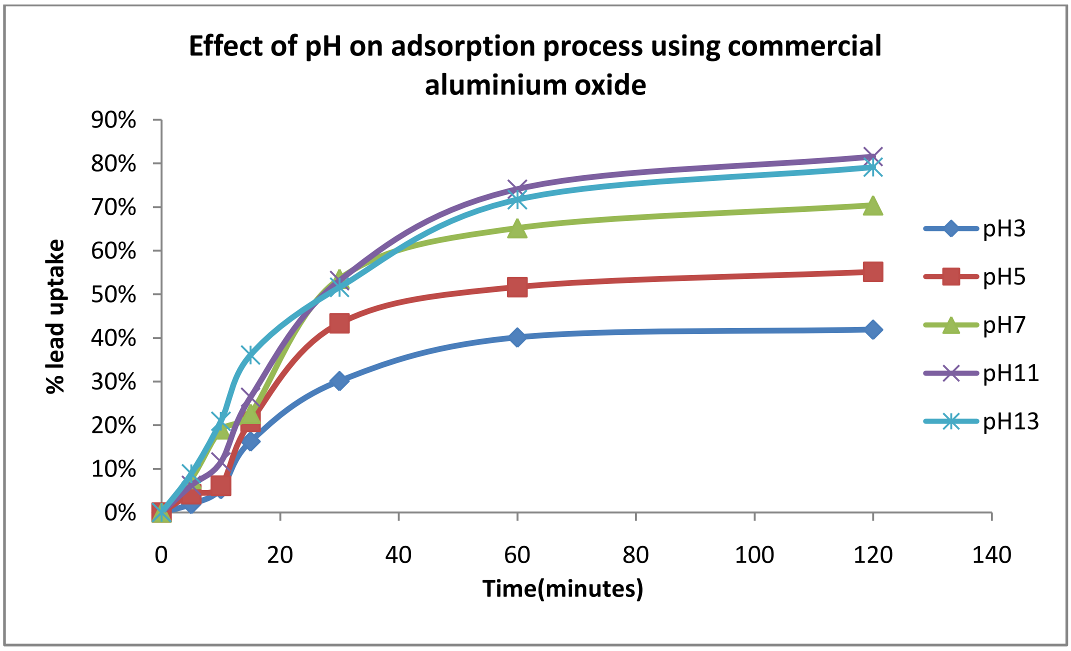

Figure 4.6: Effect of pH on adsorption process using commercial aluminium oxide

Figure 4.6 shows the effect of pH on adsorption process for commercial aluminium oxide. The adsorption is highest at pH 11 while the lowest is at pH 3. Rate of adsorption for the initial 30 minutes of pH 13 was the highest compared to others. However, the rate was slower and maintained at 80% which is lower than adsorption at pH 11.

