

# **ALUM RECOVERY FROM ALUM SLUDGE**

**(A CASE STUDY ON THE IMPRESIT WATER TREATMENT  
PLANT, CHACHANGA WATER WORKS, MINNA, NIGER  
STATE)**

**BY**

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THE AWARD OF THE DEGREE OF BACHELOR OF  
ENGINEERING, (B. ENG)**

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## DECLARATION

I, BALOGUN EBENBEN JOYCE, hereby declare that the project 'ALUM RECOVERY FROM ALUM SLUDGE (A CASESTUDY ON IMPRESIT WATER TREATMENT PLANT, CHANCHANGA WATER WORKS, MINNA, NIGER STATE)' was an original work done by me to the best of my knowledge has not been presented either wholly or in part for the award of degree or similar qualifications.

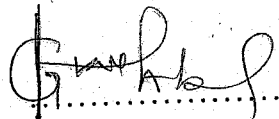
Balogun Ebenben Joyce 27/11/07

Signature/Date

# CERTIFICATION

This is to certify that this project titled ALUM RECOVERY FROM ALUM SLUDGE,(A CASESTUDY ON THE IMPRESIT WATER TREATMENT PLANT ,CHANCHANGA WATER WORKS , MINNA,NIGER STATE) was carried out originally by BALOGUN EBENBEN JOYCE of the department of Chemical Engineering, Federal University of Technology, Minna, Niger State.

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## DEDICATION

This project is dedicated to God Who is ever faithful to His word and has been faithful to me, to my parents Mr. and Mrs. Balogun ,for their undying support all through my years of study and to my sibling, Sharon, Osebi, Mosimabale, Osijobakwemu and Mosebue.



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## ABSTRACT

The research work was carried with the objective of recovering alum from alum sludge which is a waste product from water treatment plants. The Impresit water treatment plant was my case study. Acid extraction method was employed in recovering the alum. During the alum recovery process from alum sludge, the sludge from the water treatment plant containing aluminum hydroxide was acidulated with tetraoxosulphate VI acid to produce alum from the hydroxide. The sludge was acidulated until a pH of 2 was gotten. Then it was left to settle down for twenty four hours. The volumetric recovery was about 61%. The aluminum content of the alum solution was 0.466mg/L while that of the recovered alum was 0.357mg/L for the unfiltered recovered alum solution and 0.379mg/L for the filtered recovered alum solution. This indicates that the aluminum content of the recovered alum was between 76% and 80%.

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# CHAPTER ONE

## 1.0 INTRODUCTION

Surface water treatment plants particularly for large municipalities include a coagulation stage in which alum (aluminum sulphate) is commonly used in the coagulation and clarification process. Alum is the most widely used coagulant for efficient removal of particulate solids and colloids from surface water supplies in water treatment works around the world. During the treatment, alum is converted into insoluble aluminum hydroxide. Particulates colloidal and soluble contaminants are entrapped or absorbed on aluminium hydroxide flocs. This are a major component of the solid's water treatment plant sludge; which is also known as water treatment residual. The water treatment residuals (sludge) is essentially a bulky, gelatinous slurry composed of suspended inorganic particles, natural organic matters, trace amounts of heavy metal precipitate and aluminum hydroxide (SenGupta and Prakash, 2004).

Water treatment plants in the United States of America alone produce over two million tons of aluminum – laden water treatment residuals (sludge) everyday. Because of the magnitude and pervasive nature of the problem, the prospect of alum recovery from the sludge and its possible re-use has received considerable attention in recent years. A study of alum recovery and re-use was also performed by Malcolm, Pirnie Incorporated with assistance from the city of Durham, division of water resources. The purpose of the study was to investigate the potential alum recovery for solid reduction and improve solid handle-ability, and to produce a supply of recovered alum which can be recycled or re-used by the city (Malcolm, Pirnie, 1984).

The Impresit water treatment plant is one of three water treatment plants which provide portable drinking water to the city of Minna, Niger state; and its environment. It is located at the

Chanchaga Water Works, Minna, Niger State. Treatment processes at the Niger state water works includes screening, coagulation, flocculation, sedimentation and filtration. The raw water comes from a dam located at about ten kilometers away from the treatment plant. It is passed through a sieve where screening takes place. This done was to remove large particles of debris. The water is then passed into a tank where coagulation takes place. Alum solution is passed into the water which makes the tiny particle to coagulate. Thereafter it is passed into a sedimentation tank where the flocs formed settles down at the base of the tank. The water is then passed through a filter bed which traps any other particles that escaped from the sedimentation tank. After filtration, the water is disinfected with chlorine then it is stored in the storage tank.(Water board manual,2002)

However in the sedimentation unit the residue which is the sludge is discharged or dislodged at least twice a week and as the need demands. Current practice at the water treatment plant is to discharge the water treatment residuals (sludge) directly into the nearest body of water. The sludge from the four sedimentation tanks is dislodged; it moves to the drainage designed for that purpose. The filtration unit is also backwashed to remove the accumulated dirt on it. This is done at least three times a week. The backwash waste water is collected by the drainage channel when backwashing process is carried out. The drainage channels take the waste water directly to the nearest body of water. There is no form of treatment carried out on the sludge. (Water Board Manual 2002)

The capacity of the solid facilities presently available in the water works could be expanded so that treatment residuals (sludge) can be processed. However, the difficulty will be the construction of an additional process unit where the sludge can be treated. The site where the

Impresit treatment plant is located is shared by two other treatment plants. Room would not be available to add any more process unit to the already existing ones.

An alternative would be to construct a new plant on a different site and modification on the impresit plants be made. This modification should adequately address the potential for alum recovery and re-use.

## **1.2 OBJECTIVES**

The aim of this study is the recovery of alum from the chemical sludge produced at the Impresit Water Treatment plant (Minna, Niger State) by acidic extraction. The effect of some operating parameters such as pH and settling time on the extraction efficiency is also investigated.

## **1.3 SCOPE**

The focus of this research work was the potential for alum recovery to reduce pollution of the water body located close to the Impresit Water Treatment Plant, at the Chanchaga Water Works, Minna, Niger State.



