ALUMINIUM SULPHATE (ALUM) RECOVERY BY ACIDIFICATION ON THE WATER TREATMENT SLUDGE

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ALUMINIUM SULPHATE (ALUM) RECOVERY BY ACIDIFICATION ON THE WATER TREATMENT SLUDGE

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A thesis submitted in fulfillment of the
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I declared that this thesis entitled 'Aluminium Sulphate (Alum) Recovery By Acidification On The Water Treatment Sludge' is the result of my own research except as cited in the references. The thesis has not been accepted for any degree is not concurrently submitted candidature of any degree.

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Special dedicated to my beloved mother and father

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ABSTRACT

Aluminium sulphate (alum) is a solution added to the water to coagulate solids particles in the water. It is to make sure water that sent for domestic use is free of solids particles. This process is called coagulation process. Aluminium sulphate (alum) that coagulated with solids particles forming alum sludge. Alum sludge settles in the bottom of water treatment plant together with the solid particles.

The significant of this study is to recover aluminium sulphate (alum) from alum sludge. Acidification process applied to alum sludge to recover aluminium sulphate (alum). Acidification using sulphuric acid will separate particulate solids and aluminium sulphate(alum). Aluminium sulphate (alum) will be recovered in aqueous solution, while particulate solids will remain in solid form. The research also done to obtain the optimum pH for acidification to obtain the best volume recovered of aluminium sulphate (alum).

There are some other factors to recover aluminium sulphate (alum) at the optimum point that it has to do with stirring speeds and centrifugation time. These factors will give effects to the precipitation period, aluminium sulphate (alum) recovery and the percentage of aluminium recovery efficiency.

From the experiments, the best volume recovered is at 2.5 of pH values, 100 rpm stirring speeds and 40 minutes centrifugation time. The result shows 89.2 ml recovered from 100 ml sample of aqueous aluminium sulphate, 16 hours precipitation period, 1.29 mg/L Al^{3+} recovery in the solution which brings to 39.7% efficiency of recovery.

ABSTRAK

Aluminium sulfat (alum) adalah larutan yang dicampurkan ke dalam air untuk memendapkan pepejal yang terampai di dalam air. Ianya untuk memastikan air yang disalurkan kepada pengguna adalah bebas daripada pepejal terampai. Proses ini dipanggil process pengentalan/koagulasi. Aluminium Sulfat (alum) yang bertindak balas dengan pepejal terampai tersebut setelah termendap dipanggil lumpur alum. Lumpur alum ini termendap di bahagian bawah loji rawatan air bersama dengan pepejal terampai.

Kepentingan kajian ini adalah untuk mendapatkan kembali aluminium sulfat (alum) dari lumpur alum tersebut. Proses pengasidan digunakan untuk mendapatkan kembali aluminium sulfat (alum). Asid sulfuric digunakan untuk memisahkan aluminium sulfat (alum) dan pepejal yang terampai. Aluminium sulfat (alum) didapati dalam larutan akues manakala pepejal terampai kekal dalam fasa pepejal. Kajian ini juga dijalankan untuk memutuskan nilai pH yang paling optimum untuk mendapatkan kembali aluminium sulfat (alum) dalam isipadu yang paling banyak.

Faktor-faktor lain yang menyumbang kepada keadaan optimum bagi mendapatkan kembali aluminium adalah kelajuan pengadukkan dan masa pemendakan. Faktor-faktor ini memberi kesan terhadap tempoh pemendapan, dan isipadu aluminium sulfat yang didapati dan peratus aluminium sulfat dalam larutan akues tersebut.

Daripada eksperiemen, isipadu yang paling banyak didapati pada nilai pH 2.5, 100 rpm kelajuan pengadukkan dan 40 minit masa pemendakkan. Keputusan menunjukkan 89.2 ml daripada 100 ml sample adalah larutan akues aluminium sulfat, dalam tempoh pemendapan 16 jam dan isipadu aluminium tulen adalah 1.29 mg/L Al^{3+} dan peratus aluminium yang didapati adalah 39.7%.

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LIST OF SYMBOLS

$Al_2(SO_4)_3$ -Aluminium Sulphate

$Al(OH)_{3(s).}$ - Aluminium hydroxide

H_2SO_4 - Sulphuric acid

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Coagulation is critical process in drinking water treatment involving colloid charge neutralization followed by aggregation into flocs that are amenable to solid/liquid separation with subsequent processes such as acidification and filtration. The most coagulant used in water treatment is alum $Al_2(SO_4)_3 14\text{-}H_2O$, due to its effectiveness in treating a wide range of water type and relatively low cost.

Alum function as a coagulant by forming positively charged Al species that adsorb to negatively charged natural particles resulting in charge neutralization. These species are thought to be primarily monomeric, short lived and quick to precipitate to amorphous aluminium hydroxide, $Al(OH)_{3(s).}$ in most cases, the aluminium hydroxide sols are formed so rapidly (1-2s) that these amorphous solid species are responsible for the charge of neutralization of natural particles. Within the coagulation pH range of 6.0 to 7.5 that is typical in water treatment processes, these aluminium hydroxide sols are usually present in significant excess relative to natural particles. [Ronald Hart, 2003]

Water treatments in Malaysia basically use alum as a coagulant. Alum is effectively works in preserving clarity in water. Also in economy alum is much cheaper than other coagulation supernatant or membrane technology which used in Singapore.

The chemical bond in alum can treat supernatant in chemical form better than membrane which filter all the nutritious and mineral salt that human need in drinking water. So the water become over treated that result in malnutrition form which gives problems to human health. That is why alum is the common use of coagulant in some developed country.

Two very different approached for coagulant recovery in a related objectives. First approached is considering acid or alkaline condition as a sludge conditioning technique with improved thickening, dewatering and waste sludge reduction as major objectives. This approached is referred to as a sludge conditioning, although it is frequently described as coagulant recovery and reused of a aluminium contained in the sludge suspensions, with the objective of minimizing expenditure of commercial coagulant recovery. [AWWA, Research Foundation, 1993]

1.2 Problems Statements

Removing suspended particles in water treatment plant is crucial. So, the purpose of alum is beneficially used to remove colloid particles. Without any recovery alum will settle down beneath the lagoon together with sludge. Some water plant discharged alum as waste water that finally will bring pollution to environment. A review of types and volumes of wastewater discharged from water treatment plants in Malaysia would no doubt prove that alum-waste quantities are greatest.

Problem persists when ion hydroxide in the sludge will form aluminium hydroxide with alum that is usually permeable to filtration. Aluminium hydroxide gelatinous in nature and will remain in its semi-fluid state indefinitely unless something is done to change its physical characteristic. Although hydroxide sludge does settle down, it is almost impossible to dewater without prior treatment. [Fulton, 1974

The problem will become more serious if alum flooded in waste water tank as there will be no other place to dump alum-waste. So, in preventing environment pollution which will possibly bring diseases, alum has to be recycled and use again in water treatment plant. This measure not only can prevent pollution but can bear much cheaper alum in market. Alum recovery gives two benefits in one related sense.

1.3 Objectives

The main objective of this study is to recover aluminium sulphate (alum) from alum sludge using acidification process. In order to achieve this objective, there lies partitions of objectives which are to study the effect of acidification using different pH values, to study the effect of stirring speeds and to study the effect of centrifugation time. It will lead to the optimum conditions of aluminium sulphate recovery by acidification process.

1.4 Scopes

The scopes of this study is to make an experiments to determine the effect of aluminium recovery using different pH values for acidification process, different speeds of stirring and different centrifugation times. These experiments then will give the effects to the volume of aluminium sulphate (alum) aquoeus recovered, precipitation periods of particulate solids and the volume of aluminium pure recovered and the percentage of aluminium sulphate recovery. The best condition of aluminium sulphate (alum) recovery will be determined using these effects.

CHAPTER 2

LITERATURE REVIEW

2.1 Water Treatment Plant

People all over the world use water in every day life. It is proven that human being cannot last for more than 24 hours drinking. People also consume water in other cleansing activity. The water that people consume must be clean visibly or invisibly to prevent any disease due to water contamination. So, government develops water treatment plant to ensure people use water that clean from any contamination. It is essential that the supply of water for human consumption should be free from unpleasant or harmful impurity and for this reason is subjected to various method of treatment to render it fits for use, either before or after distribution to domestic or trade users.

A typical conventional water treatment process involves separation of solid from liquid in six major steps:

2.1.1. Coagulation/ flocculation

Coagulation of waters to aid their clarification has been practiced since ancient times (Baker, 1981). The common coagulant used is alum and iron salt with alum being most extensively used agent. When neutral alum added in the water, the bicarbonates present setting free hydrated aluminium hydroxide which carries down organics.

2.1.2. Sedimentation

Sedimentation is the physical separation of suspended material from water by the action of gravity. It is a common operation for water treatment and found in most water treatment plant. It is much cheaper than many other treatment operations.

2.1.3. Filtration

Significant removal of bacteria and other microbes also occurs in filtration (Craun, 1988). Filtration is the bulwark of water treatment. Filtration is the operation of removing colloid particles in water using filters. The application of water treatment is preliminary treatment of raw water with high suspended solids content. Filters with very coarse media, known as roughing filters are used.

2.1.4. pH adjustment

The operation held to stabilize the pH value suitable to consumer. Normally the suitable pH value for domestic use is pH values of 7. So, pH adjustment is used to justify the pH values of water from the river to the required pH value using organic solutions of salt to stabilize the pH value of the water according to the present pH of water. The common use of solution is potassium carbonate or sodium carbonate in a very small amount to avoid effecting the water.

2.1.5. Fluoridation

The used of fluorine in the water for dental purpose. Through this process, fluorine is added in a particular measurement to strengthen teeth. Fluorine can prevent dental disease in small amount of measurements about 1 ppm, whilst fluorine treated with higher concentration (above 5 ppm) may produce mottled enamel, and lower concentration of fluorine (below 0.2 ppm) increase the accident in dental caries.

2.1.6. Disinfection

Disinfection is the process of vanishing the pathogenic microorganism in water. It is the protection measure of the public health from waterborne disease transmitted through water without disinfecting measure.

2.2 Coagulation Process

Coagulation and flocculation are used in both water and wastewater treatment processes. In water treatment it is usually cost effective to apply coagulation and flocculation to remove colloidal and small particles that settle slowly. Coagulation-flocculation can also be applied to enhance the removal of solid in highly concentrated natural waters that contain significant amounts of settleable solids. Commonly, presedimetation without coagulant addition or a roughing filter is used to remove high concentrations of settleable solids before coagulation-flocculation-sedimentation. [Ronald L. Droste, 2003]

The ability of an agent to coagulate water is related to its charge. The size of synthetic polymers is also a factor. There is more than order of magnitude increase in the effectiveness of an ion as its charge increases by one. This is a statement of the Schultze-Hardy rule based on the work of these two researchers in 1882 and 1900, respectively.

The most common coagulants are alum (aluminium sulphate) and alum being most extensively used agent. The multivalent characteristic of these cations strongly attracts them to charged colloidal particles and their relative insolubility ensures their removal to high degree.
[Ronald L. Droste, 2003]

2.3 Acidification Process

Alum and iron salts can be recovered from the sludge by adding acid. If sulfuric acid is used, the reaction is as stated below;

$$Me(OH)_x + xH^+ + x/2SO_4^{2-} \rightarrow Me(SO_4)_{x/2} + xH_2O$$

Other metals will be solubilized along with the coagulant metals that should again be precipitated along with the coagulant. The increased rate associated with acid coagulant recovery has decreased the frequency of this practice. Careful monitoring is required to ensure that product water does not exhibit elevated metals concentrations when coagulant recovery and reused practiced. [Ronald L. Droste, 2003]

Volume/solid reduction and coagulant recovery for sludge generated from drinking water treatment have been investigated in the past by acidification of sludge through addition of bases. Other than volme/solid reduction and coagulant recovery, acidification of sludge also enhanced the dewatering efficiency and settling velocity of sludge. If the $Al(OH)^3$ precipitates were dominant species inside sludge floc, the solid reduction such as reduction of suspended solid (SS), can be expressed as the dissolution of coagulants after acid or based addition according this equation of reaction;

$$2Al(OH)_3 + 3H_2SO_4 \leftrightarrow Al_2(SO_4)_3 + 6H_2O(l)$$

$$Al(OH)_3 + NaOH \leftrightarrow Al(OH)_4 + Na(2)$$ [Peter Chen, 2003]

2.4 Acidification and Alkalization of Sludge

Coagulation process held in water treatment sludge to remove suspended solids, color and organic materials that finally forms as a large portion of sludge. After water flowed to the consumers, of the clean water and the treatment period has been done. The sludge in the water treatment plant disposed after dewatering process done. This sludge forms melted cake like forms called sludge cake. With the composition of aluminium hydroxide, $Al(OH)_3$ in the sludge, the sludge is melted as it is not hardened due to drying of sunrays. So, the sludge remains melted for a quite of some period. As the landfill for disposing this sludge become limited, we need a method to recover alum contained in sludge to make the sludge disposable for preventing environment pollution.