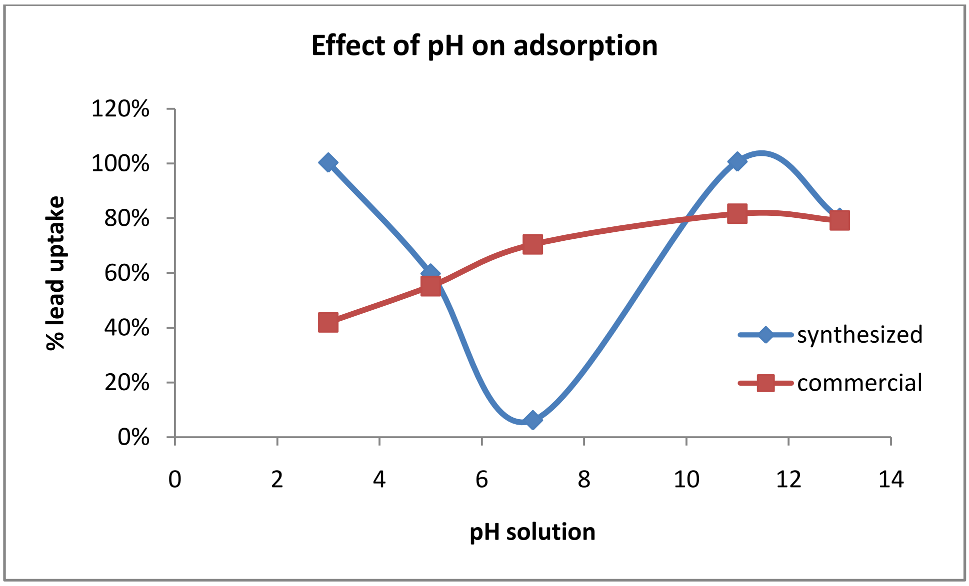

Figure 4.7: Effect of pH on adsorption

The adsorption of lead at different pH for both of the adsorbent shows different trend. Adsorption using commercial aluminium oxide shows increasing trend of adsorption percentage uptake with the increasing of pH. The rate of the adsorption at initial state also increased with increase pH of the solution. There is no specific trend for adsorption using synthesized aluminium oxide as shown in Figure 4.7. The adsorption of lead is high at pH 3 and decreasing at pH7 before increase again at pH11.

pH of the solution affect the solubility of lead in water. The decrease of acidity of the solution probably lead to stronger force for the commercial aluminium oxide. The decrease in acidity has influenced the adsorption in a way that the surface can attract more lead ion. This can be explained by the existence of positive hydrogen ion, H^+. At lower pH, the amount of the H^+ is high and it occupied the surface of the adsorbent making the attractive force towards the lead ion is lower.

It is contrast with the synthesized aluminium oxide, high acidity and high alkaline result in higher adsorption uptake. The high adsorption uptake at the higher pH can be explained as the same way for the commercial aluminium oxide discussed above. High adsorption uptake at lower pH of the solution probably influenced the adsorption activity at the pores of the adsorbent. The actual reason of the result is hard to be discussed.

CONCLUSION

The adsorbent prepared using simple precipitation method shown that it possesses properties as an adsorbent. Surface morphology and XRD analysis showed that both of the adsorbents were in crystals and in y-phase. The microstructure of the synthesized aluminium oxide show high porosity surface with higher surface area. Surface area of synthesized aluminium oxide is161.4 m2/g which is higher than commercial aluminium oxide (90-100 m2/g). Adsorption study carried out at different parameters concludes that synthesized aluminium oxide is more effective than commercial aluminium oxide. The adsorption rate for lead removal using both synthesized aluminium oxide and commercial aluminium oxide were highest at first 30 minutes. Adsorption was highest at pH 3 and 11 for synthesized aluminium oxide where as for commercial aluminium oxide, adsorption was highest at pH 13. The adsorption performance for lead removal may improve by using the aluminium oxide synthesized by simple precipitation method.

RECOMMENDATION

There are few improvements can be made to continuously improve the performance of the aluminium oxide. Further study should be carried out to investigate the adsorption process at different adsorbent dosage and concentration of solution. Investigation should also be carried out to study the suitability and effectiveness of the synthesized aluminium oxide to be used in packed bed column with continuous flow fluid.

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APPENDIX I

Figure A1 : Formation of gel-like after the titration of aluminium chloride with ammonium hydroxide

