Kwame Nkrumah University of Science and Technology, Kumasi-Ghana

Production of Alum from Bauxite waste from Awaso

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Production of Alum from Bauxite Waste from Awaso mine

By

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CERTIFICATION

I hereby declare that this submission is my own work towards the MSc. and that, to the best of my knowledge it contains no material previously published by another person or material which has been accepted for the award of any other degree of the University, except where due acknowledgement has been made in the text.

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(Principal Supervisor) Signature Date

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(Head of Dept., Civil Engineering) Signature Date

DEDICATION

This thesis is dedicated to God, my family, Etuaful Academy and friends

ABSTRACT

Turbidity and colour has been one of the major challenges facing most Water treatment plants in Ghana. The effect of high levels of turbidity is the increase in the cost of production. The most efficient way of removing and reducing turbidity and colour is by the application of coagulants, which is mainly alum in Africa. Several methods have been established for the production of alum; the most efficient one has been determined to be the use of an acid to digest the bauxite waste. This method has been chosen to be the most efficient because it is simple and cost effective.

It has been proven that a significant percentage of budgetary allocation for the treatment of water mostly in Africa and developing countries goes into the procurement of alum.

The focus of this study is to determine a protocol that can be effective, efficient and cost less in the production of alum from bauxite waste. The environmental friendliness of the protocol developed was also very important and vital to the work and duly investigated. One of the most important variables also investigated was the molecular characteristic of the alum produced using an X ray diffractometer.

This study developed a protocol, which leads to the production of alum from bauxite waste; the most effective protocol was the use of 165ml of sulphuric acid as the acid for digesting the bauxite waste, which was done strongly under refluxing conditions.

The alum produced from the bauxite waste performed comparably well to the commercial Alum. It was also realized that for every 20g of bauxite used to produce alum using the white sample protocol about 1159g of alum is produced at a cost of 35Ghana cedis, which is, cheaper than the commercially available alum at a current market price of 80 Ghana cedis.

The disposal of the sludge produced after filtration was highly acidic so the introduction of potash into the sludge before disposal was used as the means of reducing the acidic rate of the sludge.

Using the X ray diffractometer and Jar test it was established that the alum produced from the lab performed comparably well as against the commercial alum. In the residual aluminium and sulphate test, the alum produced performed better than the commercial alum.

Table of Contents

LIST OF TABLES

LIST OF FIGURES

LIST OF PLATES

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1. INTRODUCTION

1.1 Background

Aluminum sulfate ($Al_2(SO_4)_3$), commonly called alum, is produced as white crystals which are non-combustible and soluble in water. This dry hydrate ($Al_2(SO_4)_3.14H_2O$) is 17% Al_2O_3 and is also sold as a 47% w/w aluminum sulfate solution which is 8% Al_2O_3. It is also sold in solid form as granules or dust. (Romano et al, 2001)

Alum is one of the major chemical components for the treatment of water to help reduce suspended solid particles of the raw water. It is also used in the paper and pulp industry to control pitch, prevent foam, and improve the properties of broken and secondary fibers. On the Weija treatment plants/ headworks about 30% of the treatment cost goes into the purchasing of Alum. This clearly depicts that alum produced under reliable and cheap technologies can help reduce the unit cost of production. (Akabzaa, 2004):

Alum (as aluminum sulfate is commonly called) is manufactured in a simple two-step process from aluminiumtrihydrate and sulfuric acid according to the following reaction:

$2Al(OH)_3 + 3H_2SO_4 + 8H_2O \rightarrow Al_2(SO_4)_3 .14H_2O$ ΔfH= -156 kJ mol-1. ………….(1)

In the protocol for the production of the Alum, the sludge produced is extremely acidic and when discharged untreated affects aquatic life. (Barrios-Perez J, 2003)

Potassium aluminum sulfate is a chemical which conforms to the general formula $KAl(SO_4)_2$. Also known as aluminum potassium sulfate, its unique characteristics have made it an important compound to many industries example water, paper and pulp, pharmaceutics etc

The commercial production of potassium aluminum sulfate is typically accomplished by a method called hydrometallurgy. In this process, an aqueous solution of sulfuric acid is first used to extract alumina (solid Al_2O_3) from an ore called bauxite. This step, known as leaching, results in a solution which can then be reacted with potassium sulfate to form potassium aluminum sulfate. (Romano et al, 2001)

Potassium aluminum sulfate forms a solid, white powder at room temperature. It is a hygroscopic material. Depending on the amount of water molecules present, these hydrates are represented by the chemical formulae $KAl(SO_4)_2 \cdot 12H_2O$ or $K_2SO_4.Al_2(SO_4)_3 \cdot 24H_2O$. The powder form, made up of crystals, has a melting point of 198.5°F (92.5°C) and can be readily dissolved in water. Additionally, this material has a property known as astringency, which is an ability to constrict body tissues, and restrict the flow of blood. (Cornwell et al, 1979)

There have been many industrial applications of potassium aluminum sulfate. It is an important part of many products created by the pharmaceutical, cosmetic, and food industries because of its astringency property. It is also used in the manufacture of paper, dyes, glue, and explosives. Additionally, it used in the water purification process, in speeding up the hardening of concrete and plaster, and acts as a catalyst in various chemical reactions. (Cornwell et al, 1979)

1.2 Production Of Bauxite Waste At Awaso

In Ghana, bauxite is mainly mined in the Western Region at Awaso (Sefwi). Mining of bauxite has been done in that part of the region for over sixty years and production is planned to be increased as a result of which the washing plant will generate more slimes estimated to be over 100,000 t a year (Acquah et al, 1999). Acquah et al. (1999) have reported their investigation on the use of the Awaso bauxite for alum production. During mining operations at the Awaso, material from the pits is usually passed through a washing plant to remove fines and impurities such as plant material and adhering clay. The waste from the washing plant (slime material) from the washing plant is considered as having being impounded in slime ponds, over the past sixty years and this has led to the stockpiling of several thousand tonnes of slime. (Acquah et al, 1999).

The main mineral phases in the slime materials are similar to the parent bauxite material. These include gibbsite and kaolinite, as well as impurities such as iron oxide (haematite and goethite), quartz and rutile. The research by Acquah et al (1999) evaluated the characteristics of the slime waste and investigated its potential for the production of commercial aluminium sulphate (alum). They went on further to investigate how the major impurities (iron and chromium) in the leach solution could be reduced. (Acquah et al, 1999)

1.3 Problem Statement

Alum is an essential component of water treatment in Africa but the cost of procuring is affecting the cost of treating our raw water treatment. Ghana Urban Water limited spent about Ë 6,561,902 in treatment of raw water in the year 2009 out of a total amount of Ë 9,322,519 used on water treatment chemicals, which is about 70.38% of the total sum. Even with this amount of money spent just about 63.34% of the nations urban and pre urban areas have access to potable drinking water treated by GUWL. With this, about 36.66% don't have access to potable drinking, meaning more money would be spent in procuring Alum to treat water in other to attain the millennium development goal (MDG) of achieving 76% coverage or access of potable water by 2015. The increase in the population would also mean an increase in water demand thereby increase treatment cost.

The various treatment works in the country use on the average 700 bags of alum per day, which is almost 3500kg of alum. An average of 1million tonnes of bauxite waste has been produced at the Awaso mine within a duration less than a decade; with such a production line disposal of the bauxite waste is a serious environmental challenge, if the alum is produced using the bauxite waste, it would help mitigate the challenge moreover the cost of procurement of Alum would be drastically reduced. The existing methods of producing Alum is cumbersome and expensive due to the cost of sulphuric acid thereby not allowing locals to invest and produce alum locally. The existing protocol is extremely not user friendly because acquisition and handling of sulphuric acid requires skilled labour.

1.4 Justification

Even though the research was conducted in Ghana, the technology can be adopted universally to enhance the production of alum. The technology or protocol that comes out of the research if implemented can help reduce the cost of producing alum and that of the treatment of water.

It would inform and influence local investors to commence the local production of alum since the raw material is in abundance in the country.

The local production of alum in the country would help influence the management of Ghana Water Cooperation limited (GWCL) to stop importing alum but buy it locally.

The protocol would help curb the environmental challenge, which is faced by the Awaso bauxite mine if the protocol for the production of alum from bauxite waste is developed and used.

1.5 Objectives

The main objective of the study is to produce Alum from bauxite waste from Awaso bauxite mine

The Specific Objectives are;

The Specific Objectives of the study are:

1. To develop an efficient protocol for the production alum from bauxite waste generated at the Awaso bauxite mine.
2. To determine the molecular characteristics of the produced Alum using X Ray diffractometer.
3. To determine the environmental effect or impacts of the protocol.
4. To determine the cost of producing alum from the ore.

1.6 Scope of Work

The Alum being produced in this study would be applying the waste generated at the Awaso bauxite mine at Awaso specifically from the slime material. The protocol used focused on the application of sulphuric acid under refluxing conditions.