Volume or solid reduction and coagulant recovery for sludge generated after drinking water has been served to the customers had been done by many researchers before. It uses the process of acidification of sludge through addition of sulfuric acids and alkalization through addition of bases.

If $Al(OH)_3$ precipitates were dominant species inside the sludge floc, the solid reduction reduction of suspended solids can be expressed as the dissolution of coagulants after acid or base addition according to following equations:

$2Al(OH)_3 + 3H_2SO_4 \leftrightarrow Al_2(SO_4)_3 + 6H_2O(l)$
$Al(OH)_3 + NaOH \leftrightarrow Al(OH)_4 + Na(2)$

2.5 The Efficiency Determination of Recovery

The efficiency of coagulant recovery and solid reduction depended on the pH values [Abdo, 1993, Masschelein, 1985]. This has proved that solid reduction is proportional to the amount of aluminium recovery. The efficiency of aluminium Al(III) recovery can be calculated as the following formula:

Efficiency of Al recovery

$$= \frac{[Al^{3+}] \text{ in supernatant after acidification}}{[H] \text{ added}}$$

According to this formula, we calculate the efficiency of aluminium, Al(III) recovered through experiment that we determine the concentration of aluminium recovered over acid concentration added during the experiment.

From chemical reaction yield, the theoretical value of the efficiency is 0.33 as if no other by product exists during the reaction. Theoretical values for sulfuric acid added to the sludge will be at pH ranging from 2 to 2.5.

In my studies, pH values of 2.3421 are quite close to 0.32512 of efficiency which is closed to the theoretical values. For pH values above 3 give the result lower than 0.25 value of efficiency.

In order to get the specific result, some amendment had been done to the efficiency formula to gain analytical result. This is due to the cause that sludge tested in experiment containing organic materials which might consume H^+ through protonation reactions. Thus, the formula for calculating efficiency of Al(III) recovery should be rewritten as below equation:

$$\text{Efficiency of Al recovery} = \frac{[Al^{3+}]}{[H]\,Al^{3+} + [H]\,org + [H]\,pH}$$

Where $[H]\,Al^{3+}$ is the amount of acids used for dissolution of Al(III) while $[H^+]$ org and $[H^+]$ pH are for protonation reactions of organics during acidification process and acid needed for reducing the pH, respectively. When the sludge containing relatively small amount of organic materials such as sludges produced from drinking water treatment [Cornwell, 1979] or when the amount of acids used for dissolution of Al(III) is much greater than that for acid consuming materials other than those needed for dissolution of Al(III), a stoichiometry of 0.33 mole Al(III) dissolved or mole H^+ added can be derived from above equation.

This is the same value which has been reported by acidification of sludges generated from drinking water treatment plant [Cornwell, 1979].

The theoretical value of Al(III) recovery by addition of NaOH, which is 1.0 mole Al(III) dissolved/ mole H^+ added, can also derive from equation, other than volume/ solid reduction and coagulant recovery.

The percentage of Al(III) recovery, which is defined as the weight ratio of Al(III) in supernatant of treated sludge that in the raw (untreated) sludge after acid digestion as indicated.

The percentage of Al(III) recovery can be calculated as in the below equation.

$$\text{Al recovery (\%)} = \frac{\text{Al in supernatant after acidification}}{\text{Al in raw sludge after acid digestion}}$$

These values, ranging from 5.8 to 66%, increase by decreasing pH for acidification and increasing pH for alkalization. This is consistent with others [Prakash, 2003] and with chemical reactions and 2 shown above, where addition of acids or base with push the reactions far right resulting in dissolution of Al(III).

The efficiency of Al(III) recovery which is defined as the moles of Al(III) dissolved per mole of H^+ or OH added as indicated. Protonation reaction, expressive way of efficiency of Al recovery as equation and the value will quite closed to the theoretical of 0.33.

$$[H]Al^{3+} + [H]\ org + [H]\ pH \sim [H]\ Al3^{+} = 0.33$$

On the other that, when [H] $Al^{3+}H$ + org + $[H^+]$ pH or [H] $Al^{3+} < [H^+]$ org + $[H^+]$pH, the values will be less than the theoretical value of 0.33. In the same term we can express Al recovery for alkalization sample as $[Al^3]$/ [OH].

According to chemical reaction theoretical value for alkalization is 1.0. Experimental value will be much lower theoretical value because the consumption of [OH] is relatively more due to the presence of particles in the sludge. Regarding deprotonation reaction the Al recovery equation has to be rewrite as below equation:

Efficiency of Al recovery

$$= \frac{[Al^{3+}]}{[OH]\ Al^{3+} + [OH]org + [OH]pH}$$

From the above equation, [OH] Al^{3+} is the amount of acids used for dissolution of Al(III) while [OH]org and [OH]pH are for deprotonation reactions of organic substances during alkalization process and for base needed to increase the pH sludge, respectively. If [OH] Al^{3+} is approximately equal to [OH]org + [OH]pH for the experimental conditions in current study, that we can express efficiency of Al recovery using equation below:

Efficiency of Al recovery

$$= \frac{[Al^{3+}]}{[OH]\ Al^{3+} + [OH]org + [OH]pH} \sim \frac{[Al^{3+}]}{2X[OH]\ Al^{3+}}$$

According to the above citation, we can expect the organics materials contained in the sludge consume more base during alkalization than acid during acidification (Chi, 2004).

2.6 Alum Characteristic

Flocs are made from complex arrangements of solid particles, hydroxide precipitates, hydroxide precipitate and water taken in during their growth. They have irregular shapes and rather loose structure to be made up of three basic units corresponding to three scales [Bottero, 1993].

In this three-tied particle structure notation, primary particles are supposed to group into clusters (level 1) contain dry solids and associated bound water. Aggregates (level 2) are produced by association of these clusters along with internal water, retained between clusters as part of the floc structure. Under low shear rates, aggregates form large-size whilst flocs have low shear strength and are easily ruptured by external forces, aggregates and clusters are characterized by significant shear strength. During mechanical sludge dewatering, the degree of floc breakage and deformation affecting the performances of the process therefore not only depends on the equipment used but also on the floc structure which has to be considered beside the traditional dewaterability indices for dewatering process optimization.

The flocs structure can be described by the size of the different basic units and by the volume floc fractal dimension D3. actually, fractal theory has been successfully used to explain the decrease of floc density with floc size [Mendelbrot, 1982], the number n of primary particles of diameter d_0 contained in a fractal floc of size *d* follows the power law relation:

$$n = k(d/d_0)^{D3} \quad (1)$$

Therefore, assuming that flocs are spherical and made of n primary particles and water, the floc density, p floc is given by:

p floc=m particle + m water/ V floc

$= \quad p_s n[(pi)d_o^3/6] + p_w[(pi)d^3/6 – n[(pi)d_o^3/6]/ (pi)d_o^3/6]$

Leading to

p floc = $p_w + k(d/d_o)^{D3} (p_s - p_w)$ (2)

where p_s and p_w are respectively the density of the primary particles and water. A fractal dimension D3 smaller than the Euclidean dimension 3 indicates an open structure with density decreasing with size d.

2.7 Case Study on Performance of Recovered Coagulant

The performance of partial recycling of coagulant was tested in a reduced scale Pulsator described earlier (Masschelein et al., 1981). A concentrated aluminium solution (e.g 0.4 g/l) is obtained by attack of sludge from the Tailfer plant with lime in a batch reactor. The coagulation flocculation is then performed by using the recovered coagulant together with fresh polyaluminium chloride. The indicative data of Table 2.5 are reported on a relative basis, to compare the performances of virgin and recycled coagulant in the educed scale flocculator. They are not in the same order of the quality standard reached in full scale plant operation. However, several conclusion can be drawn from the data on a relative basis and are the following up to 50% of coagulant recycling, no significant decreas in efficiency was observed, except during algal bloom periods; the final ph requires adjusting in order to reduce aluminium solubility.

This operation causes no difficulty in full scale treatment but has not however been included in this preliminary evaluation which has carried out during the early summer algal bloom period (the pH of the raw water ranges from 8.5 to 9); coagulant recycling should remain limited to 25-50% of the total dosing rate (Masschelin, 1984).

Table 2.5

		Settled water			
Number	% Recycled	ext. m.v. 250 nm (1/m)	suspended solids (mg/l)	Dissolved alt (µg/l)	Final pH↑
1	0	3.78	6.1	71	7.41
	25	3.35	3.35	103	7.64
	50	5.0	5.0	88	7.78
2	0	5.3	3.72	141	7.52
	45-50	4.76	2.90	110	8.04
3	0	4.46	3.05	110	7.48
	50	5.25	5.3	247	8.1
	100	7.38	12.1	2280	8.4

The data for each run are average volume of about three trials

↑ Filtered memples

2.8 Water Treatment Plant Operation

Clean water is essential for everyday life. Water treatment plant and system operators treat water so that it is safe to drink. Liquid waste treatment plant and system operators, also known as wastewater treatment plant and system operators, remove harmful pollutants from domestic and industrial liquid waste so that it is safe to return to the environment.

Water is pumped from wells, rivers, streams, and reservoirs to water treatment plants, where it is treated and distributed to customers. Wastewater travels through customers' sewer pipes to wastewater treatment plants, where it is either treated and returned to streams, rivers, and oceans or reused for irrigation and landscaping. Operators in both types of plants control equipment and processes that remove or destroy harmful materials, chemical compounds, and microorganisms from the water. They also control pumps, valves, and other equipment that moves the water or wastewater through the various treatment processes, after which they dispose of the removed waste materials.

Operators read, interpret, and adjust meters and gauges to make sure that plant equipment and processes are working properly. Operators operate chemical-feeding devices, take samples of the water or wastewater, perform chemical and biological laboratory analyses, and adjust the amounts of chemicals, such as chlorine, in the water. They use a variety of instruments to sample and measure water quality and they utilize common hand and power tools to make repairs to valves, pumps, and other equipment.

Water and wastewater treatment plant and system operators increasingly rely on computers to help monitor equipment, store the results of sampling, make process-control decisions, schedule and record maintenance activities, and produce reports. When equipment malfunctions, operators also may use computers to determine the cause of the malfunction and seek its solution.

Occasionally, operators must work during emergencies. A heavy rainstorm, for example, may cause large amounts of wastewater to flow into sewers, exceeding a plant's treatment capacity. Emergencies also can be caused by conditions inside a plant, such as chlorine gas leaks or oxygen deficiencies. To handle these conditions, operators are trained to make an emergency management response and use special safety equipment and procedures to protect public health and the facility. During these periods, operators may work under extreme pressure to correct problems as quickly as possible. Because working conditions may be dangerous, operators must be extremely cautious.

The specific duties of plant operators depend on the type and size of the plant. In smaller plants, one operator may control all of the machinery, perform tests, keep records, handle complaints, and perform repairs and maintenance. A few operators may handle both a water treatment and a wastewater treatment plant. In larger plants with many employees, operators may be more specialized and monitor only one process. The staff also may include chemists, engineers, laboratory technicians, mechanics, helpers, supervisors, and a superintendent.

Water pollution standards are largely set by two major Federal environmental statutes: the Clean Water Act, which regulates the discharge of pollutants, and the Safe Drinking Water Act, which specifies standards for drinking water. Industrial facilities that send their wastes to municipal treatment plants must meet certain minimum standards to ensure that the wastes have been adequately pretreated and will not damage municipal treatment facilities. Municipal water treatment plants also must meet stringent standards for drinking water. The list of contaminants regulated by these statutes has grown over time. As a result, plant operators must be familiar with the guidelines established by Federal regulations and how they affect their plant. In addition, operators must be aware of any guidelines imposed by the State or locality in which the plant operates.

2.9 Elementary Study of Aluminium

The symbol in title is Dalton's symbol for alumina, which was thought to be an element at the time.