# CHAPTER TWO

## 2.0 LITERATURE REVIEW

### 2.1 HISTORY OF WATER TREATMENT

The history of water treatment is still being written as discoveries continue to document its origins. However, there is evidence that even in ancient times; people saw the importance of treating water in some way before drinking it. (Wikipedia)

In ancient Greek, and Sanskrit (India), writings dating back to 2000B.C, water treatment methods were recommended. People back then knew that heating water could purify it and they were also educated in sand and gravel filtration, boiling and straining. The major reason for water purification was for better drinking water, because people could not at that time distinguish between foul and clean water. Turbidity was the main driving force for the earliest water treatments. Not much was known about micro-organic chemical contaminants (Baker and Taras, 1981).

Ancient Egyptians treated water by siphoning water out of the top of huge jars after allowing the muddy water from the Nile River to settle. After 1500B.C, the Egyptians first discovered the principle of coagulation. They applied the chemicals for suspended particle settlement. After 500B.C, Hippocrates, known as the father of medicine discovered the healing powers of water. He inverted the practice of sieving water and obtained the first bag filter which was called the "Hippocratic sleeve". The main purpose of the bag was to trap sediments that caused bad tastes or odour. He directed people in Greece to boil and strain water before drinking it (Baker and Taras). In turn, the Romans passed water from the aqueducts through settling basins to clarify it (remove impurities). Back then, the focus was on the aesthetic quality of the water, i.e. if the water was clear and had no smell, it was considered clean. (Wikipedia)

After the fall of the Roman Empire, enemy forces destroyed many aqueducts, and others were no longer applied. The future for water treatment was uncertain. Then, in 1627 A.D, the water treatment history continued as Sir Francis Bacon started experimenting with sea water desalination. He attempted to remove salt particles by means of unsophisticated form of sand filtration. It did not exactly work, but it did paved the way for further experimentation by other scientist. (Baker and Taras, 1981)

In the 1700s, the first water filters for domestic applications were applied. These were made of wool, sponge and charcoal. In 1804, the first actual municipal water treatment plant was designed by Robert in Scotland (Baker and Taras, 1981). It was the first water facility to deliver water to an entire town. It was built in Paisley, Scotland in 1804 by John Gibb, to supply his bleacher and the town. And within three years, filtered water was even piped directly to customers in Glasgow, Scotland (Wikipedia).

In 1806, a large water treatment plant began operating in Paris. The plant's filters were made of sand and charcoal and were renewed every six hours. Water was settled for twelve hours before filtration. In 1827, Englishman James Simpson built a sand filter for drinking water purification (Wikipedia, the free encyclopedia).

In 1854, it was discovered that cholera epidemic spread through water. The outbreak seemed less severe in areas where sand filters were installed. British scientist John Snow found that the direct cause of the outbreak was water pump contamination by sewage water. He applied chlorine to purify the water and this paved the way for water disinfection. Since the water in the pump had tasted and smelled, the conclusion was finally drawn that good taste and smell alone do not guarantee safe drinking water. This discovery led to governments starting to install municipal water filters and hence the first government regulation of public water (Enzler 1996).

Before the end of the century, the filter capacity was improved. It was also discovered that filtration works much better when it is preceded by coagulation and sedimentation techniques.

However, in 1902A.D, calcium hypo-chloride and ferric chloride were mixed in a drinking water supply in Belgium, resulting in both coagulation and disinfection. In 1903 water softening was inverted as a technique for water desalination. Also in 1906 ozone was first applied as a disinfectant in France. Eventually, starting in 1914A.D, drinking water standards were being implemented for water supplies in public traffic, based on coliform growth. It would not be until the 1940s before drinking water standards applied to municipal drinking water. In 1972A.D, the clean water act was passed in the United States. Then in 1974A.D, the Safe Drinking Water Act (SDWA) was formulated. The general principle in the developed world was that every person had the right to safe drinking water (EPA, 2000).

Starting in 1970A.D, public health concerns shifted from water borne illness caused by disease causing micro-organisms, to anthropogenic water pollution such as: pesticide residue and industrial sludge are organic chemicals. Regulations now focused on industrial waste and industrial water contamination, water treatment plants were adopted. Techniques such as aeration, flocculation and active carbon adsorption were applied. In the 1980s, membrane developments for reverse osmosis were added to risk assessment were enabled after 1990A.D (EPA, 2000).

Water treatment experimentation today mainly focuses on disinfection by-products. An example is trihalomethane (THM) formation from chlorine disinfection. These organics were linked to cancer. Lead also became a concern after it was discovered that it corrodes water pipes. The high pH level of disinfected water enabled corrosion. Today, other materials have replaced

lead water pipes especially in developed countries. (Outwater, 1996).

## 2.2 PROCESSES INVOLVED IN WATER TREATMENT

Water is perhaps the most important nutrient in our diets. Some water sources can not be safely used to meet our requirements for drinking water. For instance, 99.7 percent of the Earth's water supply is not usable by humans. This un-usable water includes salt water, ice, and water vapour in the atmosphere. Only freshwater, which is contained in the rivers, lakes and underground sources, can be used for human consumption. Furthermore, many freshwater sources are not suitable for humans to drink. (Casiday et al 2002)

In addition to the water needed for drinking, humans use much larger amounts of clean freshwater in other applications. Hence, the quality of the fresh water is important for virtually every aspect of our lives. Thus, our water supplies are not pure. That is, these supplies contain other species that may make the water unsuitable for human use.

In response to this need, the government at all levels have formed organizations and passed legislations to monitor, treat and protect our water supplies. The Clean Water Act for instance in 1972A.D was passed and revised in 1978A.D. Later, the Safe Drinking Water Act of 1974A.D (amended in 1986A.D and 1996A.D) established minimum federal public-health standards for water supply. (As adopted by Enzler, 1996).

Nigeria is not left out. Section one sub-section 15, number one of the Federal Environmental Protection Agency Act provides a regulation too. It says: the agency (FEPA) made recommendations to the President, Commander-in-Chief of the Armed Forces for the purpose of establishing water quality standards for the inter-state water of Nigeria to protect the public health or welfare and enhance the quality of water to serve the purpose of the Act. (As adopted by Azeez 2000)

Public water facilities treat our water to make it safe for us to drink and appropriate for other human uses. There are four major steps in the treatment of our water. They are screening, sedimentation, filtration, and disinfection.