Figure A2: Aluminium Oxide after the drying process

Figure A3: Aluminium Oxide after calcination

APPENDIX II

BET Raw Data

Table A1:Summary of BET data

Full Report Set				
ASAP 2020 V3.04 H	Unit 1	Serial #: 1161	Page 1	
Sample:	000-083 AO-S mohamad saiful			
Operator:				
Submitter:				
File:	C:\2020\DATA\000-083.SMP			
Started:	9/30/2010 3:16:20PM	Analysis Adsorptive:	N_2	
Completed:	9/30/2010 10:47:53PM	Analysis Bath Temp.:	-195.850 °C	
Report Time:	10/1/2010 9:11:17AM	Thermal Correction:	No	
Sample Mass:	0.1696 g	Warm Free Space:	28.0956 cm³ Measured	
Cold Free Space:	83.9987 cm³	Equilibration Interval:	10 s	
Low Pressure Dose:	None	Automatic Degas:	Yes	
Summary Report				
Surface Area				
Single point surface area at P/Po = 0.248583679:	161.4369 m²/g			
BET Surface Area:	166.0590 m²/g			
Langmuir Surface Area:	244.1813 m²/g			
t-Plot Micropore Area:	11.1659 m²/g			
t-Plot External Surface Area:	154.8931 m²/g			
BJH Adsorption cumulative surface area of pores between 17.000 Å and 3000.000 Å width:	184.517 m²/g			
BJH Desorption cumulative surface area of poresbetween 17.000 Å and 3000.000 Å width:	206.9448 m²/g			

Pore Volume				
Single point adsorption total pore volume of pores less than 2070.641 Å width at P/Po = 0.990578692:	0.360317 cm^3/g			
Single point desorption total pore volume of pores less than 678.071 Å width at P/Po = 0.970617038:	0.358984 cm^3/g			
t-Plot micropore volume:	0.003825 cm^3/g			
BJH Adsorption cumulative volume of pores between 17.000 Å and 3000.000 Å width:	0.366289 cm^3/g			
BJH Desorption cumulative volume of pores between 17.000 Å and 3000.000 Å width:	0.364433 cm^3/g			
Pore Size				
Adsorption average pore width (4V/A by BET):	86.7926 Å			
Desorption average pore width (4V/A by BET):	86.4715 Å			
BJH Adsorption average pore width (4V/A):	79.405 Å			
BJH Desorption average pore width (4V/A):	70.441 Å			

Table A2:Isotherm Tabular Report

Relative Pressure (P/Po)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm³/g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
			01:19	754.3743
0.010095	7.616033	27.11062	01:34	
0.033257	25.09295	32.55549	01:40	
0.064024	48.30787	36.20341	01:43	
0.078953	59.57439	37.58778	01:47	
0.100126	75.55203	39.32148	01:50	
0.119793	90.39516	40.81263	01:53	
0.140096	105.7179	42.23286	01:56	
0.159784	120.5775	43.56959	01:59	
0.180204	135.9909	44.92199	02:02	
0.199899	150.8572	46.20043	02:05	
0.248584	187.6031	49.35302	02:08	
0.301026	227.1884	52.7879	02:12	
0.351555	265.332	56.26181	02:16	
0.400036	301.9302	59.78712	02:19	
0.44991	339.5878	63.68882	02:24	
0.499881	377.3182	67.97395	02:28	
0.549752	414.9758	72.72788	02:32	
0.599572	452.6008	78.19891	02:37	
0.648426	489.5006	85.31161	02:42	
0.697726	526.7486	95.98673	02:49	
0.738771	557.7731	109.2149	02:57	
0.769194	580.7919	123.699	03:07	
0.797979	602.5826	143.6989	03:18	
			03:20	755.149
0.819582	619.0176	164.3326	03:33	
0.840604	635.0184	188.5156	03:47	
0.863448	652.3922	211.535	04:00	
0.874856	661.0936	219.1776	04:09	
0.888119	671.1716	225.0011	04:15	
0.90449	683.5812	228.3885	04:19	
0.930877	703.553	230.2851	04:22	
0.950293	718.2471	231.0673	04:24	
0.97026	733.3589	231.7552	04:26	
0.990579	748.7372	232.9437	04:28	
0.970617	733.6693	232.082	04:30	
0.952093	719.6973	231.5109	04:33	
0.933989	706.0314	231.0888	04:35	
0.913806	690.7936	230.7129	04:37	
0.904374	683.6823	230.502	04:39	
0.889113	672.1643	230.2282	04:41	