Aluminium has the chemical symbol Al, atomic number 13, and atomic weight 26.98. The isotope with mass number 27 is the only stable isotope. It is a soft, light gray metal that resists corrosion when pure in spite of its chemical activity because of a thin surface layer of oxide. It is non magnetic and nonparking. Its density is 2.6989 g/cm^3,melting point 669.7^{o}C and boiling point 1800 ^{o}C. Its electrical resistivity is 2.824 µ-cm at 20 ^{o}C, with temperature coefficient 0.0039 ^{o}C^{-1}, the same copper's. its thermal conductivity is 2.37 W/cm-K at 300K, and the linear coefficient of expansion is 23.86 X 10^{-6} ^{o}C^{-1}. the specific is 0.2259 cal/g-K, and the heat of fusion is 93 cal/g. The first ionization potential is 1.67V positive with respect to hydrogen. When near its melting point, it becomes "hot short" and crumbles easily. As a pure metal, it is quite soft, and must be strengthened by alloying with Cu, Mg, Si or Mn before it can be used structurally. Aluminium bronze is 90 Cu, 10 Al, a strong , Golden-yellow alloy with excellent physical properties. The Young's modulus of pure aluminium is 10 X 10^6 psi, the shear modulus 3.8 X 10^6 psi, Poisson's ratio 0.33, and the ultimate tensile strength 10,000 psi, with 60% elongation. Pure aluminium is very ductile and malleable, and unsuitable as a structural material. Its hardness is 15 Brinell (500kg, 10 mm). The useful wrought alloys contain 1-7%

magnesium and 1% manganese. Its crystal form is face-centered cubic, with lattice constant a = 0.404 nm, and nearest-neighbor spacing of 0.286 nm.

Liquid aluminium easily absorb gases from air, and these gases are expelled on solidification, causing flaws in casting.

Casting alloys include silicone, and a little copper or nickel to help avoid flaws. Large crystals is maybe a problem if the aluminium, 0.156 inches per foot for casting alloys.

2.10 Chemical Properties of Aluminium

The electron configuration of aluminium is $1s^2$ $2s^2 2p^6 3s^2 3p$. The outer three electrons occupy three s^2p hybrid orbitals that point in orthogonal directions. These electrons easily form covalent bonds, as in anhydrous $AlCl_3$. This compound easily sublimates, showing that it is not ionic, and is partially hydrolyzed by water to release HCl gas. It cannot be formed by heating the hydrated form to drive off water. This only produces the oxide and HCl gas. It is now made commercially by heating aluminium oxide with carbon and chlorine. It is used in the refining motor oil, and as catalyst. Hydrated aluminium chloride is used as a personal deodorant. The acid environment it creates is unpleasant for microbes and mild enough to be non-irritating.

The spectroscopic ground state is $3s^2 3p$. Resonance line is at 396.15 nm, so the aluminium atom is not exited in the flame and gives it no color. When atom is excited, most of the lines are in the red or infrared. Aluminium is in column IIIA in the periodic table, which includes boron, aluminium, gallium, indium and thalium are typical basic metals.

Aluminium displace hydrogen from water because of its positive oxidation potential, but does not normally do because of the protection by a surface layer of oxide. This oxide has the same density as the metallic aluminium, so it does not crack or wrinkle when it is formed. A little HCl or NaoH that dissolve the protective layer ad allow the metal to be attacked.

Aluminium is attacked by hydrochloric acid, but not by oxidizing acids like nitric. If aluminium is amalgamated with mercury, the protective oxide layer is removed, the metal becomes very reactive, and will displace hydrogen from water. It can be used for handling cold nitric acid, but dissolves readily in alkalis to form aluminates, an example of amphoteric behavior of aluminium. Formation of the oxide also prevents aluminium from being soldered with ordinary Sn-Pb solder, since the solder will not wet it. Sn-Zn. Solders can be used with aluminium. A suitable alloy is 85 Sn, 15 Zn, 5 Al. 60 Zn, 40 Cd has better corrosion resistance, but it is harder to melt. The solder itself serves as a flux. With a suitable flux to protect metal, aluminium can be welded.

Aluminium was first isolated by Hans Christian Oersted (1777-1851) in 1824 by reducing it from its oxide with potassium amalgam. The reaction is:

$AlCl_3 + 3K \dashrightarrow 3KCl + Al.$

Two years later, wohler made the metal the same way. For some reason, Wohler is usually, considered the discoverer and got most of the fame. This scientific biography, the article on Oersted does not mention his work with aluminium. Refractory oxides such as alumina silica or magnesia, were considered elements earlier, when all attempts to decompose them had failed. Only electrolysis made the decomposition possible, either directly or through the production of reactive metals like sodium and potassium.

The termite reaction, discovered by Goldschmidt, is also a displacement reaction, but here aluminium reduces iron. The reaction is:

$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3$

Which liberates, a good deal of heat. The liquid metal produced is at about 2300^{o}C, which is very hot. Powdered aluminium and rust in the approximate ratio of 1: 3 to ignite it.

2.11 Aluminium Sulphate (alum) Sample Analysis

In the process of Alum recovery, the sample of the whole experiment has to be analyze to proof that the aluminium sulphate is recovered. This will proof acidification process really bear aluminium sulphate from alum sludge. Well established process of analysis is HACH 2000 analyzer.

HACH 2000 analysis process is used for water and waste water analysis. The purpose of HACH 2000 programs is to detect chemicals in the solution experimented. The method is specific for each chemical substance.

Aluminium tested for HACH 2000 method referred as Method 8012. Method 8012 tested for aluminium in range of 0.008 to 0.8 mg/L. Operating temperature of HACH programs is 20-25 ^{0}C for accurate results. All glassware must be clean with 6.0 N HCl and deionized water before use to remove contaminants from the glass.

The analysis chemical for aluminium concentration test are:

a. Ascorbic acid powder pillow
b. AluVer 3 Aluminium Reagent Powder Pillow.
c. Bleaching 3 Reagent Powder Pillow

Aluminon indicator combines with aluminium in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added to remove iron interference. The AluVer 3 Aluminium Reagent, package in powder form, shows exceptional stability and is applicable for fresh water applications. Test results are measured at 522 nm.

2.12 Pilot Scale Study on Sludge Disposal

Pilot Scale Study utilized a Chemical treatment method which was able to recover some by-products and objectives were mainly:

a). reduction of the volume and mass of solid sludge

b). beneficial reuse of recovered alum.

The observed recovered alum was 45% of the raw sludge and the average strength of recovered alum obtained was about 2.5% giving an aluminium content of about 520 mg/l.

The treatment technology employed in this study has clearly demonstrated that sludge generated from water treatment plants can be effectively treated. The plant has the potential to reduce the sludge by up to 45% in volume and furthermore, it provides tremendous saving in disposal area and handling costs.

Natural surface waters contain a variety of organic and inorganic suspended solids. These particles are diverse in shape, size and density; usually are negatively charged; generally do not agglomerate spontaneously and generally remain in colloidal suspension in the water. The purpose of chemical coagulation is the agglomeration and subsequent sedimentation and removal of these suspended solid from the raw water.

Usually, the positively charged salts of aluminium, iron or magnesium are used as primary coagulants to enhance the settling characteristic of the floc.

Most water treatment plants in Malaysia use a alum (aluminium Sulphate) as a primary coagulant. A review of the types and volumes of wastewater discharged from water treatment plants in Malaysia would no doubt prove that alum-waste quantities are the greatest. Moreover alum waste would also be considered to be among the most troublesome to condition for satisfactory disposal. The problem is with the hydroxide contained in the sludge, resulting from the use of alum in the coagulation process usually preceeding filtration. Aluminium hydroxide is gelatinous in nature and will remain in its semifluid state indefinitely unless something is done to change its physical characteristics. Although the hydroxide sludge does settle readily, it is almost impossible to dewater without prior treatment.

Aluminium hydroxides have elevated solubilities under highly acidic and highly alkaline conditions. The potential therefore exists for removal of problematic metal hydroxides from sludge suspension for two beneficial purpose

:

1). Reduction of the volume and mass of sludge solids produced for disposal

2). Beneficial reuse of coagulant metal

Coagulant recovery is approached for two very different but related objectives. One approach is to consider acid or alkali addition as a sludge conditioning technique with improved thickening, dewatering and waste sludge reduction as major objectives. This approached is best referred to as sludge conditioning, although it is frequently described as coagulant recovery even when recovery of the coagulant is of limited importance. A second approach is to maximize the recovery and reuse of aluminium contained in sludge suspensions, with the objective of minimizing expenditures of commercial coagulant recovery.

2.13 KESEDAR Water Treatment Plant (KWTP)

KESEDAR Water Treatment Plant (KWTP) is located about 30 km from of Gua Musang, Kelantan. The KWTP was commissioned in 1989 by Kelantan Selatan Development Sdn. Bhd. (KESEDAR) through Jabatan Bekalan Air (JBA) Kelantan and in June 2001 privatized the plant to Air Kelantan Sdn. Bhd. under the management, operation and maintenance contract. The raw water is abstracted from the Sungai Lebir at an intake located about *13 km from the plant. From the intake, the raw water pumped to the KWTP through 2 pipelines each with a diameter of 2050 mm. treatment processes at the plant include the use of alum (aluminium sulphate) for coagulation of turbidity and color removal.

Existing sludge facility at KWTP consists of a sludge lagoon which occupies an area of about 32,000 m^2. Sludge obtained from sedimentation and filter tanks of KWTP were disposed off into the sludge lagoon daily at a rate of 1,000 to 1,500 cubic meters. The sludge were allowed to settle in the lagoon while the supernatant were allowed to overflow across a weir to a nearby river. The problem arises when the sludge lagoon began to fill up and the present existing sludge facility were not able to cope up with the enormous quantity of sludge which in turn creates a lot of management problems.

CHAPTER 3

METHODOLOGY

3.0 Methodology

The methods of this study are divided into three different phase. The phase is divided according to the work criterion. The brief explanations of the phase are as followed:

3.1 Phase 1: Source of Sampling

Sludge sample obtained from water treatment tank. The sludge will be taken in four storage tanks purchased from hardware shops. The storage tank must have smaller mouth opening to prevent condensation and reaction of the sludge with open air. The sample then kept for a period to separate the sludge layer and alum layer. The sludge will then settle to the bottom of the tank. Alum which appear in liquid form will appear above the sludge layer. So, the distinction of sludge and alum will be visibly clear.

The sludge sample then taken to the lab for justifying pH values. The pH values justification done according to the tank. There are 4 variables of pH values required for this experiment. They are 1.5, 2.5, 3.0 and 3.5. The pH values measured after addition of acidic solution.

The sample taken then measured in a total content of Al(III) content 0.1 mg/L (supernatant). Analysis process will be done using HACH 2000 method. This method specifies for aluminium test to detect the content of aluminium in the recovered solution.

3.2 Phase 2: Laboratory analysis and Method

3.2.1 Setting up pH parameters

The sample from water treatment sludge brought to laboratory for experiment. The sample then treated with acid to fix the pH values to 4 values that are 1.5, 2.5, 3, and 3.5. The process of justifying pH done is the acidification process. Addition of sulphuric acids done to get the 4 pH values required. Sulphuric acid used are diluted 75% sulphuric acid giving concentration of acid 0.75 M.

Sulphuric acid 1.0 M diluted to get 0.75 M. Using dilution equation 187.5 ml of 1 M sulphuric acid diluted in the 250 ml volumetric flask. From theoretical method, this will give 0.75 M sulphuric acid.

These 4 parameters of pH values are obtained using titration process. Titration process is the process of adding acid into the 100 ml sample until the desired pH values achieved. Sample then put into 4, 250 ml conical flasks. The method of titration practices using pH meter to measure the pH values and pipette to run the acidification process. Using pipette, acid sulphuric solution dropped little by little into the conical flask until desired pH values achieved. The pH values measured using pH meter. This acidification process stopped when the solution reached the desired pH values.

In order to make sure the pH values recorded are accurate, the conical flask containing sludge sample then put on the magnetic stirrer apparatus to make sure Aluminium hydroxide reacted perfectly with sulphuric acid.

3.2.2 Fixing the stirring speed parameters.

This is done using magnetic stirrer apparatus. Sample in the conical flask, then stir using magnetic stirrer. There are 2 parameters of stirring speed involved in this experiment; 100 rpm and 200 rpm.

In order to fix the stirring speed parameter, stirring stic put into the conical flask. The conical flask then put on top of magnetic stirrer. The magnetic stirrer then set up at a desired stirring speed for 100 or 200 rpm. The stirring stick will stir the solution according to the stirring speed required. The stirring then left for a period of 5 minutes to 10 minutes. This stirring process done to make sure the solution reacted. After that, the supernatant from this experiment can be assume containing aluminium sulphate.

This stirring procedure, can make the solution mixed well, as the literature review shows that, sludge containing aluminium hydroxide when reacted with sulphuric acid will then produce, water and aluminium sulphate.

3.2.3 Setting up Centrifugation time.

Every sample of different pH and different speed of stirring, then put in the centrifugation bottle to be centrifuge to separate solid particles and aluminium sulphate aqueous solution. In the centrifugation process, the parameter analyzed is period of centrifugation. Period of centrifugation analyzed at 20 minutes, 40 minutes, and 60 minutes for every stirring speed and pH values.