### 2.2.1 SCREENING

Surface waters (water from lakes and rivers) often have large debris such as sticks, leaves, fish and trash floating on it. These objects must be removed before the water enters the treatment plant. Treatment facilities have large screens covering the site of water intake. This ensures that the large debris is removed. The screen must however be cleaned periodically to remove any object that have become stuck, so that they do not clog the screen and impede water flow into the plant. (Casidey et al 2002)

### 2.2.2 SEDIMENTATION

Other suspended (insoluble) particles, such as sand and dirt, are small enough to pass easily through the screens. These particles must be removed from the water by another means called Sedimentation. This is achieved by two processes known as flocculation and coagulation. These processes are used to create larger particles that will settle quickly to the bottom. In flocculation, small particles with non-rigid surfaces are made to agglomerate by mixing the water. When the agglomeration of the particles gets large enough, the aggregate can settle in still water by sedimentation. Other suspended particles that did not agglomerate are used by coagulation. Coagulation is the process of gathering particles into a cluster or a clot, often achieved by the addition of some special chemicals known as coagulants. The most common coagulant used in water treatment facilities is aluminum sulphate (alum,  $Al_2(SO_4)_3$ ). Others include poly-aluminum chloride, ferric chloride, and ferric sulphate. These salts react with ions naturally found in the water to produce a solid precipitate. As these precipitates form, other



particles are caught in the solid forming a mass that will settle to the bottom via sedimentation. (Casidey, etal, 2002)

### 2.2.3 FILTRATION

Often, the particles generated by precipitation are too small to settle efficiently by sedimentation. Filtration is therefore employed to remove these solids. In this process, water containing solid impurities (e.g. precipitates from water softening) is passed through a porous medium, typically layers of sand and gravel. The force of gravity is used to push the water through the medium. The small water molecules pass through the holes between the sand and gravel pieces.

However, the solids (from precipitation) get stuck in the holes, and are thus retained in the porous medium. The water that passes through the bottom of the filter no longer contains those solid impurities. Pressure filters have also been standardized during the last 50 years, but limited to small plants. Experience is still lacking for plants that are very large. (Culp etal 1986)

Gravity filters at water treatment plants have a pipe feeding into the under drain, the bottom layer where the clean water is collected. By adding water to the filter through this pipe, clean water can be forced upward through the filter to remove the solids that had been collected in the filter. This process is used to clean the filter. (Casidey etal 2002)

### 2.2.4 DISINFECTION

In many water supplies, the most serious health threats are posed not by chemicals, but by infectious organisms (bacteria) in the water. Chlorine is a major disinfectant that is cheap and kills most of the serious disease causing bacteria in the water. However, chlorine disinfection results in a wide variety of by-products. One class of chlorine by-products, known as tri-halo-

methane (THM) are suspected carcinogens. Because of the concerns about these by-products in the water supply, chlorine is now kept in minimal levels and other methods of disinfection are being used more frequently. Chloramines form more stable disinfectants and pose less risk of harmful by-products, but cost more to use. Other methods focus on removing the organisms through coagulation, sedimentation and improved filtration.

Below is a flowchart that shows the path water takes in the intake of the water treatment plant to the storage tank, from which it is pumped to homes, businesses and industries. This may vary in steps and sequence from one treatment plant to another.

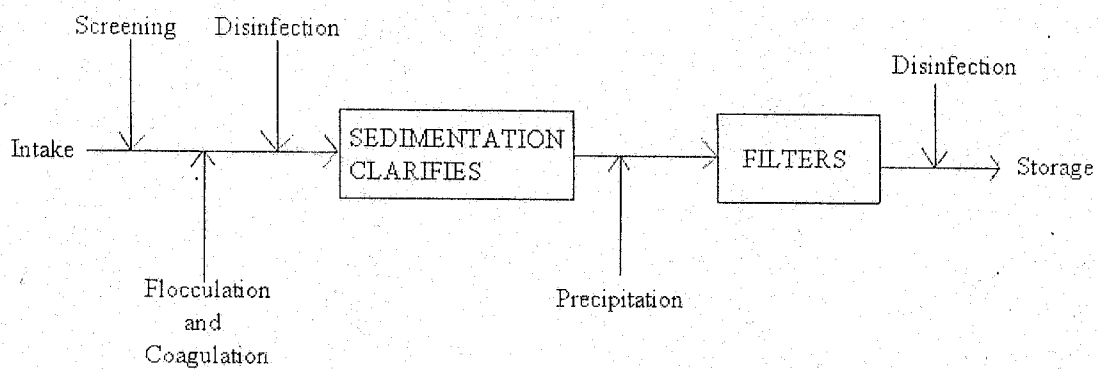


Fig 1.1 Schematic diagram of a water treatment plant.

### 2.3.0 ALUMINUM SULPHATE

Aluminum sulphate (alum) is one type of flocculant. Flocculants or flocculating agents are chemicals that are used to promote flocculation by causing colloids and other suspended particles in liquids to aggregate, forming a floc. Flocculants are used in water treatment processes to improve the sedimentation or filterability of small particles. For example, a flocculant may be used in swimming pools or drinking water filtration to aid the removal of

microscopic particles which would otherwise cause the water to be cloudy and which would be difficult or impossible to remove by filtration alone.

Many flocculants are multivalent cations such as aluminum, iron, calcium or magnesium. These positively charged molecules interact with negatively charged particles and molecules to reduce the barriers to aggregation. In addition, many of these chemicals, under appropriate pH and other conditions, react with water to form insoluble hydroxides which, upon precipitating, link together to form long chains or meshes, physically trapping small particles into the larger floc. Factors such as pH, temperature and salinity can induce flocculation or influence the flocculation rates.