2. LITERATURE REVIEW

2.1 Bauxite occurrence in Ghana

The presence of bauxite in southern Ghana was recognized in the earliest days of the Geological Survey of Ghana carried out a reconnaissance traverses across the regions and noted deposits of bauxite on the lower slopes of the Atewa range and along the base of the Kwahu Escarpment. Subsequent work by the Geological Survey over a period of fifteen decades identified numerous bauxite occurrences capping many of the highest hills in southern Ghana. (EAPL, 2004)

At the beginning of world war II. Great Britain was in dire need of secure sources of bauxite to produce aluminum. This led to the rapid development of the deposits in the Sefwi Bekwai area (Awaso) of the South Western Ghana. The selection of Awaso for development had much to do with its favorable and convenient location because of ease access to means of transport and how inhabitable the location was. By 1943, the Awaso open pit operations were fully geared up and shipping more than 100,000 tonnes of ore per year by rail to the Port of Takoradi and the operation of mining and exporting still continues to this day. (Schellmen, 1983)

2.2 Geology of bauxite

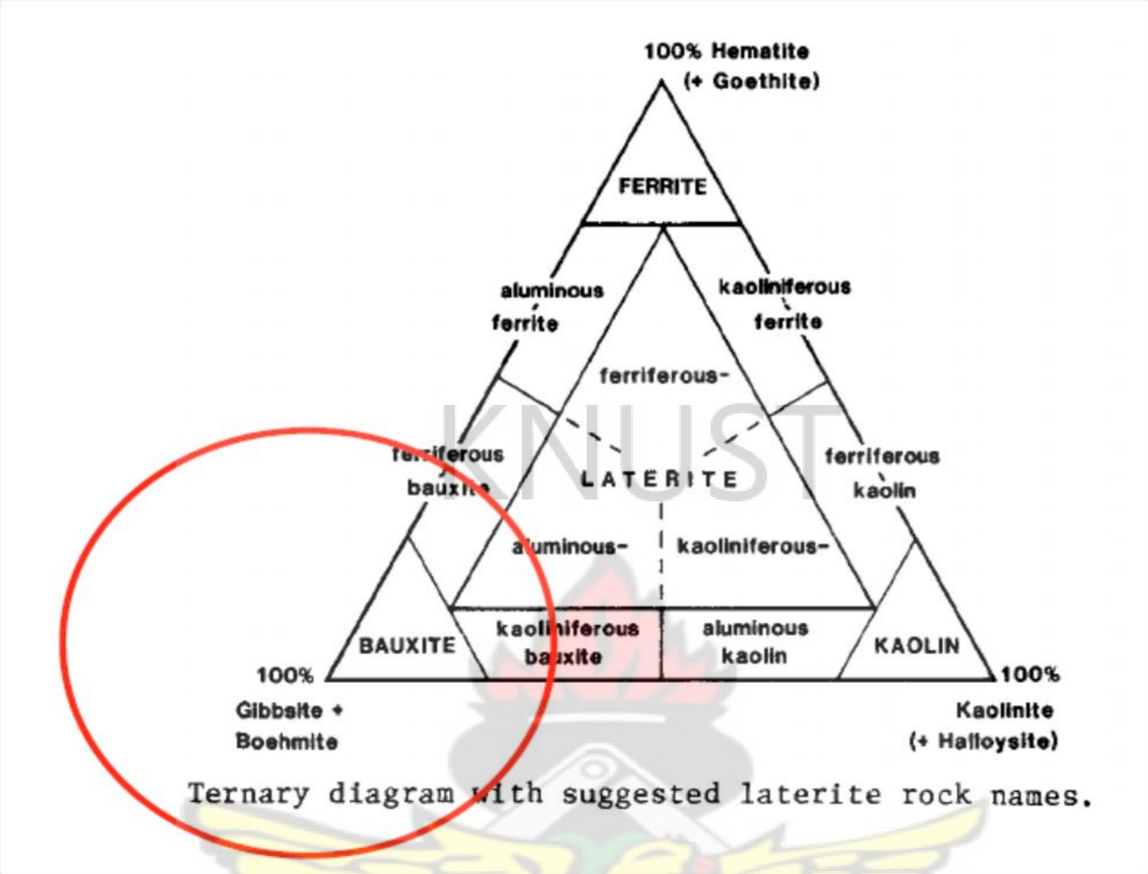
2.2.1 Composition of bauxite

Mineralogically, bauxite consists chiefly of hydrated aluminum oxide, namely, gibbsite, boehmite, diaspore with impurities of iron hydroxide minerals and titanium oxide. Based on the predominant mineralogy, bauxite may be classified into gibbsitic type, mixed gibbsitic boehmite type, boehmite type or diasporic type. Silica in bauxite is mainly found in the form of kaolinite and quartz. The other mineral impurities present in bauxite are hematite, goethite, anatase, rutile. The gangue minerals present in some bauxite are chamosite, pyrite, siderite, ilmenite, sphene, calcite and dolomite. (Schellman, 1983)

The major chemical constituents i.e. Al_2O_3, Fe_2O_3, SiO_2, TiO_2 together with "Loss on Ignition" (LOI) constitute more than 99% bauxite composition. The rest is contributed by FeO, CaO, MnO, MgO, K_2O, Na_2O and P_2O_5. The SiO_2 is present mainly in form of kaolinite and quartz respectively known as 'reactive' and 'free' silica. The bauxite, in general, has 40-50% Al_2O_3, 4-28% Fe_2O_3, less than 1-5% SiO2, 2-11% TiO_2 and LOI 18-30%. The titania content in the bauxite has a bearing on the source rock. The bauxite over basaltic rock have higher titania compared to the bauxite derived from khondalite- charnockite. (Schellman, 1983)

In extraction metallurgy of aluminium the mineralogy of bauxite plays a very significant role – boehmite content in bauxite would render the digestion process more difficult as compared to one with higher trihydrate aluminium oxide or gibbsite form. Other mineral impurities of iron, titanium and silica only increase the waste thus reducing the productivity and increasing the cost of production. In Bayer's process of metallurgy bauxite with higher 'reactive' silica increases the loss of alumina and caustic soda. The gibbsitic bauxites are best suited for metallurgy of aluminium whereas the boehmitic and diasporic varieties are suitable for refractory, abrasive and chemical industries. (Schellman, 1983)

In a bauxite profile, the alumina content generally increases gradually downwards from the top of the bauxite horizon, reaches its maximum in the middle part and decrease towards the base. A large number of lateritic bauxite contains 4-8% total SiO_2. There is generally a pronounced increase of the reactive silica at the bottom of the bauxite horizon. The majority of the laterite bauxite deposits contain 10-28% Fe_2O_3. The variability of iron vis-à-vis other major chemical components is very pronounced in bauxite-laterite profile. This is best illustrated by highly ferruginous dark brown coloured duricrust with 50-65% Fe_2O_3 occurring over white bauxite containing less than 3-5% Fe_2O_3. Commonly titania in a bauxite ranges from 1-4%. The highest range of TiO_2 content (8-10%) is reported from the basalt derived bauxite of M.P. and Maharashtra. Average "Loss on Ignition" (LOI) of the bauxite is highest 28-31% in the high alumina gibbsitic bauxite (trihydrate alumina bauxite). The lowest average loss on ignition (15-19%) is found in the deposits dominantly composed of monohydrate aluminium oxide-boehmite and diaspore. (Schellman, 1983)



Source: Schellman (1983)

Figure 2-1 Geology of Bauxite

Table 2-1 Chemical Composition of Bauxite from Mt. Supirti, Setwi Bekwai.

COMPOSITION	%SAMPLE A	%SAMPLE B
SiO_2	0.64	0.40
$A1_2O_3$	60.68	61.08
Fe_2O_3	4.2	4.1
TiO_2	1.11	0.87
H_20	32.17	32.23
CaO	0.33	0.25
V_2O_3	0.04	0.02

Source: Acquah et al. (1999)

Acquah et al. (1999) sampled from the Awaso mine and proposed such characterization of the bauxite as shown in the table above. ***Acquah et al. (1999)*** proposed that the bauxite contained an average of 0.64% of $SiO_{2,\ 60.68}$% of $A1_2O_3$, 32.17% of water and the others were contributed by $Fe_2O_{3,}$ $TiO_{2,}$ CaO, $V_2O_{3.}$ This proposal was found not to be the case with our sampling since each sample had different composition, which has no exact link to the proposal by ***(Acquah et al. 1999).***

2.3 Uses of bauxite

2.3.1 Bauxite Used for Aluminum Production

Bauxite is the principal ore of aluminum. The first step in producing aluminum is to crush the bauxite and purify it using the Bayer Process. In the Bayer Process the bauxite is washed in a hot solution of sodium hydroxide, which leaches aluminum from the bauxite. The aluminum is precipitated out of solution in the form of aluminum hydroxide, $Al(OH)_3$. The aluminum hydroxide is then calcined to form alumina, Al_2O_3. (Romano et al, 2001)

Aluminum is smelted from the alumina using the Hall-Heroult Process. In the Hall-Heroult Process the alumina is dissolved in a molten bath of cryolite (Na_3AlF_6). Molten aluminum is removed from the solution by electrolysis. This process uses an enormous amount of electricity. Aluminum is usually produced where electricity costs are very low. (Romano et al, 2001)

2.3.2 Use of Bauxite as an Abrasive

Calcined alumina is a synthetic corundum, which is a very hard material. Calcined alumina is crushed, separated by size and used as an abrasive. Aluminum oxide sandpaper, polishing powders and polishing suspensions are made from calcined alumina.

Sintered bauxite is often used as a sandblasting abrasive. It is produced by crushing bauxite to powder and then fused into spherical beads at very high temperature (800 ^{o}C - 1100 ^{o}C). These beads are very hard and very durable. The beads are then sorted by size for use in different types of sandblasting equipment and for different sandblasting applications. (Hobart, 2006)

2.3.3 Use of Bauxite as a Proppant

Sintered bauxite is also used as an oil field proppant. In drilling for oil and natural gas the reservoir rock is often fractured by pumping fluids into the well under very high pressures. The pressure builds up to very high levels that cause the reservoir rock to fracture. When fracturing occurs water and suspended particles, known as "proppants" rush into the fractures and push them open. When the pumps are turned off, the fractures close trapping the proppant particles in the reservoir. If an adequate number of crush-resistant particles remain in the reservoir the fractures will be "propped" open allowing for a flow of oil or natural gas out of the rocks and into the well. This process is known as hydraulic fracturing. (Hobart, 2006)

2.4 Bauxite production

More than 100 million tonnes of bauxite are mined each year across the world. The major locations of deposits are found in a wide belt around the equator. Bauxite is currently being mined in Australia (in excess of 40 million tonnes per year), Central and South America (Jamaica, Brazil, Surinam, Venezuela, Guyana), Africa (Guinea), Asia (India, China), Russia, Kazakhstan and Europe (Greece). (Schellman,1983)

2.5 Mining of bauxite

Bauxites are natural earthy mineral, which is mostly rocky, and about 100million tonnes are produced yearly across the world. The mostly known method of mining is through blasting of the mineral to break down the rocks, others also mining this mineral through the digging of the bauxite mineral.