The equipment use for centrifugation process is High Speed Centrifuge. This High speed centrifuge can set the period for centrifugation, temperature and and the speed of centrifugation. In this study, the parameter are only period of centrifuge, the other parameters are fixed and regarded as constant such as centrifugation speed and temperature. The temperature is fixed at room temperature and speed of centrifugation is fixed at 1000 rpm.

During the centrifugation, the sample poured into the centrifuge bottles. This experiment is done partially for every pH values. It is because, the high speed centrifuge can only centrifuge 4 centrifuge bottle at one time. So, the easiest way is to centrifuge the sample according to pH values, to avoid any mistake in experiment. With every set of centrifuge bottle, the centrifugation period then set at every particular period 20 minutes, 40 minutes or 60 minutes.

After centrifugation, the separation process can be visible, as the supernatant will be on top and the solid will be seen beneath the bottle. The top supernatant is the aluminium sulphate recovered and the solid in the bottom is the particulate solid that presence before coagulation process back in water treatment plant.

3.2.4 Determining Precipitation Time

To determine the retention time, the solution in every centrifuge bottle then filtered using filter paper. The sample then left to be filtrate for 24 hours. The determination of retention time recorded when all solution filtrated, the observation done every hour to get the best retention time after experiment. The filtration complete when the entire water drops from filter paper no longer exist. The time fluid and the solid separated then recorded for every sample taken.

3.2.5 Determination of the Volume Reduction

The solution of aluminium sulphate then measured using graduated cylinder. Readings from graduated cylinder recorded for every sample. Sample of pH values and volume recovered recorded according to speed of stirrer and centrifugation time. From the table, the volume recovered concluded in the graph to the best volume recovered using different pH values, different period of centrifuge and different stirrer speed. The graph will show the best condition for alum recovery. After measurement,the sample transferred into conical flask to perform analysis method.

3.3 Phase 3: Analysis method

For analysis method, the samples were then taken for HACH 2000 test. The sample tested for the efficiency of Al recovery in the supernatant. HACH 2000 Method used to detect aluminium composition in the solution.

First method is to wash and clean all the glassware with 6.0 N HCl and deionized water before removing contaminants from the glass. After switching on the equipment, touch HACH Programs, program named 10 Aluminum, Alumin selected. The graduated cylinder used for the analysis. The cylinder filled with the sample to 50 ml mark. The sample in the cylinder then added with Ascorbic Acid Powder Pillow. Then the content added with AluVer 3 Aluminium Reagent Powder Pillow. 25 ml from the sample then poured into the sample cell. The balance content then bleached with Bleaching 3 Reagent Powder Pillow. The sample in the graduated cylinder poured into another sample cell. Put the sample cell into the equipment compartment and touch zero. The display will show 0.000 mg/L Al^{3+} . The second sample cell then inserted into the equipment compartment and the result will appear in mg/L Al^{3+} . The method then repeated for all sample.

After the result recorded, calculations of alum recovery then perform every sample. From this calculation the highest percentage of alum recovered will determine the optimum condition for recovery alum. This will then propose for pilot plant operation of aluminium recovery in for integration of energy and best product of recovery.

3.4 Block Diagram For Acidification Process on Alum Sludge

Alum Sludge Sampling Process
Sample taken from KESEDAR Water Treatment Plant (KWTP)

Standard Sample Preparation
100 ml Alum sludge without any acidification process allowed to settle, Volume Recovered, Precipitation period and HACH analysis perform and the result recorded

pH Values Adjustment
100 ml Alum sludge of each sample then acidified to required pH values of 1.5, 2.5, 3.0 and 3.5

Stirring Speed and Centrifugation Time Set Up
During acidification applied to the every sample, sample stirred for 100 rpm and 200 rpm. Each 3 samples of stirring speeds centrifuged for 20, 40 and 60 minutes.

Gathering Sample Result
Every 24 sample allowed to settle for 24 hours, the volume recovered taken for every hour till only solid left in the cylindrical flask

Recording Result
The last volume recovered recorded in ml, the last precipitation time recorded in hours in tables

Sample Analysis
Every 24 supernatant recovered then tested using HACH 2000 Analysis Method. Every analysis result recorded in Tables

CHAPTER 4

RESULT AND DISCUSSION

Those below tables and figures are the results of every 24 sample of 100 ml alum sludge experimented for determination of pH adjustment, Stirring speeds and centrifugation periods. For standard alum sludge without any acid treatment pH values of 6.8 gives the value of 40.2 ml for 24 hours period of precipitation and gives the analysis reading of 0.04 mg/L Al^{3+}.

4.1 Effect of pH Adjustment

4.1.1 For 100 rpm Stirrer Speed, 20 minutes centrifugation period

This is the data recorded from the experiment of 100 rpm stirrer speed and 20 minutes centrifugation period. The control variables are pH values, the manipulate variables are volume recovered and the volume reduction. The constant variables are stirring speed and centrifugation period.

Table 4.1: Table for 100 rpm stirrer speed, 20 minutes centrifugation period

pH values	Volume Recovery,ml	Standard Recovery,ml	Volume Reduction,ml
1.5	80.4	40.2	40.2
2.5	88.1	40.2	47.9
3.0	78.2	40.2	38.0
3.5	72.2	40.2	32.0

The volume of every sample of aluminium sulphate (alum) aqueous recovered, and the sample without any acid added also recorded for standard recovery to calculate volume reduction.

From the table it shows that at pH 1.5 recorded 40.2 ml volume reduction, 2.5 pH value recording 47.9 ml volume reduction, 3.0 pH value gives 38.0 ml volume reduction and pH values of 3.5 gives 32.0 ml volume reduction.

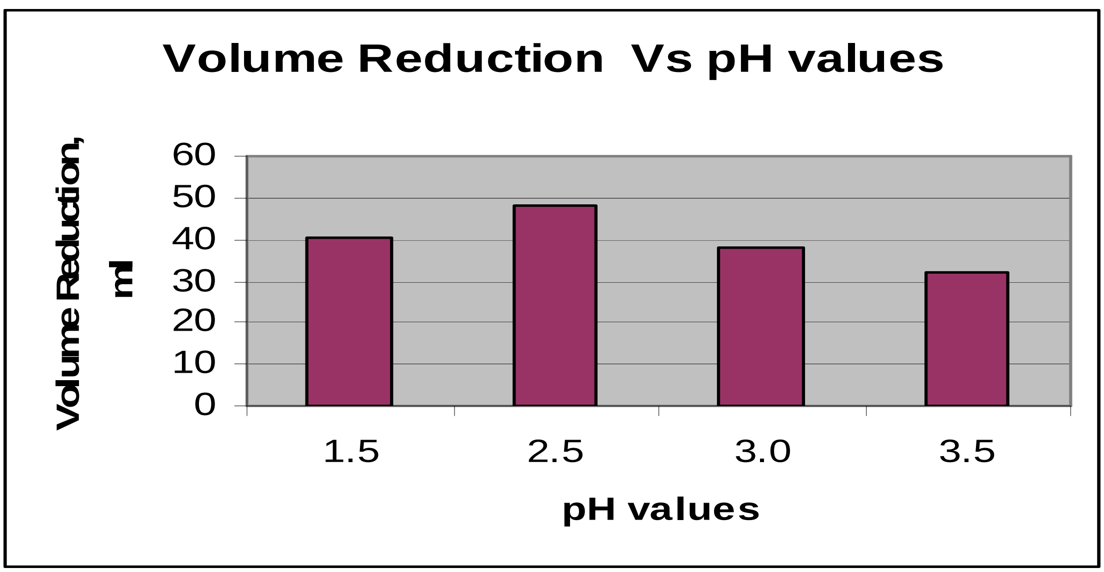

Figure 4.1: Volume reduction of 100 rpm, 20 minutes

From this graph, the highest volume reduction is pH values of 2.5., while the lowest volume reduction is 3.5. For 100 rpm stirring speed and 20 minutes centrifugation time gives the best for pH values of 2.5.

4.1.2 For 100 rpm stirring speed, 40 minutes centrifugation period

This is the data recorded from the experiment of 100 rpm stirrer speed and 20 minutes centrifugation period. The control variables are pH values, the manipulate variables are volume recovered and the volume reduction. The constant variables are stirring speed and centrifugation period.

Table 4.2: Table for 100 rpm stirrer speed, 40 minutes centrifugation period

pH values	Volume Recovery,ml	Standard Recovery,ml	Volume Reduction,ml
1.5	82.5	40.2	42.3
2.5	89.2	40.2	49.0
3.0	79.3	40.2	39.1
3.5	66.3	40.2	26.1

The volume of every sample of aluminium sulphate (alum) aqueous recovered, and the sample without any acid added also recorded for standard recovery to calculate volume reduction.

From the table it shows that at pH 1.5 recorded 42.93 ml volume reduction, 2.5 pH value recording 49.0 ml volume reduction, 3.0 pH value gives 39.1 ml volume reduction and pH value of 3.5 gives 26.1 ml volume reduction.

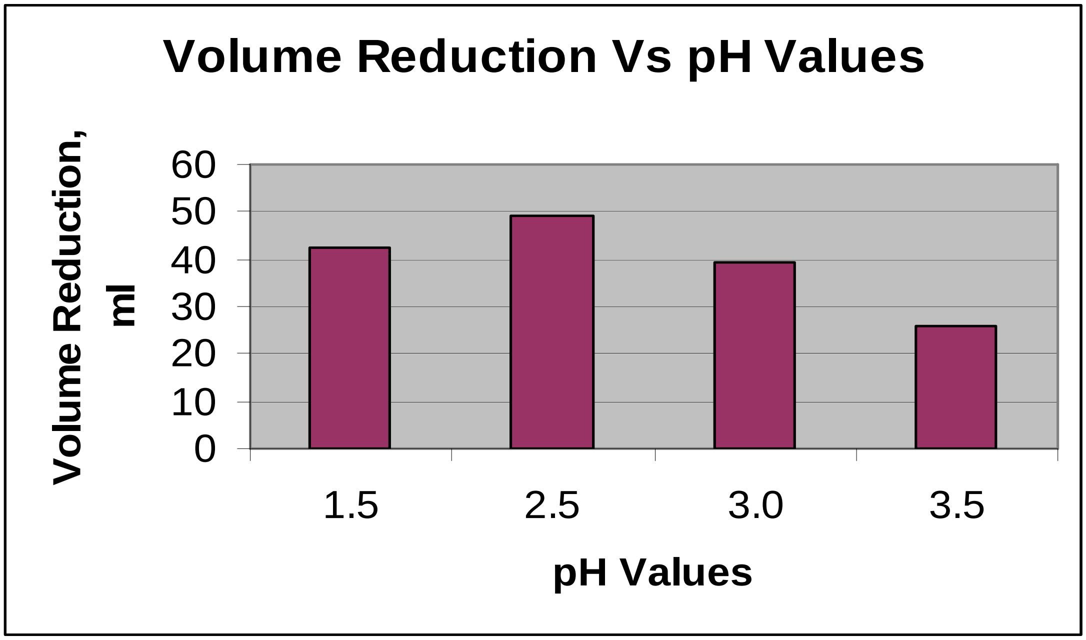

Figure 4.3: Volume reduction for 100 rpm, 40 minutes

From the above graph, the highest volume reduction is the pH values of 2.5 and the lowest volume recovered is pH Values of 3.5. This give the pH values of 2.5 is the most suitable pH value to recover alum as much as possible.

4.1.3 For 100 rpm stirring speed, 60 minutes centrifugation period

This is the data recorded from the experiment of 100 rpm stirrer speed and 20 minutes centrifugation period. The control variables are pH values, the manipulate variables are retention time and the volume recovered. The constant variables are stirring speed and centrifugation period.

Table 4.3: Table for 100 rpm stirrer speed, 60 minutes centrifugation period

pH values	Volume Recovery,ml	Standard Recovery,ml	Volume Reduction,ml
1.5	81.2	40.2	41.0
2.5	88.4	40.2	48.2
3.0	72.5	40.2	32.3
3.5	61.3	40.2	21.1

The volume of every sample of aluminium sulphate (alum) aqueous recovered, and the sample without any acid added also recorded for standard recovery to calculate volume reduction.

From the table it shows that at pH 1.5 recorded 41.0 ml volume reduction, 2.5 pH value recording 48.2 ml volume reduction, 3.0 pH value gives 32.3 ml volume reduction and pH values of 3.5 gives 21.1 ml volume reduction.