Aluminum sulphate could also serve as a coagulant. The terms flocculants and coagulants are sometimes used interchangeably, but it is more accurate to use the term coagulant for a chemical that contributes to molecular aggregation rather than particular aggregations. Usually, dissolved substances are aggregated into microscopic particles by a coagulant and then these particles may be flocculated into macroscopic floc with a flocculant. In general, coagulants will have higher net charge and a lower molecular weight than flocculants. Thus, coagulation is the irreversible clumping of particles that is; caking had occurred.

The following are other chemicals that could serve as coagulants:

1. Aluminum chlorohydrate
2. Calcium oxide
3. Iron III chloride
4. Iron II sulphate
5. Sodium aluminate
6. Sodium silicate

Other products that could be used as flocculants but are natural products include:

1. Chitosan
2. Moringa oleifera
3. Papain
4. A species of strychnos (seed)
5. Isinglass
6. Clay

### 2.3.1 COMPOSITION OF ALUMINUM SULPHATE

Alum is aluminum sulphate. Concentration and addition amounts are usually based on either:

- a. The equivalent amount of aluminum II oxide ( $AL_2O_3$ ) or
- b. The equivalent amount of the hydrate  $AL_2O_3 \cdot 14H_2O$ .

Alum is most commonly delivered as a liquid concentration, having a solids level of 8.3% as  $AL_2O_3$  or about 50% as hydrate. Alum solutions are acidic. For instance, a 1% solution has a pH of about 3. Ionic species present in alum solutions are highly dependent on the degree of reaction with hydroxyl ions. It is tempting to say that "the composition is pH dependent". However, in the vicinity of pH = 4.3, the composition of alum solutions changes a great deal with very little change in pH. At pH = 3 and lower, the main species is a hydrated form of aluminum ion ( $AL_3$ ). At a pH close to 4.3, there may be oligomers such as  $AL_{13}O_{40}H_{48}^{7+}$  or related species that contain sulphate. In a broad range of pH between about 6 and 9.5, the main species will be aluminum hydroxide  $AL(OH)_3$  (and possibly related species that contain sulphate). The soluble aluminate ion predominates at pH values greater than 9.5 (Mini-Encyclopedia).

### 2.3.2 OTHER USES OF ALUMINUM SULPHATE

A general discussion of alum use strategies has to start with the subject of pH. At pH values of about 4.5; for example, lower alum can be described as a forgiving additive. Thus, the user has a lot of flexibility with respect to where and how much alum to be used for rosin, sizing and damage.

Usually, alum is added after rosin soap size to set the size onto the fiber surface in paper making. Alum plays a very effective role in alkaline paper making, even though the equilibrium species are uncharged. Apparently, the alum is able to rapidly complex with and neutralizes carboxylate species in neutral or alkaline furnish before it has reached its equilibrium ionic composition.

In summary, alum is used for drainage enhancement, rosin sizing, part of certain retention aid programs, dye fixation, cat-ionic source, and acidic buffer.

### 2.4 HISTORY/ORIGIN OF ALUMINUM SULPHATE

Aluminum potassium sulphate was used throughout the history of paper making until the 19<sup>th</sup> century. It was then replaced by the newly developed aluminum sulphate, a cheaper and more concentrated source of aluminum compound.

In the mid 15<sup>th</sup> century, the first European alum mines were exploited in Tolfa, a volcanic area north of Rome in central Italy. Slate and shale were other minerals which yielded alum when subjected to several production steps which can be summarized as extracted in water, potassium hydroxide was added to the resulting solution; the crude alum crystals which formed in the evaporating solution were rinsed and re-dissolved in boiling water to purify the alum; the solution was transferred to large wooden casks where the alum crystals formed on the inside



walls; and finally the casks were dismantled and the crystals removed. Alum could be contaminated with by-products of its manufacture, iron-oxides and iron-sulphates. Iron compounds significantly impaired the performance of alum. The repeated re-crystallization of the alum effectively freed it from iron contaminants.

Aluminum sulphate became an industrial product in the 19<sup>th</sup> century. It was made by either bauxite or china clay with sulfuric acid. It could not be conveniently purified through re-crystallization because of its greater solubility in water. This is one of the reasons why it often contained varying proportions of silica, iron and free sulfuric acid.

By the early 20<sup>th</sup> century, commercial aluminum sulphate varieties were relatively uniform in quality. They were ranked according to grading systems and could be purchased in solid pieces as so-called "patent alum". Well known varieties include cake alum, porous alum, and turkey-red alum. Because of its greater concentration of alumina ( $Al_2O_3$ ) and cheaper production procedures, aluminum sulphate saved mill expenses and therefore replaced aluminum potassium sulphate for most purposes in the mid-19<sup>th</sup> century.

## **2.5 WASTE DISPOSAL FROM WATER TREATMENT PLANT**

In the past, waste products from water treatment plants were simply discharged to the nearest body of water. All waste products from water treatment plants could be disposed through disposal facilities. With increasingly stringent anti-pollution standards, disposal is however troublesome and expensive. However, some requirements must be considered and given minimum requirements as state water pollution control authorities may have more stringent requirements. Provisions are needed for proper disposal of water treatment plant waste such as; sanitary waste, laboratory waste, clarification sludge, softening sludge, and filter backwash

water. In locating waste potential disposal facilities, due considerations shall be given to prevent potential contamination of water supply.

Alternative methods of water treatment and chemical use should be considered as a means of reducing waste volumes and the associated handling and disposal problems. Appropriate backflow protection must be provided on waste discharge piping as needed to protect the public water supply.

### **2.5.1 SANITARY WASTE**

The sanitary waste from the water treatment plants, pumping stations, and other water works installations must receive treatments. Waste from these facilities shall be discharged directly to a sanitary sewer system, when available and feasible, or to an adequate on-site waste treatment facility approved by the appropriate reviewing authority.

### **2.5.2 PRECIPITATIVE SOFTENING SLUDGE**

Sludge from plants using precipitative softening varies in quality and in chemical characteristics depending on the softening process and the chemical characteristics of the water being softened. Recent studies show that the quantity of sludge produced is much larger than indicated by stoichiometric calculations. Methods of treatments and disposal are as follows:

**2.5.2.1 Lagoons:** - temporary storage lagoons which must be cleaned periodically should be designed. This should provide about two and a half years storage. At least two, but preferably more lagoons must be provided in order to give flexibility in operation. An acceptable means of final sludge disposal must be provided.