0.87434	661.0233	229.9594	04:44	
0.859307	649.6757	229.6598	04:46	
0.839402	634.6445	229.1304	04:48	
0.819965	619.9741	227.888	04:51	
0.802323	606.6771	223.6112	04:56	
0.773695	585.1508	201.0126	05:11	
			05:31	756.5157
0.732319	554.0109	146.5344	05:45	
0.693844	524.9039	110.1122	06:04	
0.644842	487.8334	89.49503	06:16	
0.594869	450.0274	78.96724	06:24	
0.540134	408.6198	72.04306	06:29	
0.503796	381.1297	68.43551	06:33	
0.451055	341.2304	63.6201	06:38	
0.400467	302.9595	59.25747	06:43	
0.349523	264.4194	55.44387	06:47	
0.300337	227.2099	52.02915	06:51	
0.250827	189.7545	48.76172	06:56	
0.20094	152.0141	45.53205	07:00	
0.141225	106.8386	41.56489	07:03	

Table A3:BJH Adsorption Cumulative Pore Volume

Halsey : Faas Correction	
000-083 AO-S mohamad saiful	
Pore Width (Å)	Pore Volume (cm³/g)
668.8373	0.002025
405.1464	0.00321
293.9207	0.004612
214.5992	0.008187
183.9612	0.01485
164.923	0.026592
151.4525	0.042259
130.161	0.090475
115.2267	0.142093
103.0363	0.186453
90.23862	0.229386
79.67859	0.259935
68.67292	0.286922
58.69782	0.307426
51.09962	0.319554
44.92835	0.327834
39.87087	0.334795
35.61734	0.340945
31.9745	0.346371
28.86827	0.351087
25.9748	0.355511
23.24432	0.359489
20.87793	0.362807
19.94892	0.363994
18.99364	0.365193
18.07293	0.366289

Table A4:BJH Adsorption Pore Distribution Report

Faas Correction Halsey					
t = 3.54 [-5 / ln(P/Po)] ^ 0.333					
Width Range:	17.000 Å to 3000.000 Å				
Adsorbate Property Factor:	9.53000 Å				
Density Conversion Factor:	0.0015468				
Fraction of Pores Open at Both Ends:	0.00				
Pore Width Range (Å)	Average Width (Å)	Incremental Pore Volume (cm^3/g)	Cumulative Pore Volume (cm^3/g)	Incremental Pore Area (m^2/g)	Cumulative Pore Area (m^2/g)
2069.4 - 668.8	794.936	0.002025	0.002025	0.101903	0.101903
668.8 - 405.1	473.8266	0.001185	0.00321	0.100054	0.201958
405.1 - 293.9	331.4148	0.001402	0.004612	0.169205	0.371162
293.9 - 214.6	241.4398	0.003574	0.008187	0.592174	0.963337
214.6 - 184.0	196.7621	0.006663	0.01485	1.354583	2.317919
184.0 - 164.9	173.3204	0.011742	0.026592	2.709943	5.027863
164.9 - 151.5	157.5645	0.015667	0.042259	3.97718	9.005043
151.5 - 130.2	139.0547	0.048216	0.090475	13.86966	22.8747
130.2 - 115.2	121.6941	0.051618	0.142093	16.96641	39.84111
115.2 - 103.0	108.3765	0.044361	0.186453	16.37275	56.21386
103.0 - 90.2	95.69162	0.042933	0.229386	17.94654	74.16041
90.2 - 79.7	84.21874	0.030549	0.259935	14.50936	88.66977
79.7 - 68.7	73.24768	0.026986	0.286922	14.73709	103.4069
68.7 - 58.7	62.78688	0.020504	0.307426	13.06239	116.4692
58.7 - 51.1	54.28471	0.012128	0.319554	8.936677	125.4059
51.1 - 44.9	47.54441	0.00828	0.327834	6.966018	132.3719
44.9 - 39.9	42.03759	0.006962	0.334795	6.624487	138.9964
39.9 - 35.6	37.45265	0.00615	0.340945	6.568164	145.5646
35.6 - 32.0	33.55408	0.005426	0.346371	6.467989	152.0326
32.0 - 28.9	30.22351	0.004716	0.351087	6.241145	158.2737
28.9 - 26.0	27.22869	0.004425	0.355511	6.5	164.7737
26.0 - 23.2	24.41569	0.003977	0.359489	6.516268	171.29
23.2 - 20.9	21.89632	0.003318	0.362807	6.061983	177.352
20.9 - 19.9	20.38556	0.001187	0.363994	2.328704	179.6807
19.9 - 19.0	19.4402	0.001199	0.365193	2.466554	182.1472
19.0 - 18.1	18.50276	0.001096	0.366289	2.37023	184.5175