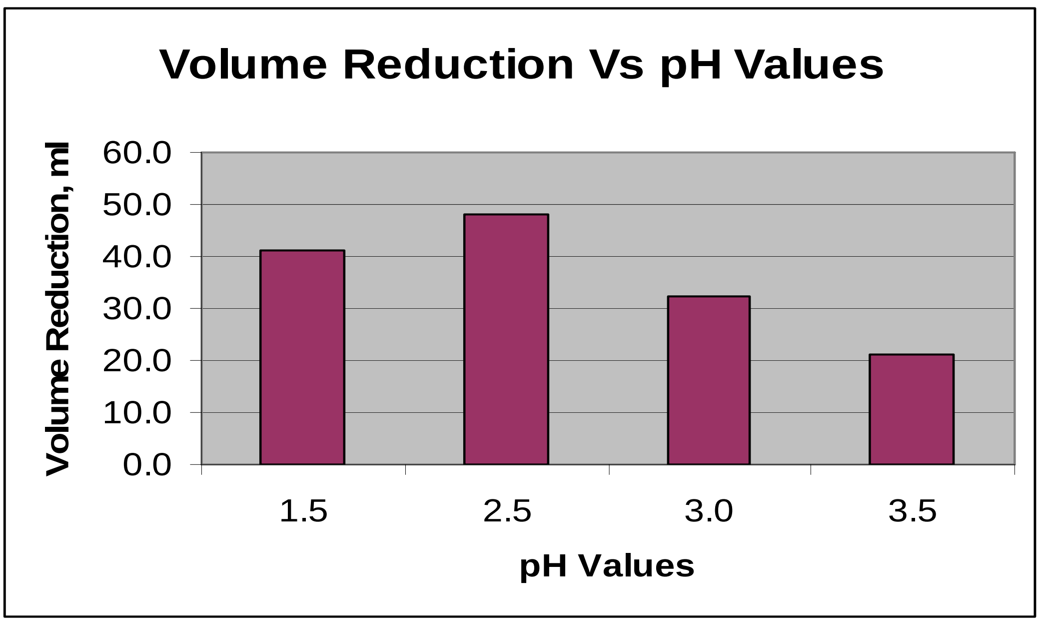

Figure 4.5: Graph of retention time versus pH values

From the above graph, the highest volume reduction is the pH values of 2.5 and the lowest volume recovered is pH Values of 3.5. This give the pH values of 2.5 is the most suitable pH value to recover alum as much as possible.

4.1.4 For 200 rpm stirring speed, 20 minutes centrifugation period

This is the data recorded from the experiment of 100 rpm stirrer speed and 20 minutes centrifugation period. The control variables are pH values, the manipulate variables are retention time and the volume recovered. The constant variables are stirring speed and centrifugation period.

Table 4.4: Table for 200 rpm stirrer speed, 20 minutes centrifugation period

pH values	Volume Recovery,ml	Standard Recovery,ml	Volume Reduction,ml
1.5	81.1	40.2	40.9
2.5	88.2	40.2	48.0
3.0	72.5	40.2	32.3
3.5	61.5	40.2	21.3

The volume of every sample of aluminium sulphate (alum) aqueous recovered, and the sample without any acid added also recorded for standard recovery to calculate volume reduction.

From the table it shows that at pH 1.5 recorded 40.9 ml volume reduction, 2.5 pH value recording 48.0 ml volume reduction, 3.0 pH value gives 32.3 ml volume reduction and pH values of 3.5 gives 21.3 ml volume reduction.

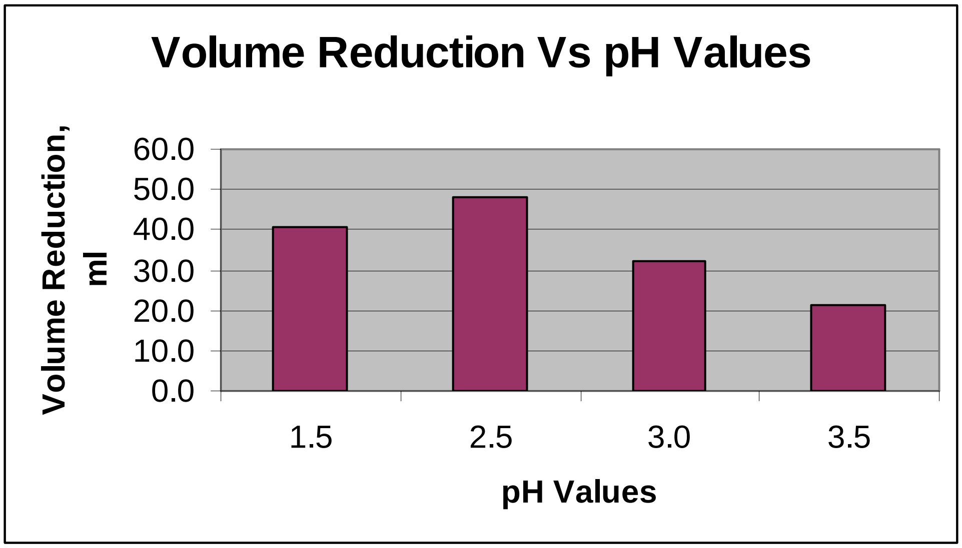

Figure 4.7: Graph of retention time versus pH values

From the above graph, the highest volume recovered is the pH values of 2.5 and the lowest volume recovered is pH Values of 3.5. This give the pH values of 2.5 is the most suitable pH value to recover alum as much as possible.

4.1.5 For 200 rpm stirring speed, 40 minutes centrifugation period

This is the data recorded from the experiment of 100 rpm stirrer speed and 20 minutes centrifugation period. The control variables are pH values, the manipulate variables are retention time and the volume recovered. The constant variables are stirring speed and centrifugation period.

Table 4.5: Table for 200 rpm stirrer speed, 40 minutes centrifugation period

pH values	Volume Recovery,ml	Standard Recovery,ml	Volume Reduction,ml
1.5	71.5	40.2	31.3
2.5	84.2	40.2	44.0
3.0	76.5	40.2	36.3
3.5	56.3	40.2	16.1

The volume of every sample of aluminium sulphate (alum) aqueous recovered, and the sample without any acid added also recorded for standard recovery to calculate volume reduction.

From the table it shows that at pH 1.5 recorded 31.3 ml volume reduction, 2.5 pH value recording 44.0 ml volume reduction, 3.0 pH value gives 36.3 ml volume reduction and pH values of 3.5 gives 16.1 ml volume reduction.

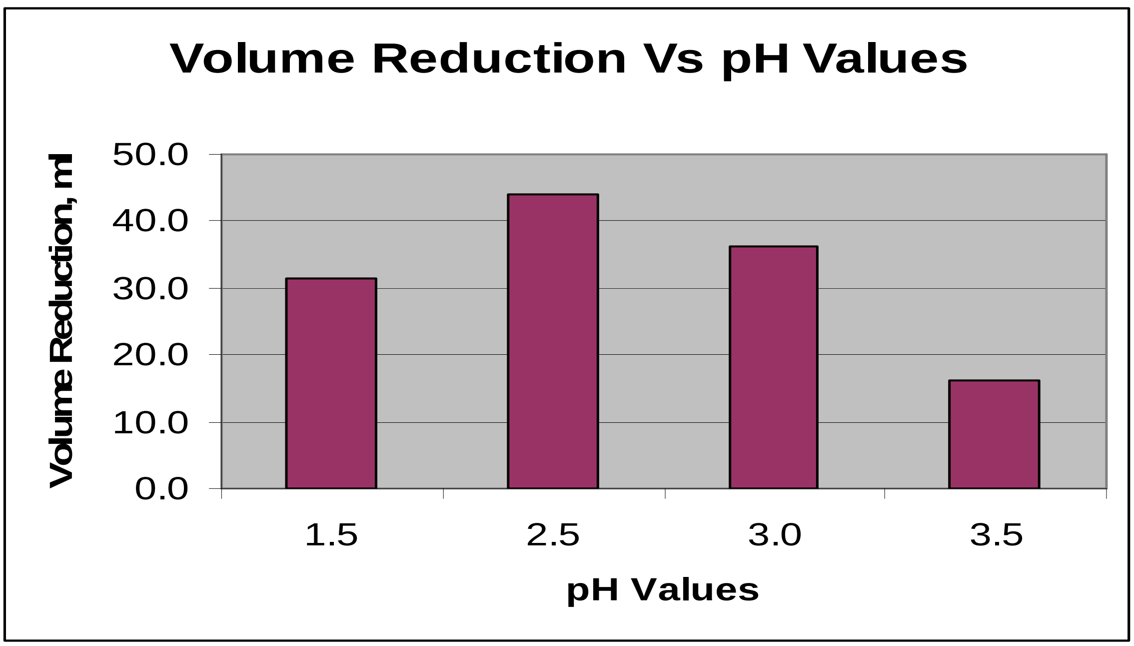

Figure 4.9: Graph of retention time versus pH values

From the above graph, the highest volume recovered is the pH values of 2.5 and the lowest volume recovered is pH Values of 3.5. This give the pH values of 2.5 is the most suitable pH value to recover alum as much as possible.

4.1.6 For 200 rpm stirring speed, 60 minutes centrifugation period

This is the data recorded from the experiment of 100 rpm stirrer speed and 20 minutes centrifugation period. The control variables are pH values, the manipulate variables are retention time and the volume recovered. The constant variables are stirring speed and centrifugation period.

Table 4.6: Table for 200 rpm stirrer speed, 60 minutes centrifugation period

pH values	Volume Recovery,ml	Standard Recovery,ml	Volume Reduction,ml
1.5	65.5	40.2	25.3
2.5	87.2	40.2	47.0
3.0	66.1	40.2	25.9
3.5	64.2	40.2	24.0

The volume of every sample of aluminium sulphate (alum) aqueous recovered, and the sample without any acid added also recorded for standard recovery to calculate volume reduction.

From the table it shows that at pH 1.5 recorded 25.3 ml volume reduction, 2.5 pH value recording 47.0 ml volume reduction, 3.0 pH value gives 25.9 ml volume reduction and pH values of 3.5 gives 24.0 ml volume reduction.

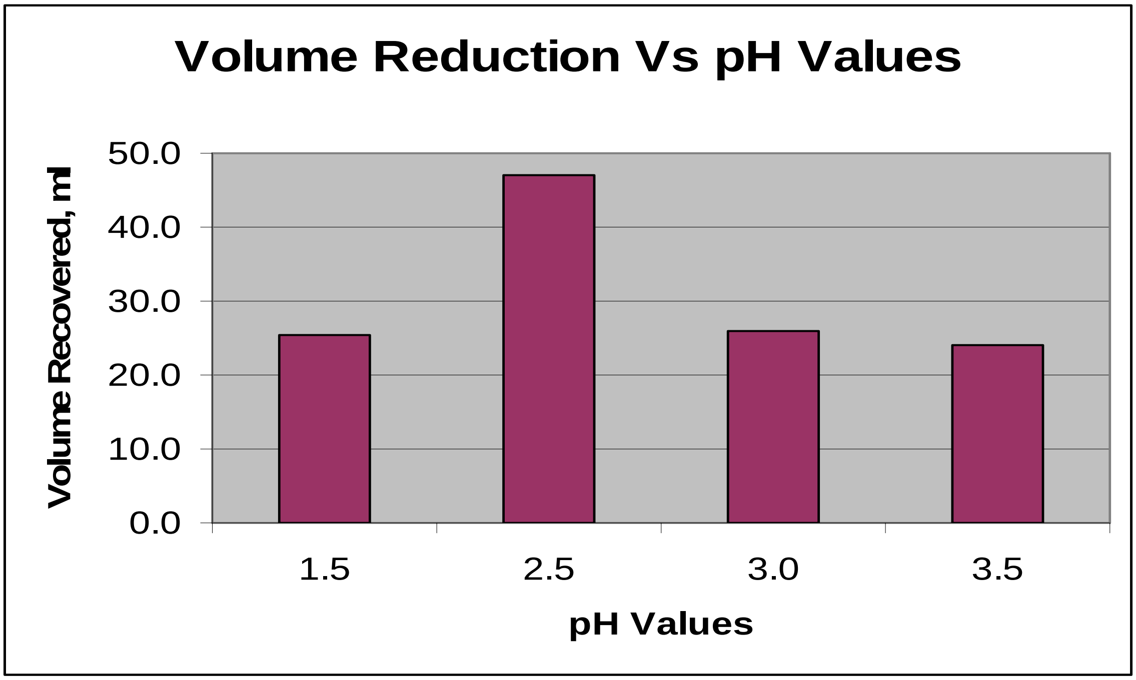

Figure 4.11: Graph of retention time versus pH values

4.2 Effect of Centrifugation Time to the Precipitation Period

4.2.1 Precipitation Period for 100 rpm, 20 Minutes

For 100 rpm stirring speed and 20 minutes centrifugation period, 4 parameters of pH values used to determine the result of precipitation period. This precipitation period will determine the best pH values to recover aluminium sulphate (alum) in the shortest duration.