**2.5.2.2** The application of liquid lime or dewatered sludge to farm land could be considered as a method of ultimate disposal. Prior to land application, a chemical analysis of the sludge including calcium and heavy metals should be conducted. Approval from the appropriate reviewing authority shall be obtained.

**2.5.2.3** Discharge of lime sludge to sanitary sewer should be avoided since it may cause both liquid volume and sludge volume problems at the sewage treatment plants. This method should be used only when the sewerage system has the capability to adequately handle the lime sludge.

**2.5.2.4** Mixing of lime sludge with activated sludge waste may be considered as a means of co-disposal.

Mechanical dewatering of sludge may be considered. Mechanical dewatering shall be preceded by sludge concentration and chemical pretreatment.

### **2.5.3 ALUM SLUDGE**

In a raw water treatment system in which alum is added to the water undergoing treatment and followed by steps including coagulation-flocculation, settling and filtering to produce product water, the treatment resulting in a sludge containing aluminum hydroxide. This sludge can be handled in a number of ways, including:

1. Lagooning, which may be used as a method of handling alum sludge. A lagoon is a body of water cut off from open sea. The size of a lagoon can be calculated using total chemicals used plus a factor for turbidity.

2. Mechanical concentration may be considered. A pilot plant study is required before the design of a mechanical dewatering installation. Freezing changes the nature of alum sludge so that it can be used for fill.

3. Acid treatment of sludge for alum recovery may be a possible alternative. This would be elaborated later in this chapter.

4. Alum sludge can be discharged to a sanitary sewer. However, initiation of this practice would depend on obtaining approval from the owner of the sewerage system as well as from the regulatory agency before final designs are made.

However, lagoons could be designed to produce an effluent satisfactory to the regulatory agency and should provide for the following:

- a. Location free from flooding
- b. Where necessary, dikes, deflecting gutters, or other means of diverting surface water so that it does not flow into the lagoon
- c. A minimum usable depth of five feet
- d. Adequate free board of at least two feet
- e. Adjustable decanting device
- f. Effluent sampling point Adequate safety provision, and
- g. A minimum of two cells, each with appropriate inlet and outlet structures to facilitate independent filling/dewatering operations.

The successful use of mechanical dewatering depends on the characteristics of the alum sludge produced, as determined by site specific studies. However, mechanical dewatering shall be preceded by sludge concentration and chemical pre-treatment.

Land application could also be considered. The alum sludge may be disposed of by land application either alone, or in combination with other waste where an agronomic value has been determined or disposal has been approved by the reviewing authority.

#### 2.5.4 WASTE FILTER WASH WATER

Filtration is one of the unit operation processes involved in the treatment of water. This takes place in filter beds; which are washed from time to time. The process of washing the filter bed is known as backwashing. Waste filter wash water have suspended solids. These suspended solids should be reduced to a level acceptable to the regulatory agency before being discharged. However, the plants used as case study has its waste filter wash water passed into the drainage channel and it is disposed into the nearest water body. It is recommended that plants should have constructed holding tanks that should return this water to the inlet end of the plant. The holding tank should have the capacity of containing the anticipated volume of waste wash water produced by the plant when operating at design capacity.

A treatment plant that has two filters should have a holding tank that would contain the total waste wash water from both filters calculated by using a fifteen minutes wash at twenty gallons per minute per square foot. In plants with more filters, the size of the holding tank will depend on the anticipated hours of operation. It is recommended that waste filter wash water be returned at the rate of less than ten percent of the instantaneous raw material flow rate entering the plant.

#### 2.6 ALUM RECOVERY FROM ALUM SLUDGE

Aluminum sulphate (commonly known as alum),  $AL_2(SO_4)_3 \cdot 14H_2O$  is the most widely used coagulant for the effective removal of particulate solids and colloids from surface water supplies in water treatment plants around the world. A chemical sludge containing aluminum hydroxide, adsorbed organic matter and other water insoluble impurities is composed of aluminum hydroxide because the aluminum sulphate is converted essentially to it and it's the major



component of the sludge. The sludge generated must be disposed of in an environmentally sound manner. The sludge which could also be termed water treatment residuals (WTR) generated is composed of aluminum hydroxide; this is because the aluminum sulphate is converted essentially to aluminum hydroxide which is the major component of the sludge (between 25% and 60%). Other components of the water treatment residuals includes: suspended inorganic particles, natural organic materials and trace amounts of heavy precipitates.

Water treatment residuals from the treatment plants were simply disposed into the nearest body of water, without any prior treatment. Disposal into landfills, water ways or through application to land is a concern in both developed and developing countries and it is receiving close scrutiny for its high aluminum content. Thus, there are anti-pollution standards in developed countries that have been put in place to checkmate these activities. For instance, in the city of Durham, North Carolina, the sludge generated in nearly all the treatment plants were ultimately disposed of in a landfill. However, in the year 1973, it was estimated that about 25000 tons of alum sludge was produced in North Carolina each year.

It has previously been proposed to dewater the waste sludge in a filter press, after suitable thickening and treatment with lime, thereby; producing cake which can be trucked away and used for landfill, etc. with large amounts of waste, large filter press capacity is required and new alum supplied as required for the treatment process. The disposal of alum sludge in a landfill results in the loss of a valuable alum asset and at the same time depletes the capacity of the landfill.

Water treatment plants in other developed countries e.g. the United States, alone produces over two million tons of aluminum land water treatment residuals everyday. Also in Portugal, an estimated amount of 66,000tonnes per year (wet weight) water treatment sludge is



being disposed of on land or at municipal solid waste (MSW) landfills (Boaventura et al 2000). Because of the magnitude of pervasive nature of the problem, the prospect of alum recovery from water treatment residuals and its re-use has received considerable amount of attention in the last three to four (3 – 4) years. Government restrictions to the practice of disposing this sludge on land or at municipal solid waste landfills as well as increasing deposition cost and the potential harmful impacts proceeding from the high aluminum content of the sludge have also lead to significant research efforts in order to evaluate different treatment alternatives namely aluminum recovery and subsequent reuse. The dewatering and disposal of alum sludge adds significantly to the cost of treating water. Alum recovery and re-use could reduce those costs.