APPENDIX III

AAS Raw data

Table A5:Raw data of adsorption kinetic

time(mins)	concentration (ppm)		Percentage uptake (%)	
	commercial	synthesized	commercial	synthesized
0	40	40	0%	0%
5	38.3421	38.1464	4%	5%
10	37.5546	35.3581	6%	12%
15	31.6886	30.2161	21%	24%
30	22.6728	20.1218	43%	50%
60	19.3421	17.3266	52%	57%
120	17.9481	16.0981	55%	60%
180	16.7908	15.3186	58%	62%

Effect of pH on adsorption

Table A6:Effect of pH solution on adsorption (synthesized aluminium oxide)

Time (mins)	Concentration (ppm)					Percent uptake(%)				
	pH3	pH5	pH7	pH11	pH13	pH3	pH5	pH7	pH11	pH13
0	**40.0000**	**40.0000**	**40.0000**	**40.0000**	**40.0000**	0%	0%	0%	0%	0%
5	**17.4574**	**38.1464**	**38.0172**	**0.5733**	**21.8045**	56%	5%	5%	99%	45%
10	**7.0624**	**35.3581**	**37.3026**	**0.0378**	**20.2855**	82%	12%	7%	100%	49%
15	**5.1093**	**30.2161**	**36.5466**	**0.3213**	**19.9235**	87%	24%	9%	99%	50%
30	**2.1798**	**20.1218**	**38.0271**	**-0.1669**	**17.9299**	95%	50%	5%	100%	55%
60	**1.8018**	**17.3266**	**37.4286**	**-0.2772**	**11.3779**	95%	57%	6%	101%	72%
120	**-0.1512**	**16.0981**	**37.5546**	**-0.2929**	**7.9444**	100%	60%	6%	101%	80%

Table A7: Effect of pH solution on adsorption (commercial aluminium oxide)

Time (mins)	Concentration (ppm)					Percent uptake(%)				
	pH3	pH5	pH7	pH11	pH13	pH3	pH5	pH7	pH11	pH13
0	**40.0000**	**40.0000**	**40.0000**	**40.0000**	**40.0000**	0%	0%	0%	0%	0%
5	**39.2343**	**38.3421**	**36.9299**	**37.5237**	**36.4543**	2%	4%	8%	6%	9%
10	**37.8623**	**37.5546**	**32.3779**	**35.3745**	**31.6363**	5%	6%	19%	12%	21%
15	**33.4937**	**31.6886**	**30.9444**	**29.4364**	**25.5634**	16%	21%	23%	26%	36%
30	**27.9539**	**22.6728**	**18.5733**	**18.7334**	**19.3564**	30%	43%	54%	53%	52%
60	**23.9573**	**19.3421**	**13.9237**	**10.3463**	**11.3233**	40%	52%	65%	74%	72%
120	**23.2394**	**17.9481**	**11.8345**	**7.3643**	**8.3453**	42%	55%	70%	82%	79%