In Ghana bauxite is mainly mined or extracted at Awaso and the method used there is blasting of the rocks after which the rocks are crushed. (Barrios- Perez, 2003)

2.6 Environmental effects of bauxite

Aluminium being the third most abundant element in the earth's crust, is an obvious fact that the availability of the raw material used for the production would also be in abundance or almost unlimited.

The raw material for the production of aluminium is mainly extracted by open-cast mining, which has a variable and highly site-specific effect on the local environment. The primary ecological concerns connected to this operation are related to the clearing of vegetation, effect on local flora and fauna, and soil erosion. (EAPL, 2004)

One of the major concerns of environmentalist after mining of any mineral is restoring the vegetation of the land. Currently just about 6 % of the world's bauxite mines is done in a rain forest region, affecting a total area around 1.5km^2. (European Aluminium Association).

2.7 Physical properties of alum

Table 2-2 Physical properties of alum

PROPERTY	
Physical State	White crystals/ powder/ granular
Melting Point	92 – 93 C
Boiling Point	200 C (Decomposes)
Specific Gravity	1.757
Solubility	Extremely Soluble
pH	3 – 3.5 (Decomposes)
Vapour Density	16.4
Particle Size	1 – 2mm
Vapour Density	16.4

Source: EPA, (2005)

2.8 Chemical properties of alum

Table 2-3 Chemical Properties of Alum

PROPERTY	
Fe	0.02% max
Pb	0.02% max
Water Insolubles	0.01% max

Source:EPA, (2005)

The chemical and physical property of commercial alum as shown in the table above is the international accepted standards and therefore the alum to be produced should meet the internationally accepted standards.

2.9 Health and environmental impacts of bauxite

The main environmental problems associated with bauxite mining are related to the revegetation of mined-out areas and the disposal of tailings. Emissions of dust consisting of clay and bauxite particles from dryers' chimneys can also pose problems, (EPA, 2001). Earth trembling and tremors causes cracks in their neighboring buildings being occupied by inhabitants.

2.10 Production of alum

The production of Alum involves the Extraction of Alumina (digestion), Production of Alum (filtration, addition of potassium sulphate) and Crystallization.

2.10.1 Extraction of alumina from bauxite waste

2.10.1.1 Digestion

Digestion is the process of treating a substance (raw material) by means of heat, enzymes, or a solvent to promote decomposition or extraction essential components. In most cases the solvent used to extract the essential mineral or component is allowed a minimum of 3hours contact time with constant heating. Digestion involves leaching process and/or enzymatic decomposition. Digestion that doesn't involve enzymes, strong acid is used for the extraction. (Romano et al, 2001)

2.10.1.2 Sulphuric acid

Sulphuric acid is the main chemical needed for the production of alum. High concentrations (above 2N) of sulphuric acids are extremely hygroscopic, making the produced alum from the bauxite extremely hygroscopic. In some other methods of production, digestion is done in the open allowing water to be lost, after which water is added to reduce the concentration of sulphuric acid after digestion (Momade, Sraku-Lartey, 2010).

2.10.2 Production of the alum

Aluminum sulfate ($Al_2(SO_4)_3$), commonly called alum, is produced as white crystals which are non-combustible and soluble in water. This dry hydrate ($Al_2(SO_4)_3.14H_2O$) is 17% Al_2O_3, alum can also be obtained as 47% w/w aluminum sulfate solution which contains 8% Al_2O_3. It can also be obtained in solid form as granules or dust. (Romano et al, 2001)

In the preparation of the alum from the slime waste the digestion method was used which is one of the easiest and fastest way of preparing alum from slime waste.

2.10.2.1 Crystallization

This is the process at which liquids are forced to form crystals. The process of crystallization is such that a slightly hot liquid is instantly cooled which aids crystallization to occur quickly. (Oxford dictionary, 6th Edition)

2.10.3 Test for the potency of the alum produced

The alum produced can only be termed and called alum when certain confirmatory test has been conducted on it to ascertain if really the product produced is alum but not any chemical. Some of the test conducted was

1. Passing the sample through an X ray diffractometer.
2. Conducting coagulation test using the Jar test Equipment
3. Thermo gravimetric Analysis
4. Energy dispersive X Ray test

2.10.3.1 X ray diffractometer

It's a measuring instrument used in analyzing the structure of a material from scattering patterns produced when a beam of radiation or particles such x rays interact with it. An x ray diffractomer just like any other diffractometer consists of a radiation source, a monochromator to help choose the wavelength, slits to help adjust the shape of the beam, a detector and last a very dry grinded sample. XRD helps in the determination of the compounds that exist in samples, which helps to know the exact components of the substance, or samples being analyzed. (Arai et al, 2001)

Plate 1 X Ray Diffractometer

2.10.3.2 Jar test

Jar test uses or applies the principle of coagulation/flocculation; coagulation/flocculation is the process of binding small particles in the water together into larger, heavier clumps which settle out relatively quickly at a faster rate. The larger particles are known as floc. Properly formed floc will settle out of water quickly in the sedimentation basin, removing the majority of the water's turbidity. Different dosages of coagulants are tested using a jar test, which mimics the conditions found in the treatment plant. The first step of the jar test involves adding coagulant (in this case Alum) to the source water and mixing the water rapidly (as it would be mixed in the flash mix chamber) to completely dissolve the coagulant or mix uniformly in the water. Then the water is mixed more slowly for a longer time period, mimicking the flocculation basin conditions and allowing the forming floc particles to cluster together. Finally, the mixer is stopped and the floc is allowed to settle out, as it would in the sedimentation basin. This method is used to measure the turbidity, pH, conductivity and Total Dissolved Solids. (Tchobanoglous *et al,* 1985)

Plate 2 Jar Test Apparatus

2.10.3.2.1 Turbidity

In a raw water to be used as drinking water undergoes certain treatment procedures one of which is coagulation. Coagulation is the removal of colloids particles from the raw water. The removal of colloidal particles enhances the turbidity. Turbidity in other words can be termed as how clear water is. It’s also the measure of the extent to which light is either absorbed or scattered by suspended material in the raw water. Turbidity is not a direct measure of suspended solids. When water is turbid its mostly aesthetically unpleasant. Some colloidal particles adsorbs certain chemicals that might be harmful to human health. The most common ways of causing natural water bodies to be highly turbid is erosion of colloidal material into those water bodies. The World Health Organization considers water with 5 NTU as generally potable. Turbidity when higher than the WHO standard makes the water unwholesome. (Naeem and Malmberg, 1985)

2.10.3.2.2 Total Dissolved Solids

TDS is the remaining material in the treated water after filtration. Dissolved solids can be both organic and inorganic in nature. Water with a TDS of less than 600mg/litre is generally considered to be good; drinking water becomes significantly and increasingly unpalatable at TDS levels greater than about 1000mg/l. the presence of high levels of TDS may also be objectionable to consumers, owing to excessive scaling in piped water, heaters, boilers and household appliances. No health based guideline value for TDS has been proposed (WHO, 2006). Dissolved solids when exceedingly high have a lot of adverse (Naeem and Malmberg, 1985).

2.10.3.2.3 pH

This is the numerical expression of the hydrogen ion concentration indicating the degree to which water is acidic or alkaline. These various degrees are represented on a scale of 0 to 14, with 0 being the most acidic, 14 most alkaline and 7 neutral, the scale mostly called pH scale. (Tchobanoglous, et al 1985)

Acidic water has very harmful effect on the production and distribution so as it has on the human system. Low pH (acidic) levels are highly unacceptable because of the corrosive effects on metal pipes and fixtures, causing leaching of lead from solder in pipes in older homes, lead from brass plumbing fixtures and the metallic taste that is often associated with low pH water. High pH levels are undesirable since they may impart a bitter taste to the water. Additionally, dissolved iron in low pH water stains porcelain plumbing fixtures. Furthermore, the high degree of mineralization associated with the alkaline waters will result in the encrustation of water pipes and water using appliances. The combination of high alkalinity and calcium with low pH levels may be less corrosive than the water with combination of high pH, low alkalinity and calcium content. High pH levels also depress the effectiveness of disinfection by chlorination, thereby requiring the use of additional chlorine or longer contact times. A range of 6.5-8.5 was determined by WHO as that pH which would achieve the maximum environmental and aesthetic benefits, which would also make drinking water very potable. (Naeem and Malmberg, 1985)

2.10.3.2.4 Settling Rate

This is the rate floc formed after coagulation settles to enhance the turbidity and colour of the raw water. The settling rate is influence by the nature of flocs formed, no disturbance to the raw water, and also the nature of the coagulant. The settling rate depicts the time to which the tiniest floc or particle would settle to enable filtration. The shorter the settling rate the better the coagulant since it would take very little time for the flocs to settle therefore making the work of the filters easier. The rate of settling also helps in the choice of the concentration of alum to use and at what time it would be best to start filtration. (Chen et al, 1976)

2.10.3.2.5 Residual Aluminum and Sulphate

2.10.3.2.5.1 Sulphate

High concentrations of sulphates in drinking water have three effects: tend to form hard scales in boilers and heat exchangers, cause taste effects and lastly cause laxative effects with excessive intake. Sulphates can also induce diarrhea at levels greater than 500mg/l but typically near 750mg/l. While Sulphate imparts a slightly milder taste to drinking water than chloride, no significant taste effects are detected below 300mg/l. it cannot readily be removed from drinking water, except by distillation, reverse osmosis or electro dialysis, but these are expensive methods. High levels of sulfates in water also irritate the mucous membranes of the respiratory tract and promote the development of chronic respiratory diseases. (EAPL, 2005)

2.10.3.2.5.2 Aluminium

Aluminium happens to be the major component of Alum therefore it is expected that the residual aluminium percent in the treated water would be high but high residual aluminium is one of the things that has serious health implication. High levels of aluminium cause serious non-curable disease like Alzheimer, which affects the brain of people. In view of this the

WHO has as its standard 20 – 50 microgram per liter been the amount of aluminium that can and must be left as residue. (EAPL, 2005)

2.10.3.3 Thermogravimetric Analysis

Thermogravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). (Coats and Redfern, 1963). TGA can provide information about physical phenomena, such as second-order phase transitions, including vaporization, sublimation, absorption, adsorption, and desorption. Likewise, TGA can provide information about chemical phenomena including chemisorptions, desolvation (especially dehydration), decomposition, and solid-gas reactions (e.g., oxidation or reduction).

TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles (such as moisture). (Coats and Redfern, 1963)

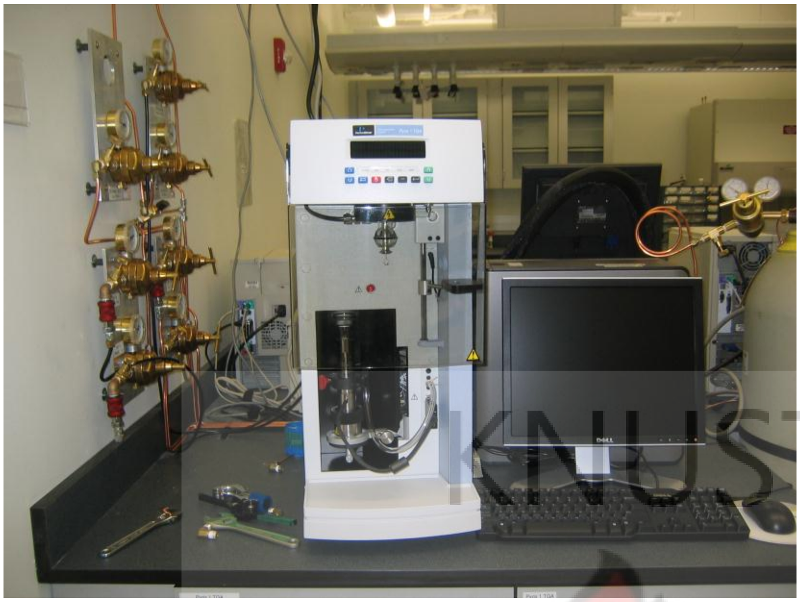


Plate 3 Thermogravimetric Apparatus

2.10.3.4 Energy Dispersive X Ray Test

Energy-dispersive X-ray spectroscopy is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons, or a beam of X-rays, is focused into the sample being studied. (Goldstein, 2003)

Plate 4 Energy Dispersive X Ray Apparatus

3 RESEARCH METHODOLOGY

The following methods were used to achieve the objectives of the study.

This chapter covers the procedures followed to obtain the desired laboratory results. The chemicals as well as the apparatus required in performing each of these experiment are mentioned in this chapter. All chemicals used throughout this study were of analytical grade.

3.1 Laboratory Experiment

The laboratory experiment included the production of alum, characterization and analysis of the Alum, tests conducted to establish the efficiency of the alum, preparation of model water.

3.1.1 Production of Alum

To achieve the main objective of this research work, bench top experiment was conducted.

3.1.1.1 Apparatus

- Magnetic Stirrer
- Measuring cylinder
- Measuring scale
- Volumetric Flask
- 1L Beaker
- Bunsen burner
- Furnace
- Filter paper

3.1.1.2 Reagents

Potassium sulfate

Sulphuric acid

DI water

3.1.1.3 Procedure For The Production of Alum

20g of bauxite waste were calcined at 600 °C for an hour using a furnace. The normality of the sulphuric acid used was calculated with varying volumes using the equation below:

$$\textbf{Vol} = \frac{100 * \text{molarity} * \textbf{Normality}}{\text{basicity} * \%\text{purity} * \text{density}}$$

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

Varying volumes of acid used was diluted in 1L volumetric flask. The acid was then mixed with the calcined bauxite waste and heated till it boils for 4 hours (digestion). Refluxing was used for the digestion process of production of Alum to avoid the loss of water during heating. The solution was allowed to stand for 20mins and later filtered with a grade A filter. The filtrate was again heated for 30mins and 176g of potassium Sulphate was added to the clear filtrate and cooled on ice instantly to aide the crystallization process.

Plate 5 Bauxite waste in a furnace

Plate 6 Bauxite waste in a refluxing set up before digestion

Plate 7 Bauxite waste in a refluxing set up after for 4hrs of digestion

Plate 8 Waste left on the filter paper before and after filteration

3.1.2 Characterization and Analysis of the Produced Alum

In the characterization of the produced Alum, x ray diffractometer was used to determine the percentage of the various elements in the sample.

3.1.3 Tests Conducted To Establish The Efficiency Of The Alum

The test was performed to ascertain the efficiency of the alum produced and how reliable the protocol developed was. The test conducted included coagulating property of the Alum using a jar test apparatus, effect on pH, particle size, colour, residual Aluminium and sulphate.

3.1.3.1 Jar Test

Jar test uses or applies the principle of coagulation/flocculation. The coagulation/flocculation is the process of binding small particles in the water together into larger, heavier clumps, which settle out relatively quickly. The larger particles are known as floc. Properly formed floc will settle out of water quickly in the sedimentation basin, removing the majority of the water's turbidity. Different dosages of coagulants are tested using a jar test, which mimics the conditions found in treatment plant that practice coagulation.

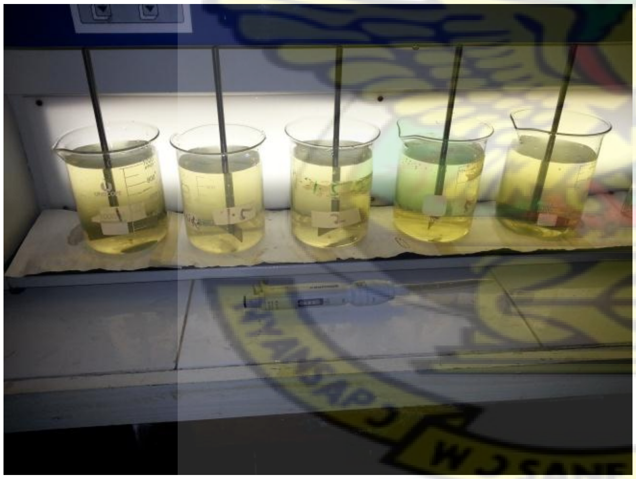


Plate 9 Beakers with solution in it undergoing Jar test

Plate 10 Beakers with solution, allowing the solution in it to settle after Jar test

3.1.3.1.1 Apparatus

- Volumetric flask (1,000 mL)
- Analytical balance
- Coagulants and coagulant aids
- Magnetic stirrer (optional)
- A stirring machine with six paddles capable of variable speeds from 0 to 100 revolutions per minute (RPM)
- Beakers (1,000 mL)
- Pipets (10 mL)
- Watch or clock
- Turbidometer and sample tubes

3.1.3.1.2 Reagents

Aluminum Sulfate

DI water

3.1.3.1.3 Procedure

A stock Solution of the alum was prepared. (0.9g/l). Six 1L beakers were filled with 1000ml of the raw water. The turbidity and pH of the raw was taken. Vigorous mechanical stirring (150 – 200rpm) was carried in all beakers for about 1 minute. Varying known volumes (1ml, 1.5ml, 2ml, 5ml, 10ml) of the stock solution was pipetted and added to the beakers in ascending other. Slow mix (15rpm) during 20minutes. The stirrers were turned off and solutions in beakers left to stand for 30minutes to allow suspended particles and flocs formed to settle. Periodically 50ml was taken from the solutions in the beaker whilst avoiding re suspension of the settled material using a pipette. The turbidity and pH of the samples taken from the 1L beaker were recorded.

3.1.4 Quality Parameter Tests on Coagulated Water

The following parameters were analyzed for, using the samples taken; turbidity, hardness, pH, conductivity, color, total dissolved solids, and total suspended solids,

3.1.5 Costing

3.1.5.1 Cost of chemicals

- Sulphuric Acid = 120ghcedis = 2.5l
- 165ml = 7.92cedis
- Potassium Sulphate = 500g
- 500g = 70cedis
- 170g = 23.8cedis

3.1.5.2 Energy Cost

- Calcined of bauxite waste
- Digestion of samples for 4hours
- Energy used by the furnace to calcine the bauxite waste for an hr. = 1.5kw
- Energy used during digestion = 700w

- Energy used throughout the process = 2.2kw
- Electricity Company of Ghana tariffs
- For commercial / Non residential Rates
- 1 – 300units = 0.2527 * 2.2 = 0.55594 = 60p

3.1.5.3 Others

- Filter paper 60per box = 60 cedis
- 25sheets are used per filtration
- Cost of filters 50cedis per 100pcs
- Maximum filter used 5pcs.
- Cost of 5 filter papers = 2.5cedis

3.1.5.4 TOTAL COST

Total cost of alum produced = 0.55594 + 2.5 + 7.92 + 23.8

= 34.77594 = 35cedis

The cost of producing 1159g of alum is 35cedis

The costing excludes labour cost and other administrative cost.

4 RESULTS AND DISCUSSION

This study was undertaken to produce alum from bauxite waste. This chapter therefore presents the data and the findings that were made under the study. Base on the coagulating test, the characterization of the alum, cost of the protocol, effect of the pH of the alum on raw water, residual aluminium and sulphate the best protocol was chosen out of the four major protocols developed. Jar test was conducted to ascertain the potency of the alum produced. The pH, total dissolved solids, conductivity and colour were also determined.

4.1 Production of Alum

To produce Alum from bauxite waste four major protocols were developed out of the numerous trials.

Protocol one, which produce the white sample as indicated in the methodology. Protocol one had 20g of the bauxite waste calcined at 600 ^{o}C for an hour. The calcined bauxite waste was digested for about four hours with 165 ml as the volume of conc sulphuric acid used during the digestion under refluxing conditions. The digested solution was allowed to stand for 20mins and later filtered. The filtrate was then reacted with 170g of Potassium Sulphate and heated to boil, which was cooled in ice cubes to aid crystallization.