Table 4.7: Precipitation period for 100 rpm, 20 minutes

pH values	Precipitation Period, hr
1.5	18
2.5	16
3.0	20
3.5	22

From table 4.7, precipitation period for four pH values recorded. For pH value of 1.5 the precipitation period is 18 hours, 2.5 pH value shows precipitation period of 16 hours, 3.0 pH value recorded result for 20 hours and pH values of 3.5 shows the result of 22 hours precipitation period.

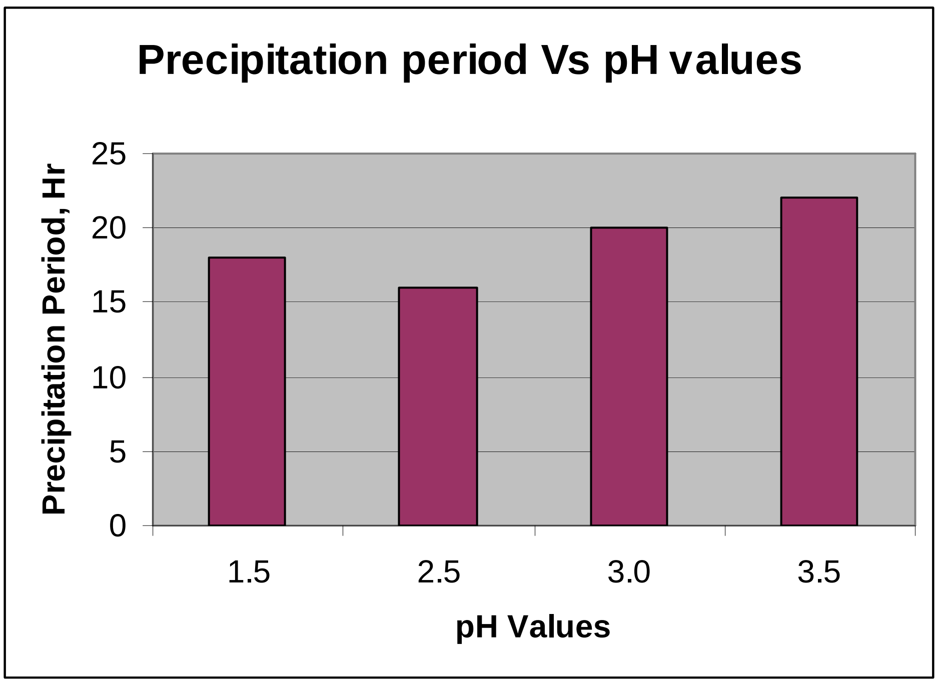

Figure 4.7: Precipitation Period for 100 rpm and 20 minutes

From graph in figure 4.7, it shows that pH values 2.5 give the shortest precipitation period for 100 rpm stirring speed and 20 minutes centrifugation time. The longest precipitation period is pH value of 3.5.

4.2.2 Precipitation Period for 100 rpm, 40 Minutes

For 100 rpm stirring speed and 40 minutes centrifugation period, 4 parameters of pH values used to determine the result of precipitation period. This precipitation period will determine the best pH values to recover aluminium sulphate (alum) in the shortest duration.

Table 4.8: Precipitation period for 100 rpm, 40 minutes

pH values	Precipitation Period, hr
1.5	17
2.5	16
3.0	19
3.5	21

From table 4.8, precipitation period for four pH values recorded. For pH value of 1.5 the precipitation period is 17 hours, 2.5 pH value shows precipitation period of 16 hours, 3.0 pH value recorded result for 19 hours and pH values of 3.5 shows the result of 21 hours precipitation period.

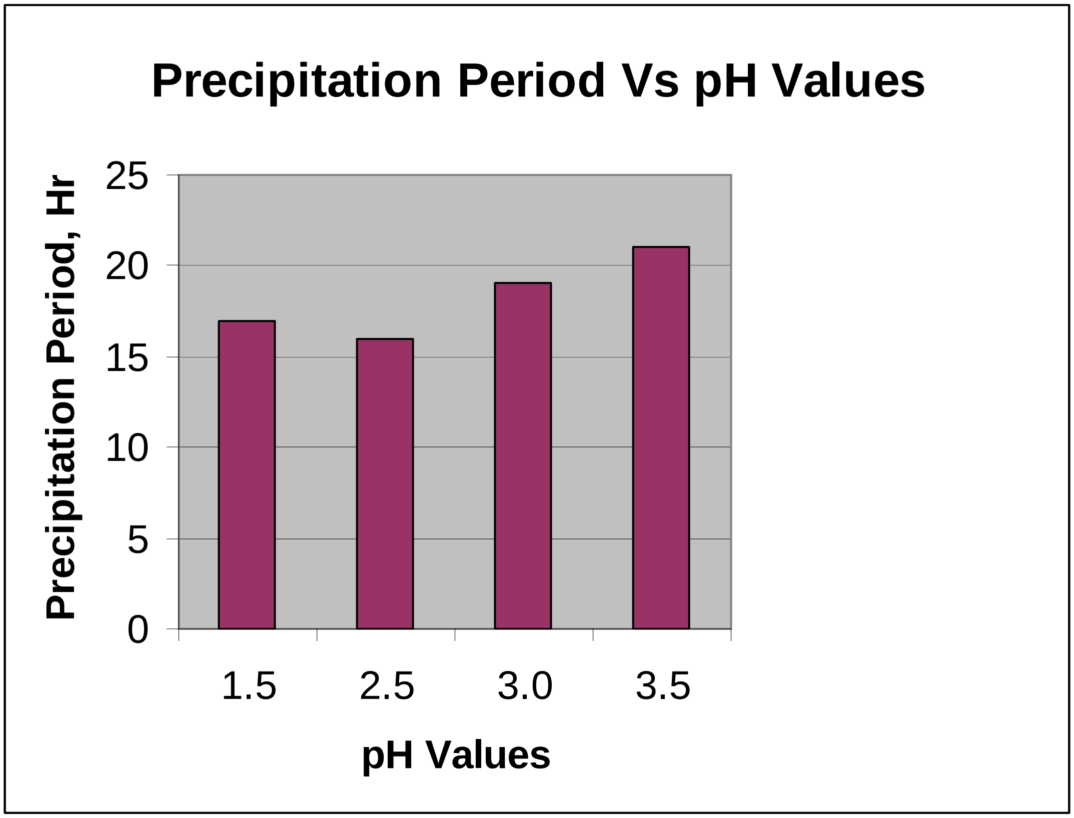

Figure 4.8: Precipitation Period for 100 rpm and 40 minutes

From graph in figure 4.8, it shows that pH values 2.5 give the shortest precipitation period for 100 rpm stirring speed and 40 minutes centrifugation time. The longest precipitation period is pH value of 3.5.

4.2.3 Precipitation Period for 100 rpm, 60 Minutes

For 100 rpm stirring speed and 60 minutes centrifugation period, 4 parameters of pH values used to determine the result of precipitation period. This precipitation period will determine the best pH values to recover aluminium sulphate (alum) in the shortest duration.

Table 4.9: Precipitation period for 100 rpm, 60 minutes

pH values	Precipitation Period, hr
1.5	18
2.5	16
3.0	19
3.5	21

From table 4.9, precipitation period for four pH values recorded. For pH value of 1.5 the precipitation period is 18 hours, 2.5 pH value shows precipitation period of 16 hours, 3.0 pH value recorded result for 19 hours and pH values of 3.5 shows the result of 21 hours precipitation period.

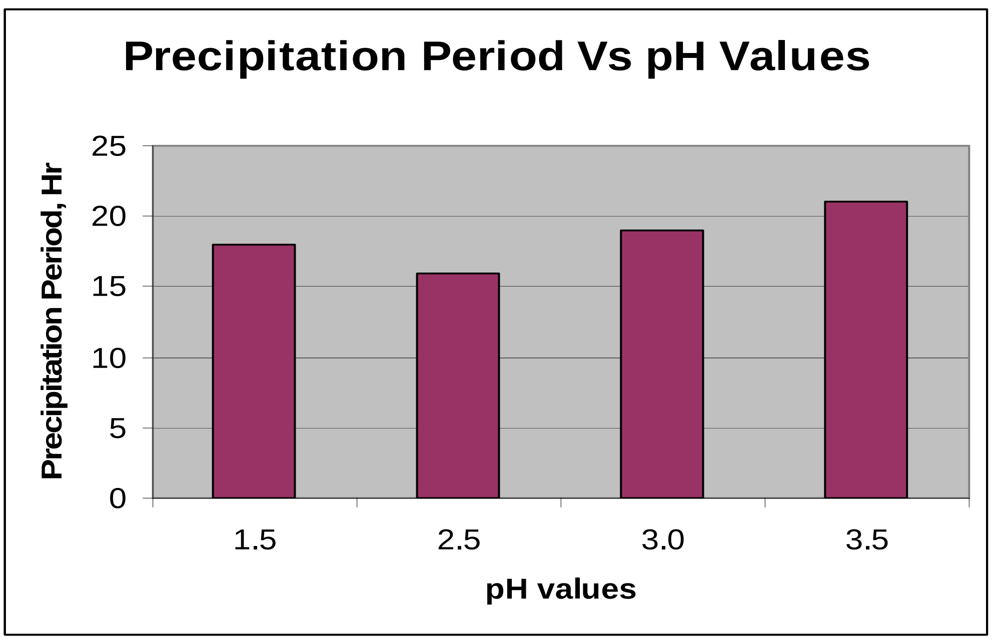

Figure 4.9: Precipitation Period for 100 rpm and 60 minutes

From graph in figure 4.9, it shows that pH values 2.5 give the shortest precipitation period for 100 rpm stirring speed and 60 minutes centrifugation time. The longest precipitation period is pH value of 3.0

4.2.4 Precipitation Period for 200 rpm, 20 Minutes

For 200 rpm stirring speed and 20 minutes centrifugation period, 4 parameters of pH values used to determine the result of precipitation period. This precipitation period will determine the best pH values to recover aluminium sulphate (alum) in the shortest duration.

Table 4.10: Precipitation period for 200 rpm, 20 minutes

pH values	Precipitation Period, hr
1.5	17
2.5	16
3.0	21
3.5	19

From table 4.10, precipitation period for four pH values recorded. For pH value of 1.5 the precipitation period is 17 hours, 2.5 pH value shows precipitation period of 16 hours, 3.0 pH value recorded result for 21 hours and pH values of 3.5 shows the result of 19 hours precipitation period.

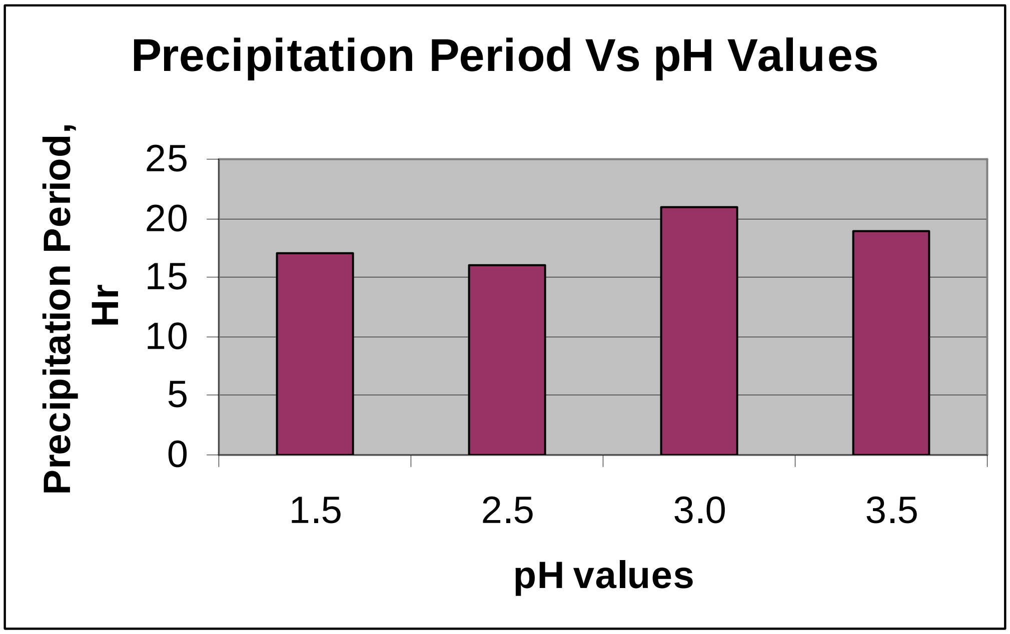

Figure 4.10: Precipitation Period for 200 rpm and 20 minutes

From graph in figure 4.10, it shows that pH values 2.5 give the shortest precipitation period for 200 rpm stirring speed and 20 minutes centrifugation time. The longest precipitation period is pH value of 3.5.