In some developing countries, e.g. Nigeria, attention has not yet been drawn to this area. At the Impresit water treatment plant located in Chanchaga water works in Minna, Niger state, the water treatment residuals is disposed directly into the nearest body of water, thus, pollution the water body. There are no anti-pollution standards protecting these bodies of water. Even if there are, there is no law enforcing agency that ensures that these standards are not violated or the law is not broken. Alum recovery could be one of the standards as these would reduce the amount of alum wasted, ensure re-use and in the long run this would reduce cost.

Systems have been described and attempts have been made to recover alum. Membrane based separation and liquid ion exchange processes have been studied for this purpose. Acidic and alkaline extraction methods are still been explored to obtain a product susceptible of use as coagulant for industrial waste water treatment processes (Boaventura et al, 2000).

Full scale testing was conducted at the Williams water treatment plant in 1985 to evaluate

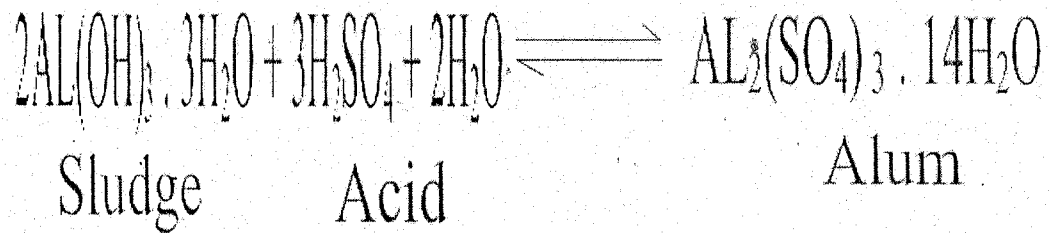
alum recovery. Two test were conducted, one in August and the other in September. The objectives were:

1. To evaluate alum recovery
2. To determine the dewater-ability of the solids remaining after alum recovery on sand-drying beds, and
3. To evaluate the effectiveness of the recovered alum as a coagulant in the water treatment plant.

Also, in the Allentown water treatment plant in Pennsylvania in the United States, attempts were made to recover alum through the acid digestion process in which the sludge is acidified with sulphuric acid. In addition, a simple to operate Donna membrane or Donna dialysis process was recently developed allowing selective alum recovery from water treatment residuals.

Aluminum sulphate is recovered for re-use through the acid digestion process or acidulation of the aluminum hydroxide in the sludge. In this process, the water treatment residuals (sludge) is sufficiently acidified with sulfuric acid that insoluble aluminum hydroxide is dissolved in the form of dilute liquid alum. The supernatant liquid, which is rich in dissolved aluminum, is subsequently decanted. This is done using settling tanks; the remaining waste is drained off and thereafter neutralized and dewatered.

The stoichiometry of this reaction can be written as follows for alum based water treatment residuals.



Although, the operation is simple, the process is non-selective; that is, along with alum, it recovers all other substances that are soluble under acidic conditions or that exists as colloids.

Naturally occurring organic materials, which are generally removed quite well by alum coagulation will be present in the recovered alum as dissolved organic matters. If the recovered alum is re-used as a coagulant, tri-halo-methane formation potential (THMFP) in the treated water upon chlorination would tend to increase significantly. The non-selective dissolution would mean that toxic metals are also dissolved in the decanted alum.

Furthermore, if an alkali digestion process is tried, given the atmospheric nature of aluminum oxide, the process will dissolve aluminum at a higher pH. However, the simultaneous dissolution of natural organic matters will still be an issue. More than seventy percent (70%) of the aluminum was recovered in twenty hours from the residuals of the Allenton plant.

Recovering alum using the Donna dialysis was a more promising alternative presented. The key features of the process are:

1. Recovered alum is essentially free of natural organic matters and particulate matters.
2. The concentration of aluminum in the recovered alum can be significantly greater than that in the water treatment residuals.
3. The process works on an electro-chemical potential gradient across a cat-ion exchange membrane, avoiding fouling of the membrane caused by natural organic matters or particulate matters.
4. The volume of disposable sludge is greatly reduced and sulfuric acid is the only chemical required for the process.

The recovered alum by Donna dialysis was transparent and clear, with practically no turbidity and naturally occurring organic matters; similar to fresh liquid alum.

These systems permit for recycling and recovered alum so that smaller amount of new alum is required. During the period of recycling, the acidulation may resolubilize precipitates formed during the water treatment process. The principal concern is likely to be iron and manganese. As recycling proceeds, they accumulate the recovered alum solution sufficiently to impair the water treatment or at least to become a potential problem. Other undesirable elements might also be present; this depends on the turbidity of the raw water. It is therefore advised that the recovered alum be eliminated at intervals depending on the composition of the raw water and other treatment conditions and new alum be used.

In summary, during the alum recovery cycle, sludge from the water treatment system containing aluminum-hydroxide is thickened, acidulated with sulfuric acid to produce alum from the hydroxide, and conditioned with an inert additive for facilitating filtering. The conditioned sludge is supplied under pressure to a filter press to produce cake from the solids therein and a filtrate containing alum. The alum in the filtrate is then returned to the water treatment system for re-use. Although a high percentage recovery of alum is possible, it is unlikely to be one hundred percent in practice and new make-up alum is added as required. Under unusual conditions, where the alum recovery cycle might threaten the quality of the product water, the end of the cycle is initiated.

#### **2.6.1 ECONOMIC EVALUATION OF RECOVERY**

Preliminary cost calculations indicate that the savings accrued from the re-use of recovered alum and lesser residual disposal costs will make the proposed process economically viable, particularly in large metropolitan areas where solid waste disposal is expensive and also a sensitive issue from a socio-political view point.



The present practice of the disposal of alum sludge into the nearest body of water result in the loss of a valuable asset (aluminum sulphate, alum) and at the same time pollutes the body of water. Thus, the dewatering and disposal of alum sludge add significantly to the cost of treating water. Alum recovery and re-use reduces those cost and the overall cost of water treatment.