Protocol two, which produce the brown sample as indicated in the methodology. Protocol two had 20g of the bauxite waste calcined at 600 ^{o}C for an hour. The calcined bauxite waste was digested for about four hours with 150ml as the volume of conc sulphuric acid used during the digestion under refluxing conditions. The digested solution was allowed to stand for 20mins and later filtered. The filtrate was then reacted with 170g of Potassium Sulphate and heated to boil, which was cooled in ice cubes to aid crystallization.

Protocol three and four, produced the light brown and deep brown samples respectively. Both protocol had 20g of the bauxite waste calcined at 600 ^{o}C for an hour. The calcined bauxite wastes were digested for about four hours with 200ml and 180ml respectively as the volume of conc sulphuric acid used during the digestion under refluxing conditions. The digested solution was allowed to stand for 20mins and later filtered. The filtrates were then reacted with 170g of Potassium Sulphate and heated to boil, which was cooled instantly in ice cubes to aid crystallization.

The volume of acid used, hygroscopic nature of the samples and the colour of the alum produced were the preliminary observations that help in the selection of the most appropriate protocol, after which confirmatory test (XRD, and coagulating test) were conducted to ascertain the choice.

The sludge produced in all the protocols were acidic. The sludge produced when allowed to stand for an hour unattended to began to harden. The acidic nature of the produced sludge if not treated but discharged into the environment would cause the environment to be acidic and when leached into the streams would destroy aquatic life in the stream and destroy the flora of the environment.

The protocols developed had certain basic similarities such as calcine of the bauxite waste for an hour, digesting of the bauxite waste and the sulphuric acid solution (Sludge) for four hours using refluxing, the filtration method and the mass of the potassium sulphate added.

Variations were also evident in certain aspects of the four protocols; typical amongst them is the volume of the sulphuric acid used. All the protocol had as one of its unique features being that the color of the sludge turning creamish from reddish brown after the four hours of heating.

4.2 Jar Test

4.2.1 Turbidity

As stated earlier in chapter 3, jar test was conducted to ascertain the coagulating feature of all the alum produce from the various protocols. The performance of all the protocols in relation to coagulation is demonstrated and represented in Fig 4- 1. The turbidity of the raw water before the commencement of the coagulation process was 63.84NTU. Fig 4-1 proved that every single alum produced from the lab had a coagulating feature but the difference was shown from the rate and the concentration of alum used for the coagulation purpose.

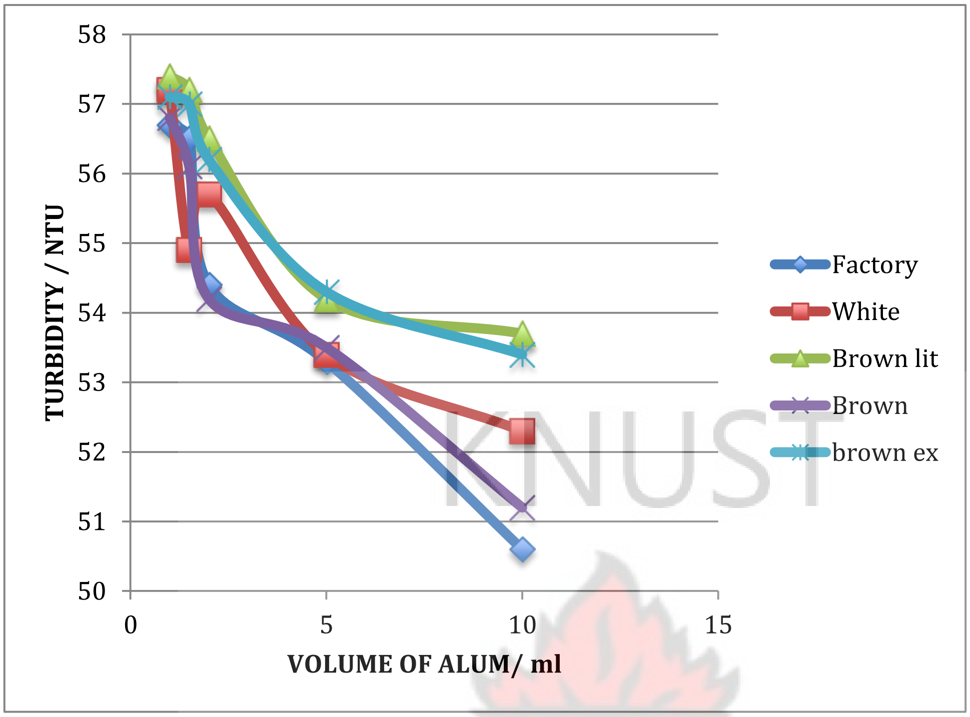


Figure 4-1 Relationship between Turbidity and Volume of the samples

Fig 4-1 represents the results of four different protocols used in the production of alum and their output in relation to turbidity. In all, the samples produced compares very well with the commercially available Alum. The Coagulating property of all the alum produce behaves like the commercially available alum.

4.2.2 pH

After the turbidity of the various water samples was recorded the pH was now taken to determine the total number of hydrogen ion concentration in the solution. The various pH as the alum solution was put into the raw water during the jar test and after jar test experiment was completed was recorded this is represented in the graphs shown below.

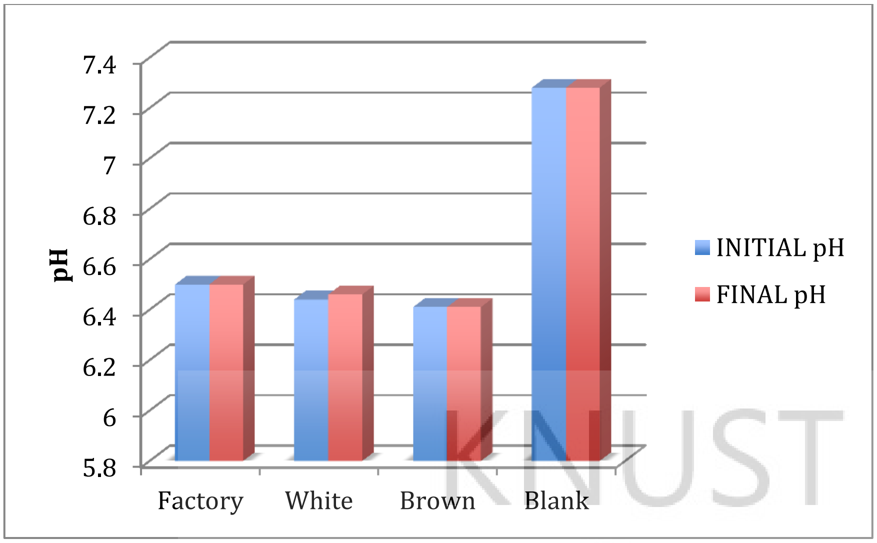


Figure 4-2 Initial and Final pH for a volume of 1.5mL during Jar Test

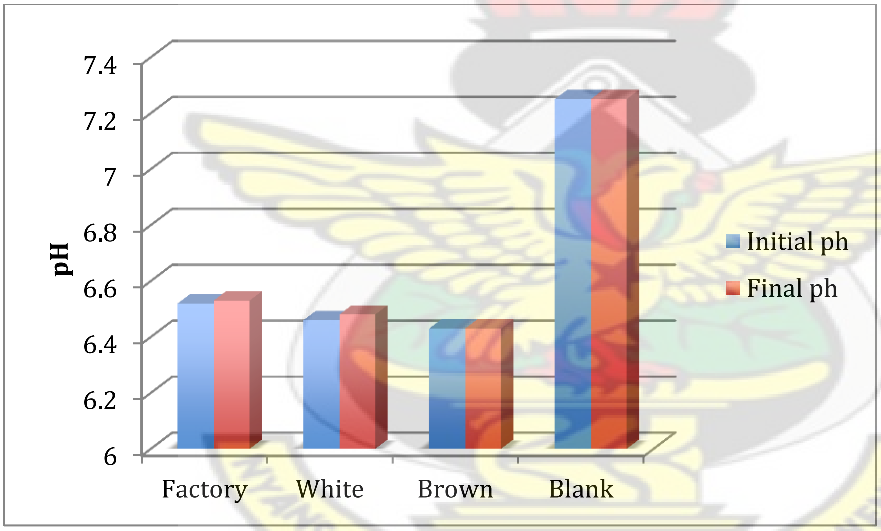


Figure 4-3 Initial and Final pH for a volume of 1.0mL during Jar Test

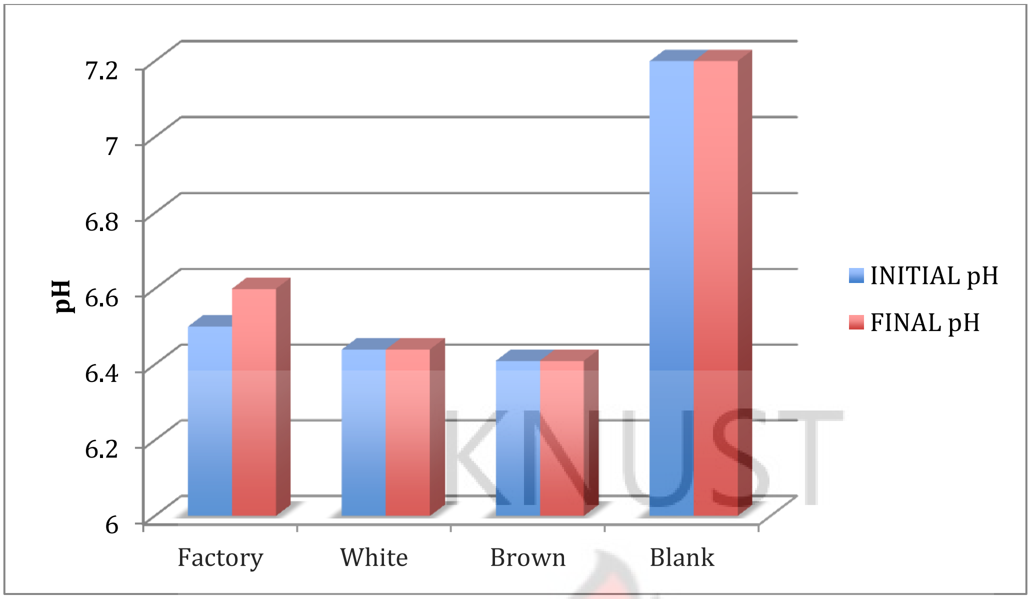


Figure 4-4 Initial and Final pH for a volume of 5.0mL during Jar Test

The figures shown above showed that all the samples contributed some amount of acid to the raw water, making the water acidic than it was initially, this can be explain with the following chemical equations. The chemical formula for potassium aluminium sulphate is $KAl(SO_4)_2.12H_2O$

$KAl(SO4)_2$ dissociates in water to make K^+, Al^{3+} and SO_4^{2-}.