4.2.4 Precipitation Period for 200 rpm, 40 Minutes

For 200 rpm stirring speed and 40 minutes centrifugation period, 4 parameters of pH values used to determine the result of precipitation period. This precipitation period will determine the best pH values to recover aluminium sulphate (alum) in the shortest duration.

Table 4.11: Precipitation period for 200 rpm, 40 minutes

pH values	Precipitation Period, hr
1.5	18
2.5	16
3.0	19
3.5	20

From table 4.11, precipitation period for four pH values recorded. For pH value of 1.5 the precipitation period is 18 hours, 2.5 pH value shows precipitation period of 16 hours, 3.0 pH value recorded result for 19 hours and pH values of 3.5 shows the result of 21 hours precipitation period.

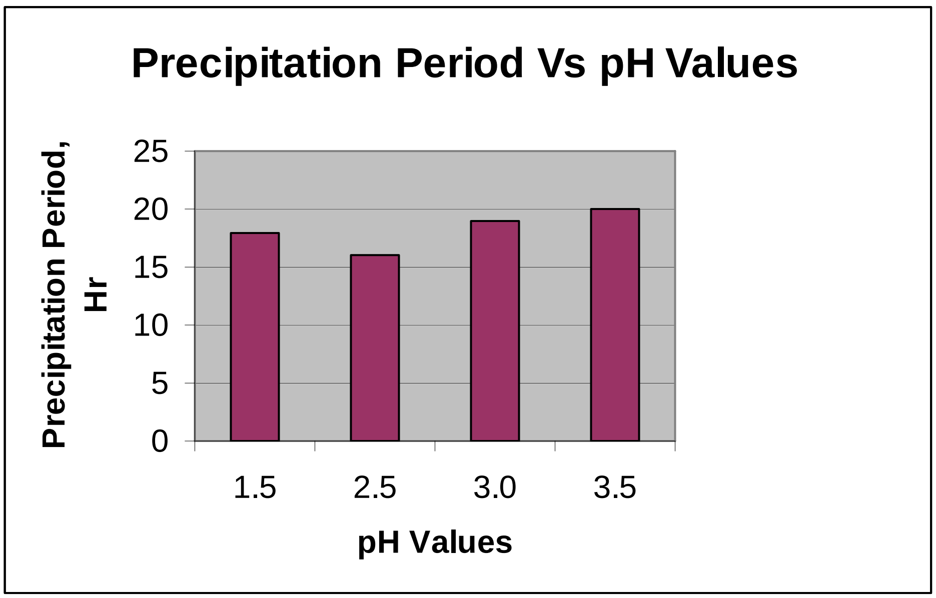

Figure 4.11: Precipitation Period for 200 rpm and 40 minutes

From graph in figure 4.10, it shows that pH values 2.5 give the shortest precipitation period for 200 rpm stirring speed and 40 minutes centrifugation time. The longest precipitation period is pH value of 3.5.

4.2.4 Precipitation Period for 200 rpm, 60 Minutes

For 200 rpm stirring speed and 60 minutes centrifugation period, 4 parameters of pH values used to determine the result of precipitation period. This precipitation period will determine the best pH values to recover aluminium sulphate (alum) in the shortest duration.

Table 4.11: Precipitation period for 200 rpm, 40 minutes

pH values	Precipitation Period, hr
1.5	19
2.5	17
3.0	18
3.5	21

From table 4.12, precipitation period for four pH values recorded. For pH value of 1.5 the precipitation period is 19 hours, 2.5 pH value shows precipitation period of 17 hours, 3.0 pH value recorded result for 18 hours and pH values of 3.5 shows the result of 21 hours precipitation period.

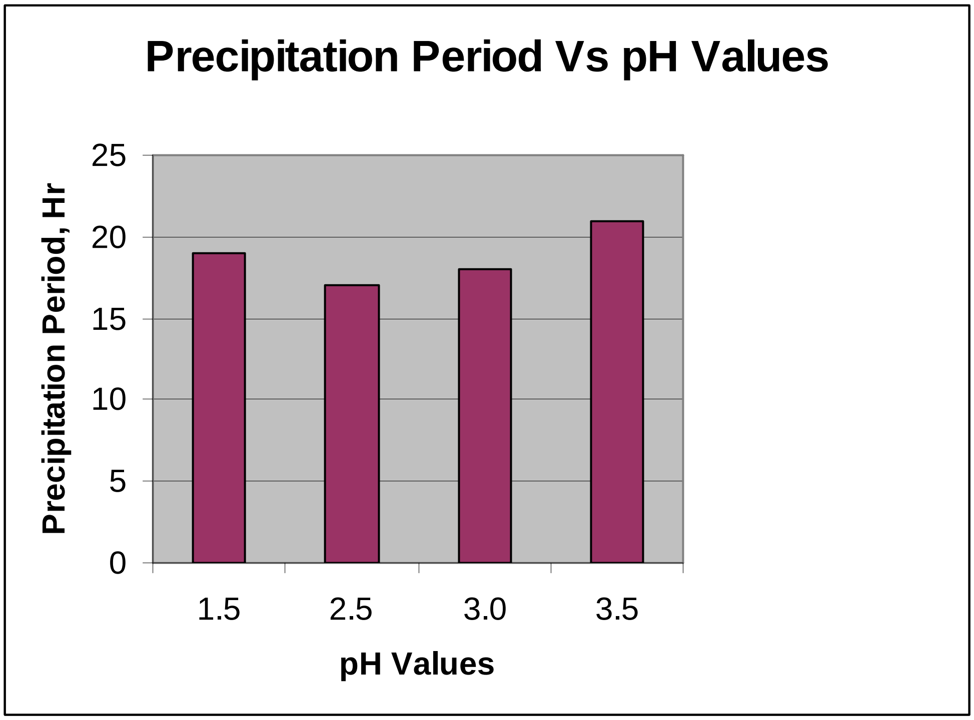

Figure 4.11: Precipitation Period for 200 rpm and 40 minutes

From graph in figure 4.10, it shows that pH values 2.5 give the shortest precipitation period for 200 rpm stirring speed and 60 minutes centrifugation time. The longest precipitation period is pH value of 3.5.

4.3 Analysis Result

$$\text{Efficiency of Al recovery} = \frac{[Al^{3+}]}{[H]\ Al^{3+} + [H]\ org + [H]\ pH}$$

$[Al^{3+}]$ – Concentration of aluminium detected from HACH 2000 Method of analysis

[H] – Concentration of sulphuric acid added to the sample.

4.3.1 Analysis For 100 rpm, 20 minutes

Table 4.7: Aluminum content in sample of 100 rpm, 20 minutes

pH values	alum recovered mg/L Al	% Al recovery efficiency
1.5	0.29	8.923076923
2.5	0.88	27.07692308
3.0	0.51	15.69230769
3.5	0.17	5.230769231

4.3.2 Analysis For 100 rpm, 40 minutes

Table 4.8: Aluminum content in sample of 100 rpm, 40 minutes

pH values	alum recovered mg/L Al	% Al recovery efficiency
1.5	0.71	21.84615385
2.5	1.29	39.69230769
3	0.02	0.615384615
3.5	0.65	20

4.3.3 Analysis For 100 rpm, 60 Minutes

Table 4.9: Aluminum content in sample of 100 rpm, 60 minutes

pH values	alum recovered mg/L Al	% Al recovery efficiency
1.5	0.08	2.461538462
2.5	1.09	33.53846154
3.0	0.35	10.76923077
3.5	0.41	12.61538462

4.3.4 Analysis For 200 rpm, 20 minutes

Table 4.10: Aluminum content in sample of 200 rpm, 20 minutes

pH values	alum recovered mg/L Al	% Al recovery efficiency
1.5	0.71	21.84615385
2.5	0.93	28.61538462
3.0	0.24	7.384615385
3.5	0.04	1.230769231

4.2.5 Analysis For 200 rpm, 40 minutes

Table 4.11: Aluminum content in sample 200 rpm, 40 minutes

pH values	alum recovered mg/L Al	% Al recovery efficiency
1.5	0.29	8.923076923
2.5	0.21	6.461538462
3.0	0.01	0.307692308
3.5	0.44	13.53846154

4.2.6 Analysis For 200 rpm, 60 minutes

Table 4.12: Aluminum content in sample 200 rpm, 60 minutes

pH values	alum recovered mg/L Al	% Al recovery efficiency
1.5	0.08	2.461538462
2.5	0.81	24.92307692
3.0	0.35	10.76923077
3.5	0.02	0.615384615

4.4 Result Discussion

From overall result, the best condition for aluminium sulphate recovery is at stirring speed of 100 rpm and period of centrifugation 40 minutes. This observation is proved by graph of volume recovery. For 100 ml alum sludge sample taken for experiment, volume recovered almost constant and high. The highest volume recovered from overall experiment also in this condition. This can be the best proof that 100 rpm stirring speed at 40 minutes of centrifugation period perform highest yield of alum recovery.

4.4.1 Stirring Speed

In terms of scientific explanation, every reaction need a stirrer to make sure the reaction perform in the overall solution. This will determine the yield of reaction. In natural condition every reaction take longer period and longer reaction time to give the effective result, but the reaction of alum recovery need an optimum condition to make sure the solution react at its best. So the result of 100 rpm stirring speeds proven to give the best yield for aluminium sulphate recovery.

4.4.2 Centrifugation Period

Normally, longer period of centrifugation give the best retention time. But, this is different with aluminium recovery, The best period of centrifugation is 40 minutes compare with 20 minutes and 60 minutes. This is proven to give the best result in the period of 40 minutes and the speed of 100 rpm.

4.4.3 Precipitation Period.

Precipitation period achieve when the solution of form a distinct fraction of solid and liquid. The distinction is visible every single experiment. Observation is done every one hour to be recorded in table of precipitation periods with pH values. Every set of samples observed for 24 hour before the composition sample filtrated.

From the observation, best retention time recorded is 16 hours. It means that 16 hours taken to settle solids in the bottom of the centrifugation bottle. So, complete separation of the solid with aluminium sulphate solution achieved for more than 16 hours.

From literature review the best retention time recorded for 17 hours. Compare to the literature review, this method of recovery gives the better result.

4.3.4 Aluminium recovery from analysis

Using HACH 2000 method, aluminium recovery in mg/l concentration the result give the best yield to the pH value of 2.5 in every set of sample. the most efficiency of aluminium recovery is 39.69 %. The best volume of aluminium recovered is 1.29 mg/l Al^{3+} . this value of efficiency recorded in 100 rpm stirring speed, 40 minutes period of centrifugation. From literature review, the efficiency of aluminium recovery is 33%. So my research is proven a better technique of alum recovery.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From overall result depicted, the best condition from alum sludge in water treatment tank to be recover are stirring speed at 100 rpm, and centrifugation period of 40 minutes. The best pH value for alum recovery is 2.5. This means that, alum sludge of pH 6.8 reacts more than 25% at pH value of 2.5.

So, in the process of alum sludge recovery, the sludge has to be treated with sulphuric acid (H_2SO_4), to form sludge solution of 2.5 pH values. If any water treatment plant practicing alum recovery I will suggest the pH value of 2.5. The critical condition applied on this recovery process is at stirring speed of 100 rpm and period of centrifugation of 40 minutes at 1000 rpm centrifugation speed.

I would like to consider my experiment as a success as I can achieved more that 30% concentration of aluminium sulphate in every 100 ml sample alum sludge. This process also regarded as an environmental friendly process. The waste of sludge is natural colloidal particle of solids which coagulated with aluminium sulphate. It can be easily disposed in the environmental as natural sludge. Moreover, this sludge is non-gelatinous that it can perform pure solid in the environment and not as slurry. This is practically different with alum sludge that it gelatinous in nature and it still remain in slurry form and gives a bad effect to health of direct contact with living organism.

5.2 Recommendation

This study is more accurate if the experiment done in the scheduled time. in term of time, there are inconstant period of every step for experiment. For example, the gap of the sample stirred and acidified with the time to perform process of centrifugation are not constant for every experiment. This will give the error in some retention period. during this experiment it is almost impossible to set the constant gap time between each and every experiment. This is because the lab equipments in use when the first experiment done. This situation will make the gap between one experiment to another is not constant. Some samples slowly settle down before taken into centrifuge.

I recommend that this experiment can be expanded to pilot plant scale. This alum recovery is believed to be the best method to prevent environmental pollution to the nature. Once this pilot plant of alum recovery is complete, the result of this plant can be applied to the water treatment plant. This is due to the cause that, aluminium presence in water consumed by people can bring disease if exposed in excess concentration. As we know, the amount of alum sludge settle beneath the water treatment plant cant be determined. So, in order to give the best service to people, we can apply this acidification process to the sedimentation tank.

