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Preparation and Characterisation of Alum from Aluminum Can For Waste Water Treatment

[1]Sadisu Girigisu, [1]Mohammad, L.B., and [1]Adeyemi, C.O
[1]*Department of Biochemical and Chemical Sciences, The Federal Polytechnic Offa, P.M.B. 420, Offa, Nigeria*
Corresponding author: Sadisu GIRIGISU

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ABSTRACT
Alum of the form $K_{4.00}Al_{4.00}S_{8.00}$ and JCPDS NO: 96-101-1178 was succesfully synthesized from this research, the synthesized alum was accordingly characterized using X-ray diffractometry , X-ray flouresence and Fourier transformed Infrared Spectroscopy techniques respectively to identify the presence of alum.The XRF analysis reveal the sample to consist of aluminium oxide of the form Al_2O_3 (14.825 %), silicon of the form silicon dioxide SiO_2 (0.674 %) and potassium oxide of the form K_2O (25.126 %). The FTIR analysis also reveal the sample to contain major aluminium oxide absorbtion bands at 465.9 and 603.8 cm^{-1}respectively. The synthesized alum was using in carrying out coagulation on waste water succesfully.

Keywords : Aluminium can, Alum , XRD, XRF, FTIR and Coagulation

I. INTRODUCTION

Alum, a colourless to white crystalline substance is a constituent of the mineral alunite $KAl(SO_4)_2{\cdot}2Al(OH)_3$ and occurs naturally as potassium aluminium sulphate (Kalunite) $KA1(SO_4)_2{\cdot}12H_2O$ (Considine G D. and Van Nostrand, 2005). It belongs to a class of hydrated double sulphate salts of univalent and trivalent cation and has a general formulae MI SO_4, $MIII_2(SO_4)3{\cdot}nH_2O$. Other common alums are ferric ammonium alum $NH_4Fe(SO_2)_2{\cdot}12H_2O$, and sodium chrome alum $NaCr(SO_4)_2{\cdot}12H_2O$

Due to the increasing demands for water utilization by man and for other uses, it is imperative to monitor the means of water purification from various sources. Consequently, one of the major purification processes for water is coagulation which often requires the use of a coagulant such as alum. pure bauxite (which is of higher alumina content) is the main conventional raw material for alum production, Apparently this mineral is not available in large quantities for commercial alum production in Nigeria (Adekola et al, 2017). Bauxite ore must undergo an expensive and energyintensive procedure to be converted into alum for industrial use (Ding, X. et al. 2018). In addition to using a lot of energy, the process also causes soil erosion (Zheng, Y. et al. 2016), deforestation (Jo, J.-Y. et al. 2021), and air pollution (Moshammer, H. 2010) that contributes to environmental distress.

Therefore, for economic consideration, energy saving and reduction of waste in the enviroment,the use of aluminium can generated from aluminium waste can be recycled via chemical treatment to produce alum crystal, Aluminum recycling (2013 and Chris, O.O 2010). Aside this, the economic implication of importing this raw material is becoming very enormous as the exchange rate has gone on a higher side. Thus the use of aluminium can from wastes generated from a variety of activities associated with industries polluting our environment can be recycled.

Alum has always been produced using aluminum beverage cans. The output of the product was observed to increase with increasing the amounts of sulfuric acid and potassium hydroxide used (Yun Ming, L. et al. 2015). Ugwekar (2012), also found that increasing the amounts of H_2SO_4 , K_2SO_4 , and KOH led to an increase in the yield of potash alum.

Therefore, the objective of this research is to synthesise alum crytal from waste aluminium can and characterise the produced alum using XRD and XRF respectively.

II. MATERIAL AND METHOD

MATERIAL

The material used in this research includes aluminium can collected within the premises of Federal polytechnic Offa, Kwara state, sand paper

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REAGENT

The reagent used includes distilled water, Potassium hydroxide, sulphuric acid, Sodium chloride all of analytical grade

METHOD

Paint coatings over the empty aluminium cans were removed using sand paper and the empty cans were broken into small pieces. This was thoroughly washed and rinsed with demonized water.1.0 g of clean aluminium pieces was weighed and transferred into empty 250 ml beaker, this was followed with the addition of 50 ml of 1.4 M KOH solution slowly and the resulting solution was placed in a fume cupboard.