K^+ is the cation of a strong base and it does not hydrolyze.

$Al^{3+} + 4HOH \text{-->} [Al(OH_4)]\text{-} + 4H+$ ……………(2)

This lowers the pH, by increasing the concentration of H+

$SO_4^{2-} + HOH \text{<==>} HSO_4 + OH\text{-}$ …………………. (3)

This raises the pH, by increasing the concentration of OH-

The net effect is that the production of H+ is greater than the production of OH-, resulting in an overall increase in H+ and a lowering of pH.

In the three figures above it was realized that the white sample was always basic as compared with the brown sample, which can be explained by the chemical equations above. In equation 2, which was proven, that Al^{3+} when hydrated produces 4moles of hydrogen ions, which contributes to the acidity of the solution. In equation 3, which happens to be the hydration of SO_4^2 it was shown at the product end that OH ions were formed contributing to basicity, with this two equations, it can be concluded that the higher the concentration of SO_4^2 in the produced sample the higher the pH making it basic and the lower the concentration of SO_4^2 in the produced sample the lower the pH making it acidic. The white sample was produced using 165ml of sulphuric acid and that of the brown with 150ml meaning the alum produce from the white sample would have high concentration SO_4^2 thereby contributing to its basicity as shown in equation 3.

4.2.3 Settling Rate

The introduction of Alum in raw water source causes the agglomeration of colloids to form flocs. The agglomeration of particles increases the effective size and therefore the settling velocities hence the increase in the settling rate and the less time it takes for the flocs to settle. The settling rate is one of the most important parameters to ascertain the potency of the coagulant since the time at which the flocs settle are the most important role or function of coagulants. The graphs showed below are the settling rate of the alum produced out of two different protocols at different concentration (1ml, 1.5ml, 5ml). Different volumes of the alum were used, just to know the exact volume of alum needed for the coagulation at the best settling rate.

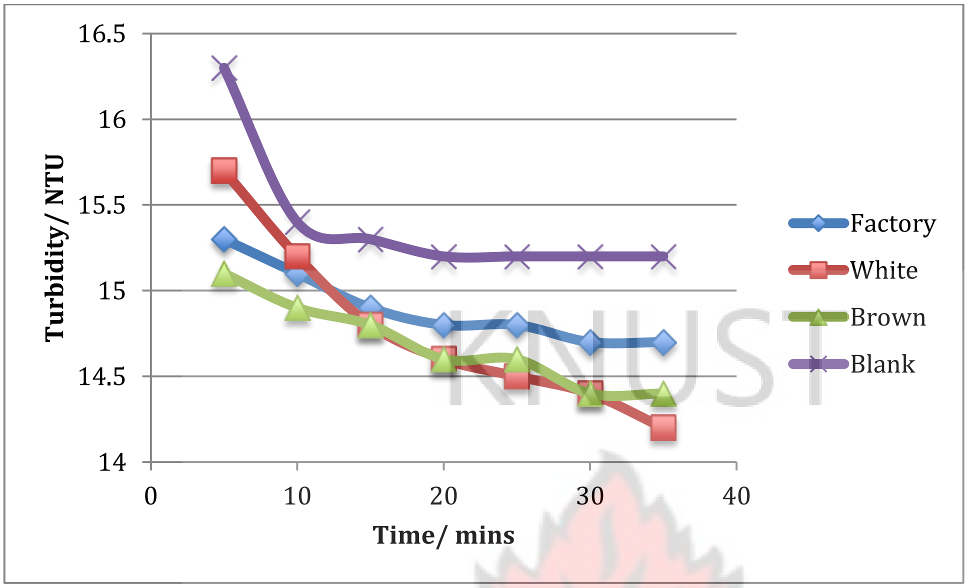


Figure 4-5 Settling Rate of Samples at a volume of 1.0mL

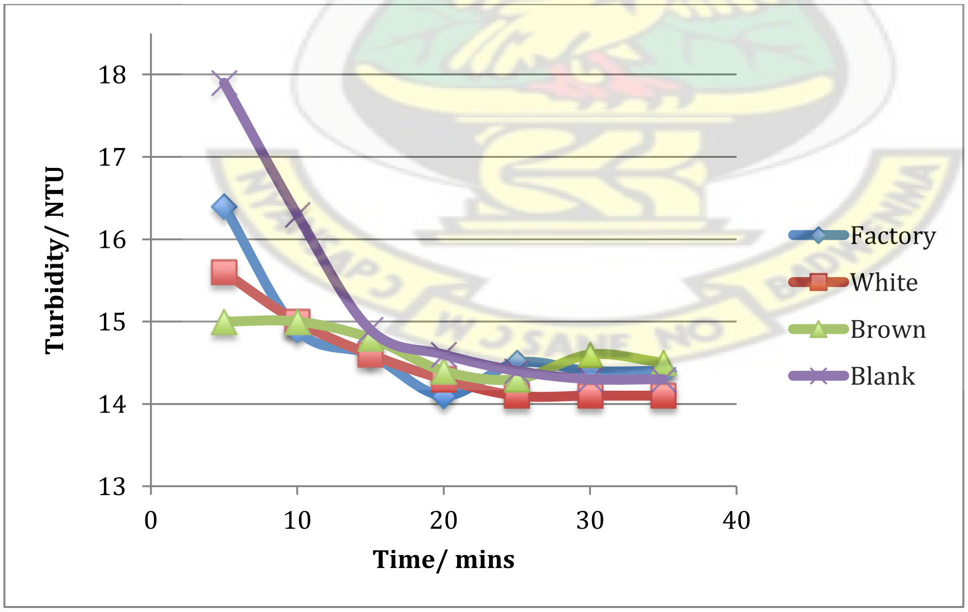


Figure 4-6 Settling Rate of Samples at a volume of 1.5mL

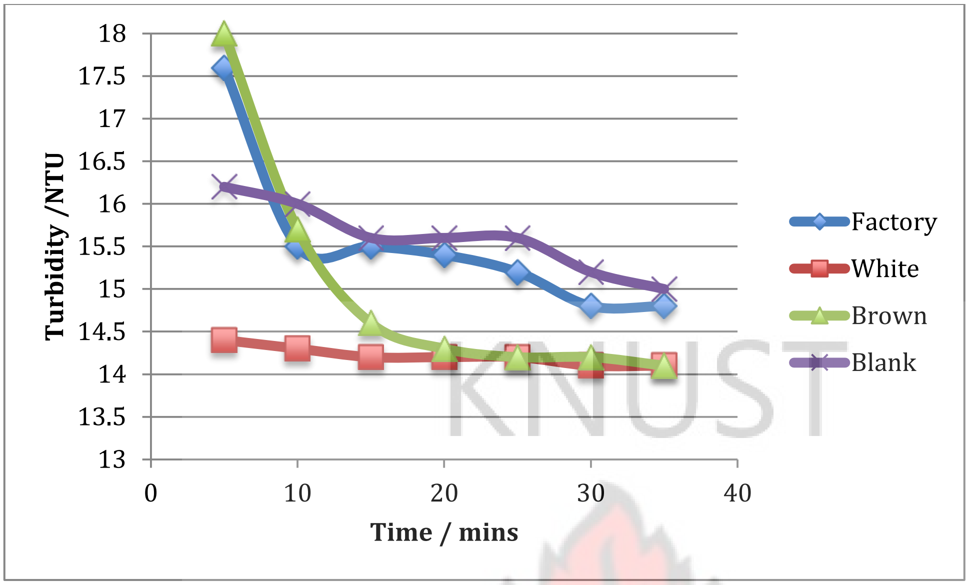


Figure 4-7 Settling Rate of Samples at a volume of 5.0mL

In all the three graphs it was observed that coagulation and settling occurred as expected. The settling rate of the white sample was faster than all the other samples used at concentration 1ml of the alum. With 1ml used to dose the raw water, the settling rate of the brown and white samples are all comparable to the commercially available one. This observable feature was same for all the concentrations. In all the graphs one clearly observable feature was that they all worked better than the blank raw water, which was allowed to settle naturally without disturbance as indicated in Momade and Sraku lartey, 2010.

4.2.4 Residual Aluminium and Sulphate

4.2.4.1 Sulphate

The concentration of sulphates in the water after coagulation was determined and the results are shown in Fig 4-8.