## **2.7 ADAPTATION OF DESIGN IN TREATMENT PLANTS**

Evaluations have shown that alum recovery is feasible and can reduce both operations and disposal cost. The process is a viable method of reducing sludge-handling requirements. The recovered alum can be used at the water treatment plant and also at the waste water plants.

The chemical cost of the recovered alum is about half that of commercial alum, also adding to the attractiveness of the process. It is recommended that preliminary report to add to the already existing plant facilities should be done. Also, for any design of water treatment plant, due considerations should be given to include facilities that provide for the processes involved in alum recovery.

# CHAPTER THREE

## 3.0 EXPERIMENTAL PROCEDURE

Samples of sludge and alum solutions were collected from the Impresit water treatment plant, one of the three water treatment plants located in at the Chanchaga Water Works, Minna, Niger State. The samples were taken to the Quality control and monitoring laboratory, Federal Ministry of water resources, Minna, Niger State for analysis. The following analyses were carried out on the samples:

<b>Parameters</b>	<b>Materials</b>	<b>Manufacturers</b>
1) pH value	pH meter	Wagtech International
2) Turbidity	Turbidity meter	Wagtech International
3) Total suspended solids	Weighing balance	Mettler Toledo
4) Aluminum contents	Photometer	Wagtech International

## 3.1 EQUIPMENTS

<b>Equipments Used</b>	<b>Manufacturers</b>
1. Weighing balance	Mettler_Toledo Gmbtt At 200 Model
2. Sample bottles	Kartel Plastic, Made in England
3. Conical flask	Fisherband EU
4. pH meter	Wagtech International
5. Measuring cylinder	Pyrex USA
6. Pipette	Fisherband EU Class B
7. Turbidity meter	Wagtech International



8. Photometer

Wagtech International

9. Filter papers

Whitman International Limited,

Maiden, England

10. Oven

Gollenkemp Hat Oven, UK

11. Desiccator

### 3.1.1 REAGENTS USED

1. Distilled water
2. Concentrated tetraoxosulphate VI acid
3. Wagtech aluminum No. 1 tablets
4. Wagtech aluminum No. 2 tablets

### 3.2 PROCESS DESCRIPTION

Samples were collected and placed in sample bottles then they were taken to the Quality control and monitoring laboratory. The pH meter was calibrated using distilled water to a pH of 7. Then the pH of the samples collected was carried out. Then, the turbidity of the raw sludge was also carried out on the raw sludge. However, because of the concentration of the sludge, it was diluted 2.5ml of the raw sludge was placed in 100ml of distilled water. The Wagtech turbidity meter was used to take the readings.

The total suspended solid of the raw sludge was taken. This was determined by the following number of steps. They are

1. The filter paper to be used was placed in an oven for an hour then it was
2. The weight of the filter paper was weighed using a Metter Toledo weighing balance,

3. 100ml of raw sludge was filtered using the filter paper. It was left to drain for about twenty four hours,

4. The residue on the filter paper was placed in an oven to oven dry for one hour at a temperature of  $103^{\circ}\text{C}$ .

5. The the filter paper and residue was weighed. Then the TSS was determined by subtracting the initial weight of the filter paper.

250ml of the raw sludge was placed in four different conical flasks, which was acidulated with concentrated tetraoxosulphate VI acid of about 96% having a molecular weight of 98.08 and a molarity of 18 Molar. It was done with an endpoint in mind. This was added to the sludge until a pH of 2.0 was achieved.

The total suspended solid of the acidulated sludge was taken. This was done thirty minutes after the raw sludge was acidulated. This was done thus;

1. The filter paper to be used was placed in an oven to oven dry at a temperature of  $103^{\circ}\text{C}$  for 1 hour. The filter paper was placed in a desiccator and left to cool for a few minutes. This was done to remove any form of moisture on the filter paper.

2. A Metter Toledo weighing machine was used to weigh the filter paper before and after it was oven heated. This weighing machine is a digital machine, thus, it was zero before it was used.

3. 100ml was filtered, left to drain for one hour then oven dried.

4. The weight of the filter paper and the residue taken and difference between the initial weight of the filter paper and the present weight was taken to give the TSS.

The other three samples were labeled A, B, C were left to settle overnight. A volumetric split of the supernant and the residue was noticed. The result was then taken for the three samples.

One of the supernant was decanted from the settled sludge, and then the total suspended solids (TSS) were taken. Then, using one of the other supernants, the aluminum content was determined using the Wagtech photometer method. However, because of the concentration of the supernant, 0.2ml of the supernant was placed in 100ml of distilled water. In the Wagtech Aluminum method, the necessary reagents are incorporated into two test tablet. The test is simply carried out by adding one of each tablet to a sample of the diluted supernant.

The Wagtech aluminum No.1 tablet acidifies the sample to bring any colloidal aluminum into the solution and while the second tablet, which is Wagtech No. 2 tablet, buffers the solution to provide the correct conditions for the test. The test procedure is as follows:

1. The test tube was filled to the 10ml mark
2. One Aluminum No. 1 tablet was added to the test tube, crushed and mixed to dissolve.
3. One Aluminum No. 2 tablet was added, crushed and mixed gently to dissolve.
4. It was then allowed to stand for five minutes to in other to allow full colour development.
5. A wavelength of 570nm was selected on the photometer.
6. Then the photometer reading was recorded.

# CHAPTER FOUR

## 4.0 RESULTS AND DISCUSSION OF RESULTS

### 4.1 EXPERIMENTAL RESULTS

The results of the tests and analysis carried out on the samples collected are shown below.

These samples were the sludge and the alum solution used at the treatment plant.

The pH of the raw sludge	5.82
Turbidity of the raw sludge	34,040 NTU
The pH of the alum solution	2.88
Total suspended solid of the raw sludge	40.9441g
Molecular weight of the acid used	98.08
Molarity of the acid	18.0Molar
Total suspended solid of the sludge after thirty minutes of acidulation	24.7272g

Volume of the supernant after the acidulated sludge was left to settle overnight is shown in the table below.