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APPENDIX A

Calculation for Aluminium Recovery By Analysis Using HACH 2000

1. For 100 rpm Stirring speed, and 20 minutes centrifugation period

$$\text{Efficiency of Al recovery} = \frac{[Al^{3+}]}{[H]\,Al^{3+} + [H]\,org + [H]\,pH} \times 100\%$$

$[Al^{3+}]$ – Concentration of aluminium detected from HACH 2000 Method of analysis

[H] – Concentration of sulphuric acid added to the sample.

1.1 pH value of 1.5

$$\text{Efficiency of Al recovery} = \frac{[0.29]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 8.923076923\ \%$$

1.2 pH Value of 2.5

$$\text{Efficiency of Al recovery} = \frac{[0.88]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 27.07692308\ \%$$

1.3 pH value of 3.0

$$\text{Efficiency of Al recovery} = \frac{[0.51]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 15.69230769\ \%$$

1.4 pH value of 3.5

$$\text{Efficiency of Al recovery} = \frac{[0.17]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 5.230769231\ \%$$

2.0 For 100 rpm stirring speed, 40 minutes centrifugation period

2.1 pH Value of 1.5

$$\text{Efficiency of Al recovery} = \frac{[0.71]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 21.84615385\ \%$$

2.2 pH value of 2.5

$$\text{Efficiency of Al recovery} = \frac{[1.29]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 39.69230769\ \%$$

2.3 pH value of 3.0

$$\text{Efficiency of Al recovery} = \frac{[0.02]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 0.615384615\ \%$$

2.4 pH value of 3.5

$$\text{Efficiency of Al recovery} = \frac{[0.65]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 20\ \%$$

3.0 For 100 rpm stirring speed, 60 minutes centrifugation period

3.1 pH value of 1.5

$$\text{Efficiency of Al recovery} = \frac{[0.08]}{0.0 + 0.75 + 2.5} \times 100\%$$

$= 2.461538462\ \%$

3.2 pH value of 2.5

$$\text{Efficiency of Al recovery} = \frac{[1.09]}{0.0 + 0.75 + 2.5} \times 100\%$$

$= 33.53846154\ \%$

3.3 pH value of 3.0

$$\text{Efficiency of Al recovery} = \frac{[0.35]}{0.0 + 0.75 + 2.5} \times 100\%$$

$= 10.76923077\ \%$

3.4 pH value of 3.5

$$\text{Efficiency of Al recovery} = \frac{[0.41]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 12.61538462\ \%$$

4.0 For 200 rpm stirring speed, 20 minutes centrifugation period

4.1 pH value of 1.5

$$\text{Efficiency of Al recovery} = \frac{[0.71]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 21.84615385\ \%$$

4.2 pH value of 2.5

$$\text{Efficiency of Al recovery} = \frac{[0.93]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 28.61538462\ \%$$

4.3 pH value of 3.0

$$\text{Efficiency of Al recovery} = \frac{[0.24]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 7.384615385\ \%$$

4.4 pH value of 3.5

$$\text{Efficiency of Al recovery} = \frac{[0.04]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 1.230769231\ \%$$

5.0 For 200 rpm stirring speed, 40 minutes centrifugation period

5.1 pH value of 1.5

$$\text{Efficiency of Al recovery} = \frac{[0.29]}{0.0 + 0.75 + 2.5} \times 100\%$$

$$= 8.923076923\ \%$$

5.2 pH value of 2.5

$$\text{Efficiency of Al recovery} = \frac{[0.21]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 6.461538462 %

5.3 pH value of 3.0

$$\text{Efficiency of Al recovery} = \frac{[0.01]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 0.307692308 %

5.4 pH value of 3.5

$$\text{Efficiency of Al recovery} = \frac{[0.44]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 13.53846154 %

6.0 For 200 rpm stirring speed, 60 minutes centrifugation period

6.1 pH value of 1.5

$$\text{Efficiency of Al recovery} = \frac{[0.08]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 2.461538462 %

6.2 pH value of 2.5

$$\text{Efficiency of Al recovery} = \frac{[0.81]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 24.92307692 %

6.3 pH value of 3.0

$$\text{Efficiency of Al recovery} = \frac{[0.35]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 10.76923077 %

6.4 pH value of 3.5

$$\text{Efficiency of Al recovery} = \frac{[0.02]}{0.0 + 0.75 + 2.5} \times 100\%$$

= 0.615384615%

APPENDIX B

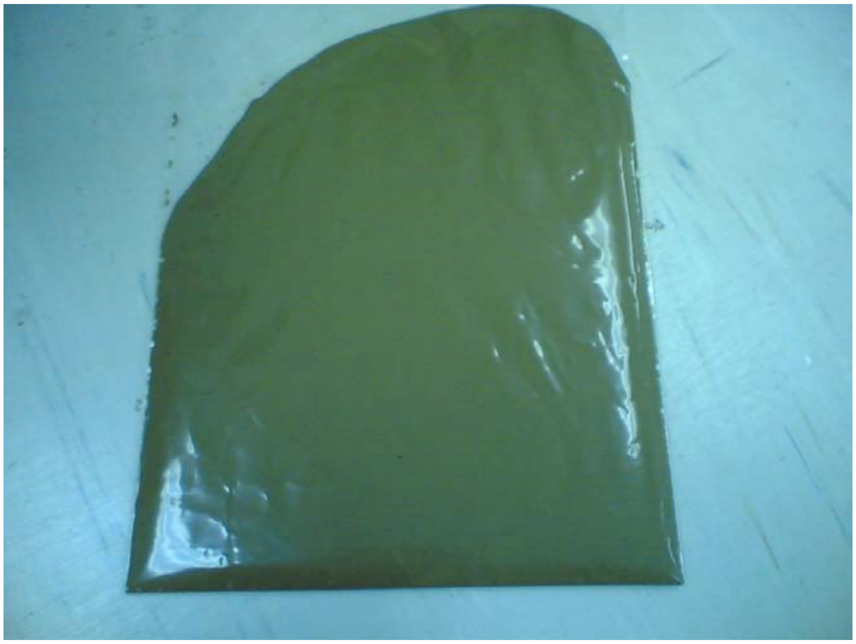

Figure B-1: Alum Sludge sample

Figure B-2: Alum sludge sample proven for gelatinous property

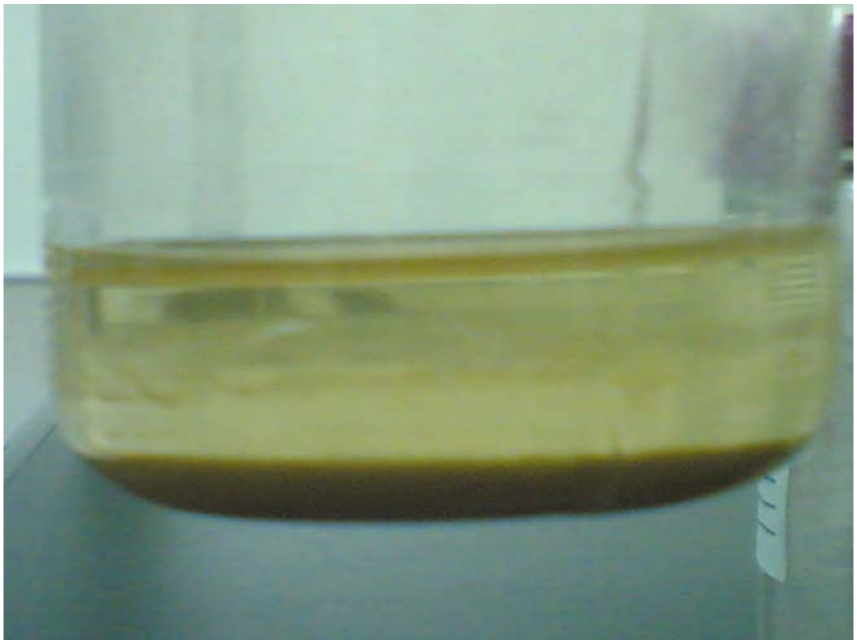

Figure B-3: Process of settling/ precipitate of alum sludge sample

Figure B-4: Colloidal solids extracted from alum sludge

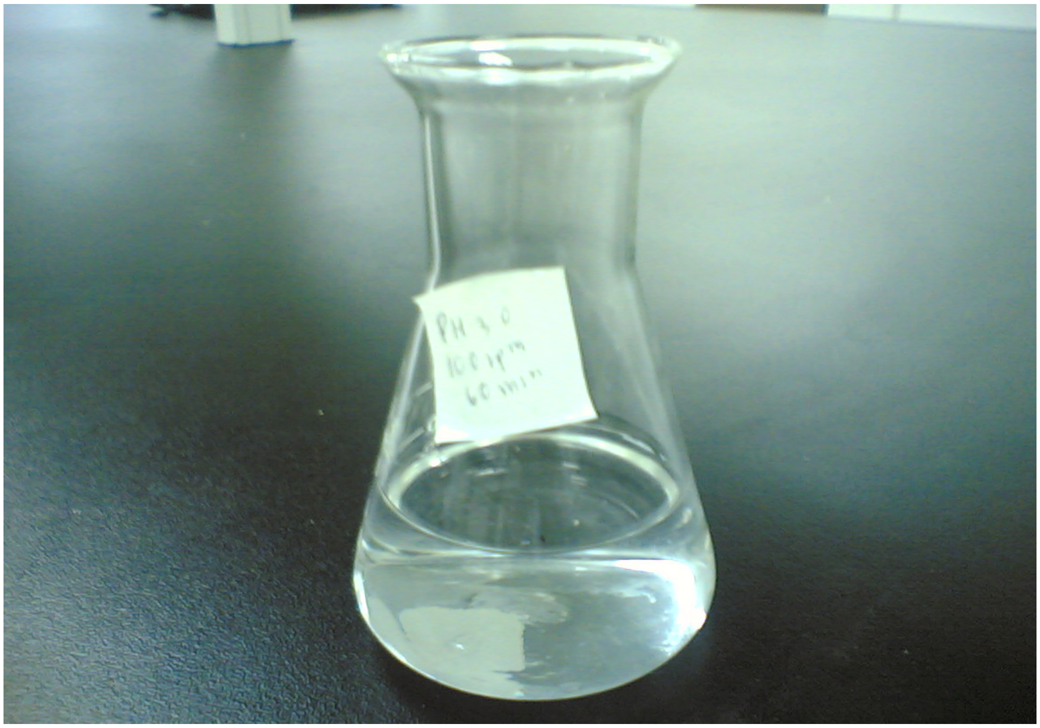

Figure B-5: Supernatant Aluminium Sulphate extracted from alum sludge

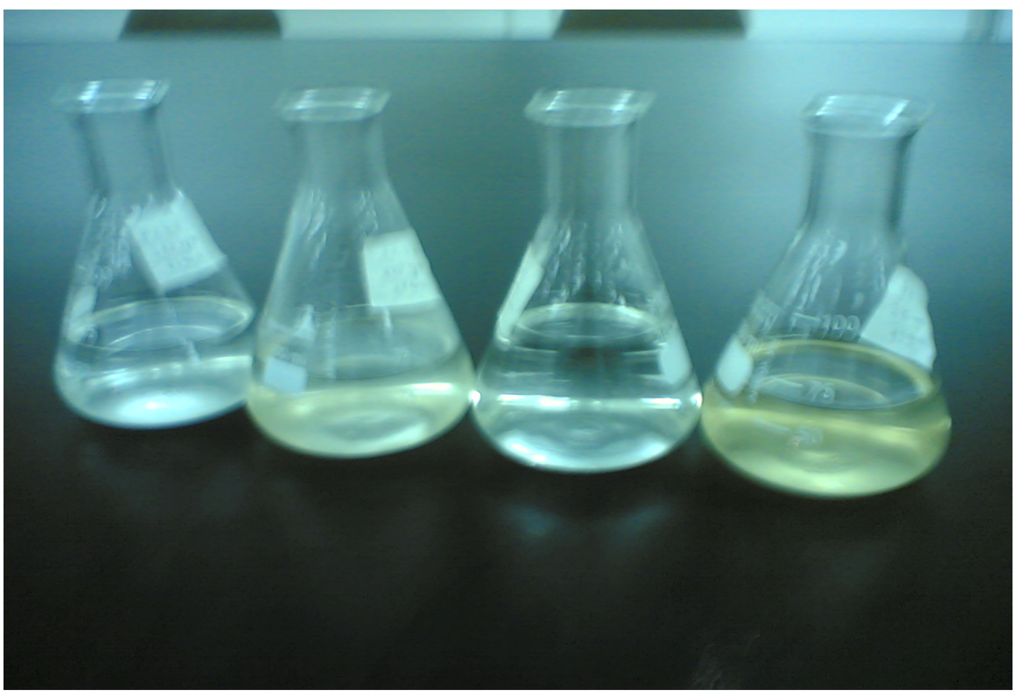

Figure B-6: Aluminium Sulphate recovered from Alum Sludge