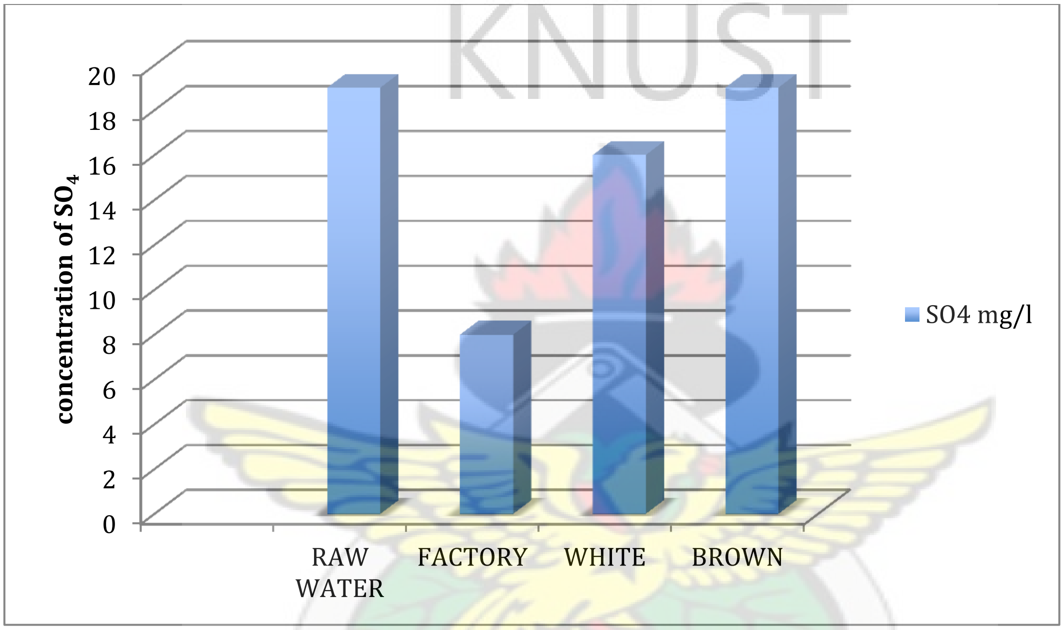


Figure 4-8 Concentration of SO4 mg/l in water sample after Jar Test

High concentrations of sulphates in drinking water have three effects: tend to form hard scales in boilers and heat exchangers, cause taste effects and lastly cause laxative effects with excessive intake. The WHO has its standard at 250mg/l meaning the two basic samples produced meets the WHO standard, hence wont cause the three effects stated above when used as a coagulant to treat drink water. The sulphates levels of all the alum produced had it residual sulphate levels been extremely low as compared to the WHO standard of 250mg/l for drinking water.

4.2.4.2 Aluminium

Fig 4-9 below explains the residual aluminium after treatment but it is evident how the locally produced alum performed far better than that of the factory produced one. When Alum is dissolved in water, the Al^{3+} hydrates to form an aquometal ion $Al(H_2O)_6^{3+}$. The aquometal reacts to form monomeric and dimeric hydroxo complexes and possibly polymers. At high pH, negative species are formed, reducing the effectiveness of the coagulation and resulting in high Al residuals (Naeem and Malmberg, 1985). This explains why the commercially available alum which produced water solution of high pH after coagulation having high levels of residual aluminium whilst the brown and white record low levels of residual aluminium. AWWA and WHO set a standard of 0.05mg/l as the safe zone for drinking water in relation to residual aluminium but with this work none of the samples were able to attain the AWWA and WHO standards, which was partly due to the pH of the raw water solutions after coagulation.

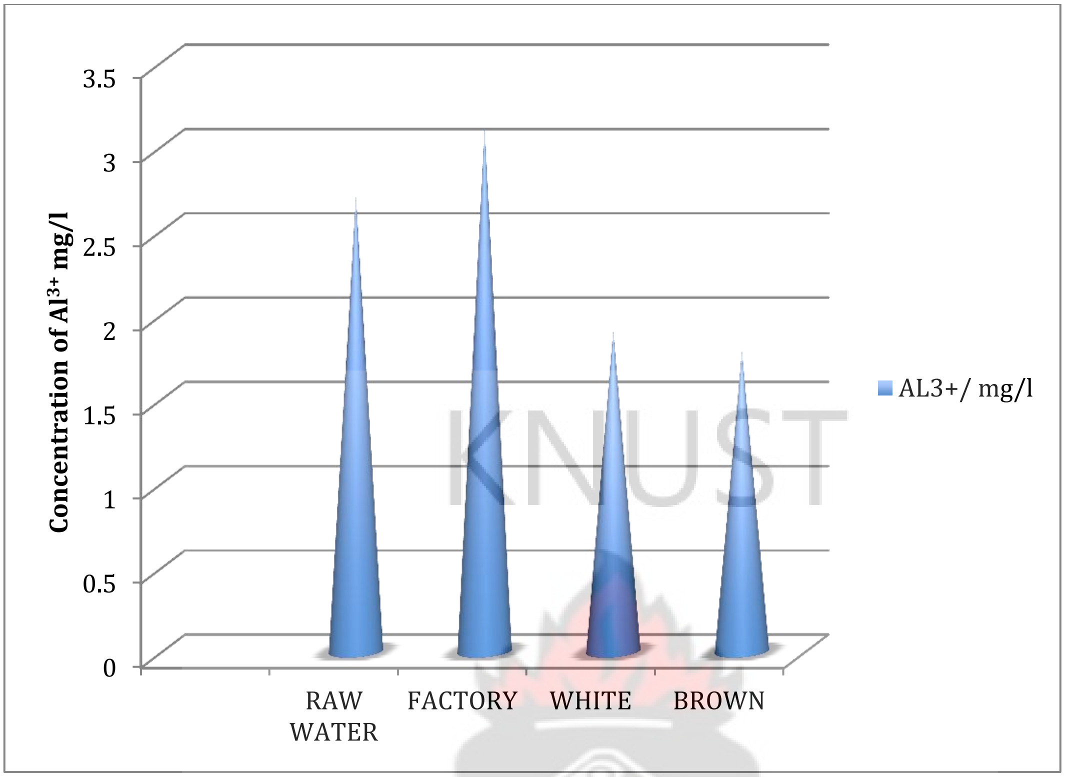


Figure 4-9 Concentration of Al^{3+} mg/l in water sample after Jar Test

4.3 X Ray Diffractomer.

XRD is used in the characterization of the produced Alum, x ray diffractometer was used to determine the percentage of the various elements/compounds in the sample. The white and brown samples produced out of two different protocols proved positive to all the parameters to ascertain its potency, hence a final confirmatory test to check the chemical components or constituents of the samples produced. The results from an x ray diffractometer is shown below.

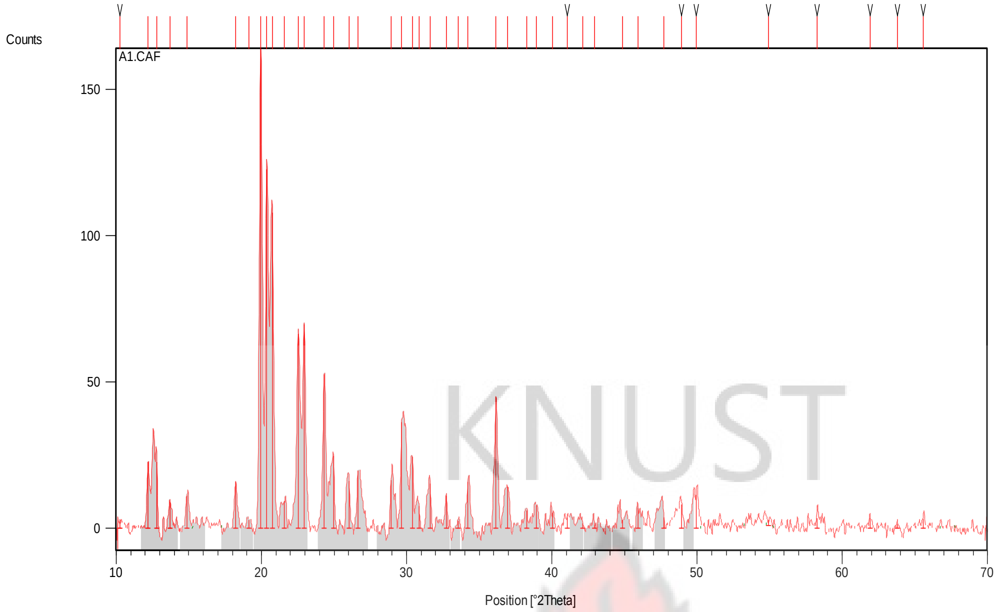
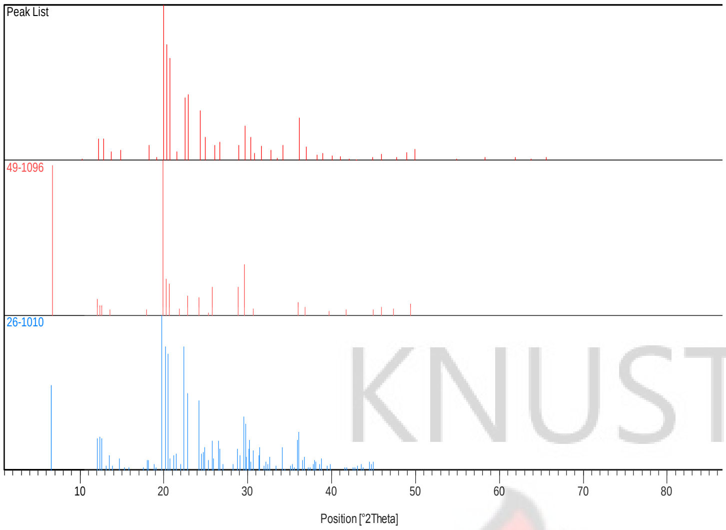


Figure 4-10 An XRD scanned graph displaying the molecular characterization of the white sample

Table 4-1Exact components of the white sample

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	49-1096	44	Aluminum Sulfate Hydrate	0.000	0.983	$Al_2(SO_4)_3.16H_2O$
*	26-1010	36	Alunogen	0.000	0.336	$Al_2(SO_4)_3.17H_2O$

Fig 4-10 shows a scanned graph of the first sample (white), which indicates that Sample white has mainly aluminium sulphate hydrate as the major compounds present. It could be observed that the plot of identified phases matched the scanned peaks. In the said sample (white) is another compound identified to be alunogen. Alunogens are other forms of alum that is also colourless contaminated with some impurities.



WHITE
SAMPLE

Figure 4-11 An XRD scanned graph comparing sample white and factory produced one

The second scanned sample was the brown sample, the scanned phase showed that the sample doesn't contain aluminium sulphate, however it contains some Al-S-O compounds. The nature of the sample and the noise levels made it very difficult to identify the specific Al-S-O compounds present. The peaks gotten out of the scanned sample is shown below, and it depicts the noise level.