**Table 4.1 TOTAL SUSPENDED SOLIDS**

	Raw sludge	Acidulated sludge after 30 minutes	Acidulated sludge after 24 hours
Weight of filter paper(g)	1.5459	<b>1.5600</b>	1.4103
Weight of residue and filter paper(g)	42.4900	26.2200	1.5668
Weight of residue(TSS) (g)	40.9441	24.7272	0.1565

Source; Author's Result of analysis



**Table 4.2 VOLUME OF THE SUPERNANT**

Sample	A	B	C
Volume of the sludge(ml)	250	250	250
Volume of the acid added(ml)	2.7	2.6	2.9
Volume (ml)	150	155	153

Source; Author's Result of analysis

**Table 4.3 ALUMINUM CONTENT**

SAMPLE	ALUMINUM CONTENT
Alum solution	0.466mg/L
Treated unfiltered sludge (supernant)	0.357mg/L
Treated filtered sludge (supernant)	0.379mg/L

Source; Author's Result of analysis

### **DISCUSSION OF THE RESULTS**

The pH of the alum solution was 2.88 while that of the raw sludge was 5.82. This indicates that the pH dropped after the treatment of the raw water with the alum solution. The equation for the reaction is seen in appendix A. The total suspended solids of the treated sludge when compared with the raw sludge also reduced after it was acidulated with tetraoxosulphate VI acid about thirty minutes later. This indicates that the insoluble aluminum oxide dissolved in the presence of the acid. This is seen in appendix B. From the results, it was discovered that the total

suspended solids reduced by 40%. This was achieved by adding tetraoxosulphate VI acid to the raw sludge till a pH of 2 was achieved.

When the setup was left to settle overnight, a volumetric split was discovered from the three setups A, B, C. An average of 152.67ml was recovered from the setup. This shows that about sixty percent of the alum solution was recovered from the sludge.

The aluminum content was determined to know the concentration of the recovered alum solution. This was then compared with that of the alum content of the alum solution used for coagulation which gave 0.466mg/L. The value of the aluminum content of the supernatant was taken before it was filtered, and it gave 0.357mg/L. After it was filtered, it gave 0.379mg/L. This shows that the concentration of the solution recovered was about eighty-five percent (85%) of the alum solution used. To achieve the concentration needed for the treatment of raw water, more alum could be added to the solution to make up to the concentration needed for adequate coagulation to take place.

This implies that less amount of alum is required if the sludge is recycled and less contamination of the water bodies. From the analysis, less alum would be needed and thus, it would in the long run for the coagulation/clarification process.



## CHAPTER FIVE

### 5.0 CONCLUSION AND RECOMMENDATION

#### 5.1 CONCLUSION

This research project was undertaken to recover alum from sludge and also to evaluate the effectiveness of the recovered alum solution.

This was done by acid extraction method, where the sludge is treated with tetraoxosulphate VI acid. This process is a viable method of sludge-handling requirements. The recovered alum can be used at the water treatment plant. However, for efficiency, the concentration should be increased by adding more solid alum to the solution. The chemical cost of the recovered alum is more than half the amount that is used for the treatment of raw water.

#### 5.2 RECOMMENDATION

From the history of water treatment, little or no attention was given to the disposal of sludge.

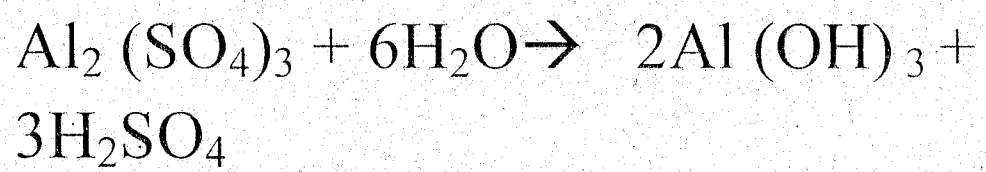
Several methods have been tested in the developed countries. This method of alum recovery from the sludge is one that would not only reduce pollution but would also reduce the cost of production of portable water.

It is therefore recommended that more analysis be carried out in this area. These analyses should include the cost benefits and also determine if the process is feasible economically. Then water treatment plants could be redesigned and a unit that serves as the recovery unit could be introduced into the plant. From time to time, the concentration of the alum solution recovered should be checked to determine its efficiency. When the need arises, the recovered alum could be disposed off if it is not fit to be used, as a result of contamination by undesired materials.

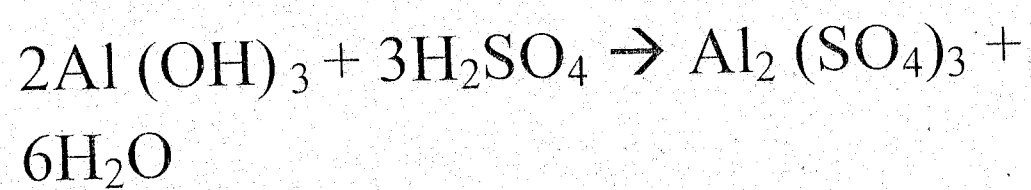
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## Appendix A



## Appendix B



## Appendix C

### UNITS

NTU – Nephelometric turbidity unit

mg/L – Milligrammes per litre

ml – Milliliters

nm – Nanometer

g – Grammes



## Appendix D

Turbidity meter reading = 851 NTU

2.5ml of raw sludge was placed in 100ml of distilled water

Conversion factor =  $\frac{100}{2.5} = 40$

2.5

Therefore the actual turbidity reading gives

$(851 * 40) \text{NTU}$

$= 34,040 \text{NTU.}$



## APPENDIX E

To calculate the aluminum content for the solutions

0.2 ml of each was diluted in 100ml of distilled water. Therefore the conversion factor is  $100\text{ml}/0.2\text{ml} = 500$

1. For alum solution 233mg/L was the photometer reading. Therefore the actual reading will be equal to  $233/500 = 0.466\text{mg/L}$ .
2. For treated unfiltered sludge 178.5mg/L was the photometer reading. Therefore the actual reading will be equal to  $178.5/500 = 0.357\text{mg/L}$ .
3. For treated filtered sludge 189.5mg/L was the photometer reading. Therefore the actual reading will be equal to  $189.5/500 = 0.379\text{mg/L}$ .