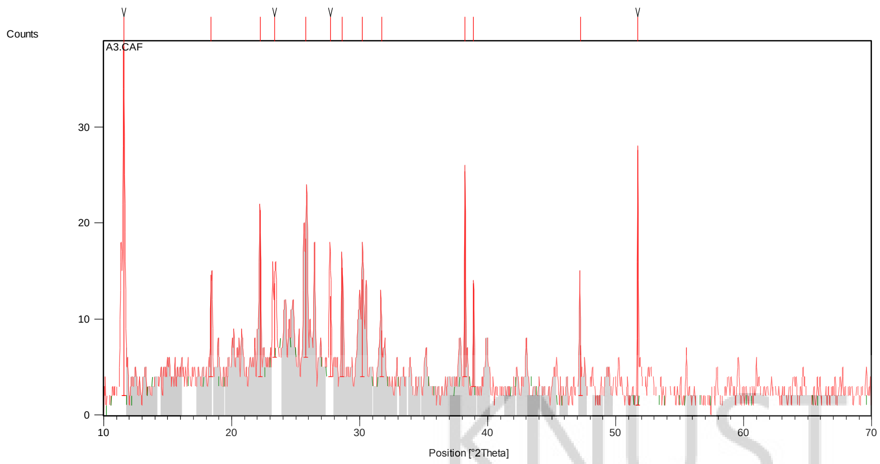
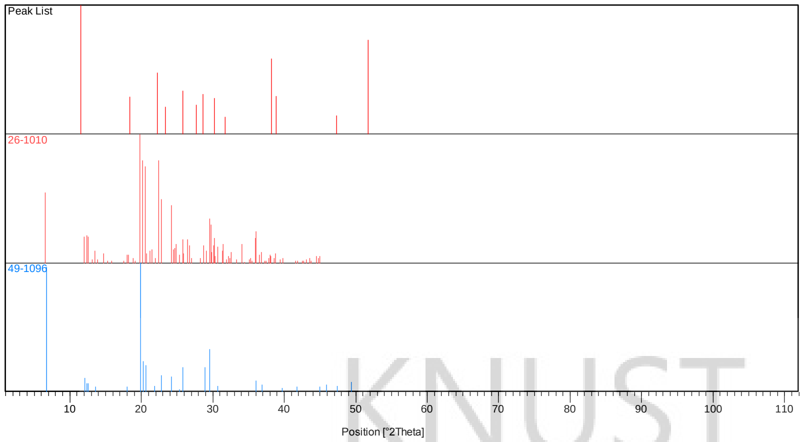


Figure 4-12An XRD scanned graph displaying the molecular characterization of the brown sample

Table 4-2Exact components of the XRD scanned graph of the brown sample

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	26-1010	Unmatched Strong	Alunogen	0.000	1.273	$Al_2(SO4\ )_3$ .17 H_2O
*	49-1096	1	Aluminium Sulfate Hydrate	0.000	1.393	$Al_2(SO_4)_3$ .16 H_2O



The two major samples produced out of the protocols were also compared and it was discovered that there were some major disparities in the samples as shown below

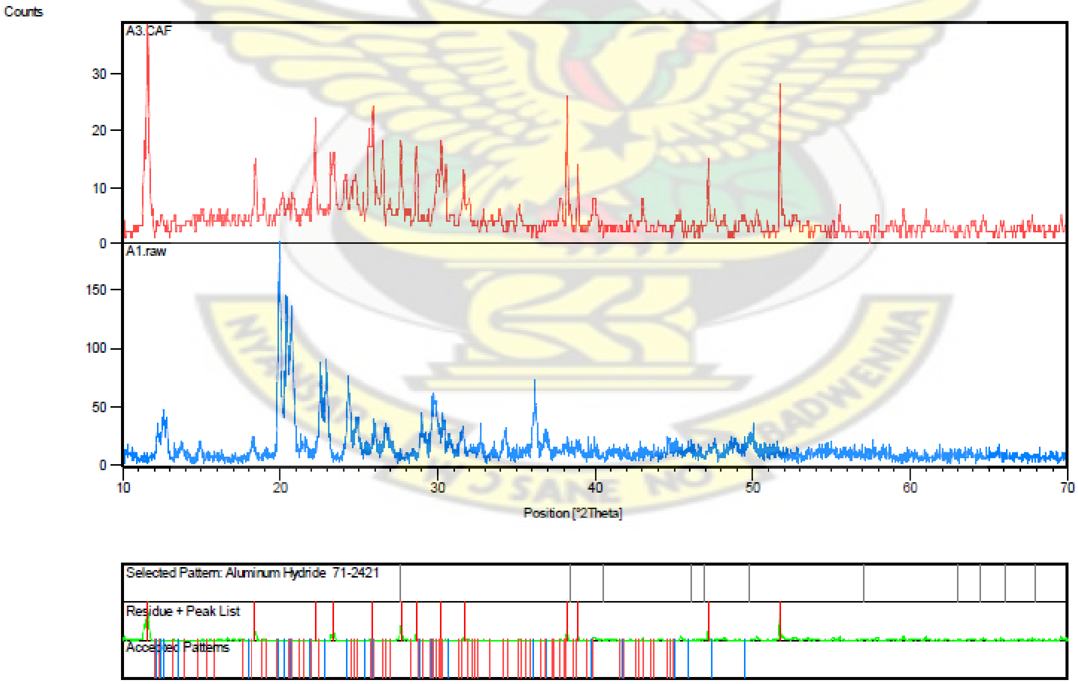


Figure 4-13 A comparism of the molecular characterization of the white and brown samples

In the diagram above the sample white is the one with blue peaks and sample brown has red peaks. The disparity is mainly shown in the nature of their peaks. The sample brown has so much noise in the background that the peaks couldn't stand out clearly.

4.4 Costing

Alum produced from the developed protocol was twice cheaper than the commercially available one sold on the market, the total cost of producing Aluminium sulphate was 35 cedis per 1.16kg and the one sold on the market is about 80 cedis per 1kg making the protocol very cheap as compared to the one on the market. The cost of the alum produced in the lab went high due to the cost of the potassium sulphate used and the cost of the filter papers was also high. The costing of our protocol excluded labour and other administrative cost (marketing, and packaging). Assuming that the cost of the protocol would increase by 100% if these other factors of cost are added, meaning that our costing would leave us at a price of 70cedis, which is still lower than the commercial available one.

4.4.1 Cost of producing 1.2kg of Alum

Item	**Cost (Ghana Cedis)**
CHEMICALS	
Sulphuric acid (165mL)	7.92
Potassium Sulphate (170g)	23.8
ENERGY	
Energy consumed for digestion and calcine (2.2KW)	0.60
Filter paper (5)	2.5
Total	**34.82 = 35**

5. CONCLUSION AND RECOMMENDATION

5.1 Conclusions

From the study conducted it can be concluded that:

Production of alum from bauxite waste is possible. The alum produced from the waste performed comparably well as the commercial Alum.

The protocols developed were four, of all the four protocols it was realized that the white alum protocol gave the best yield in terms of performance and its efficiency.

The white protocol was the protocol in which 165ml of the sulphuric acid was used and refluxing method was used as the means of digestion.

The concentration of the sludge produced after filtration is extremely acidic so the best means of disposal is by reducing the acidic level of the sludge. This was done by mixing it with ash being burnt plantain peels, this solves two problem; it reduces the difficulty in the disposal of the plantain peels and also decreases the acidity of the sludge. The sludge is then disposed off into the environment.

The peaks obtained from the X ray diffractometer when analysis of the white sample was done, produced similar peaks and patterns as obtained from the commercially available Alum.

The cost of producing alum locally is cheaper as compared to the foreign alum been sold and used on the market.

5.2 Recommendation.

Alternate source of sulphuric acid and potassium sulphate should be explored which would reduce the cost of production

Suction Filtration should be the means of filtration since that would increase the rate of filtration, which would reduce production time and also increase the aesthetic value of the produced alum.

Cost effective means should be looked at, at every single stage of the protocol developed to reduce cost.

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Appendices

Table 3 Turbidity of various using 1mL of the alum during Jar Test with time interval of 5mins

		Volume of Alum used/ml (1ml)		
Time of Settling	**Turbidity /NTU**	**Turbidity/NTU**	**Turbidity/NTU**	**Turbidity/NTU**
mins	Factory	White	Brown	Blank
5	15.3	15.7	15.1	16.3
10	15.1	15.2	14.9	15.4
15	14.9	14.8	14.8	15.3
20	14.8	14.6	14.6	15.2
25	14.8	14.5	14.6	15.2
30	14.7	14.4	14.4	15.2
35	14.7	14.2	14.4	15.2

Table 4 Turbidity of various samples using 1.5mL of alum during Jar Test with time interval of 5mins

		Volume of Alum used/ml (1.5ml)		
Time of Settling	**Turbidity /NTU**	**Turbidity/NTU**	**Turbidity/NTU**	**Turbidity/NTU**
mins	Factory	White	Brown	Blank
5	16.4	15.6	15	17.9
10	14.9	15	15	16.3
15	14.6	14.6	14.8	14.9
20	14.1	14.3	14.4	14.6
25	14.5	14.1	14.3	14.4
30	14.4	14.1	14.6	14.3
35	14.4	14.1	14.5	14.3

Table 5 Turbidity of various samples using 5mL of alum during Jar Test with time interval of 5 mins

		Volume of Alum used/ml (5ml)		
Time of Settling	**Turbidity /NTU**	**Turbidity/NTU**	**Turbidity/NTU**	**Turbidity/NTU**
mins	Factory	White	Brown	Blank
5	17.6	14.4	18	16.2
10	15.5	14.3	15.7	16
15	15.5	14.2	14.6	15.6
20	15.4	14.2	14.3	15.6
25	15.2	14.2	14.2	15.6
30	14.8	14.1	14.2	15.2
35	14.8	14.1	14.1	15

Table 6 Residual Sulphate in the water sample after Jar Test using the various produced alum.

SAMPLE	SO_4 mg/l
RAW WATER	19
FACTORY	8
WHITE	16
BROWN	19

Table 7 Residual Aluminium in the water sample after Jar Test using the various produced alum

SAMPLE	AL^{3+}/ mg/l
RAW WATER	2.7
FACTORY	3.1
WHITE	1.9
BROWN	1.78