During the reaction, the initially colourless mixture turned to dark grey blackish solution. The cold black solution formed was filtered and a clear fliterate colourless solution was obtained. The clear filtrate was transferred into a clean beaker, cooled by placing the beaker in an ice bath . Slowly and carefully, (with a graduated cylinder) and stirring quickly with care, 20 mL of 4.0 M solution of H_2SO_4 was added to the cooled and colourless solution till the solution get warm.

Initially, a thick, white, gelatinous precipitate was formed as more acid was added. The solution was boiled to evaporate excess water. Reaction beaker was kept into the ice-water bath to chill. The mixture was allowed to chill for 15 minutes, and as the solution cools, solid alum precipitated out forming alum crystals.

Finally, the alum crystal was removed from the solution after 24 hours by filtration and washed with a mixture of 20 % v/v aqueous ethanol . This serves to wash the isolated crystal as it dries and to remove any form of impurity. The alum was placed on filter paper and allowed to dry overnight and then re-weighed. The overall reaction is therefore of the form:

$$2Al_{(s)}+2KOH_{(aq)}+4H_2SO_{4(aq)}+22H_2O_{(l)}\rightarrow 2KAl(SO_4)_2.12H_2O_{(s)}+3H_{2(g)}$$

III. RESULT AND DISCUSSION

The crystals of alum were removed from solution by gravity filtration and were then washed in an ethanol:water mixture. This helps remove any contaminants from the crystals, and also helps dry the crystals quickly.

Fig. 1:Process of precipitation of the Alum

Fig. 2: Alum crystal formed after washing and drying

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CHARACTERISATION OF THE PRODUCED ALUM USING XRD, XRF AND FTIR

The produced alum was characterised with Xray diffractometry techniques in order to determine the essential peaks of the crystal of alum formed

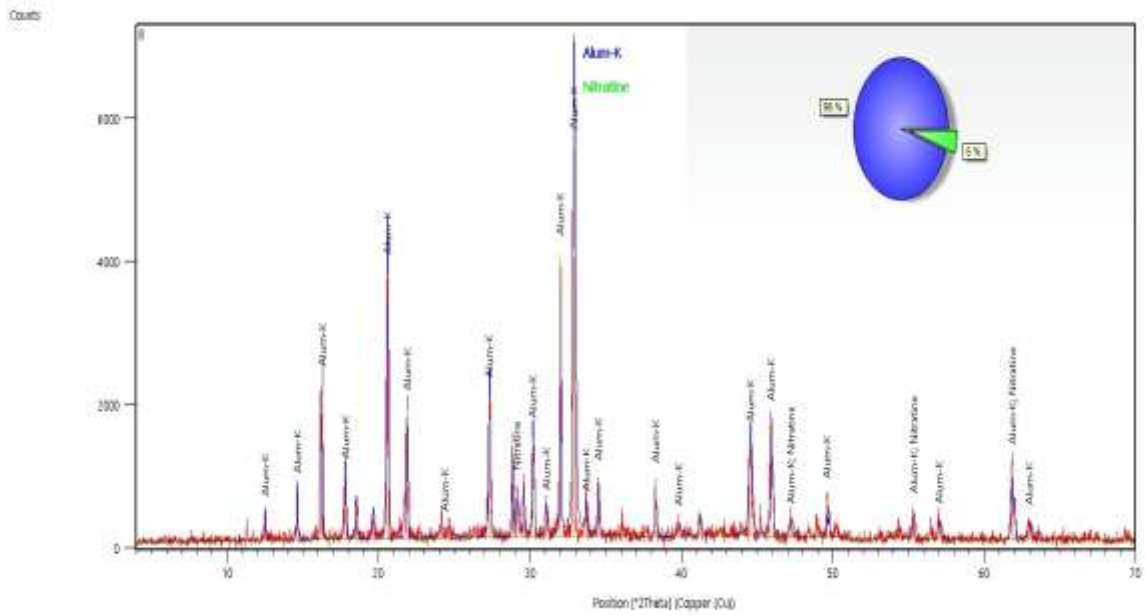

Fig.3:X-ray diffraction pattern of the leached product showing the identified product:Alum ($K_{4.00}Al_{4.00}S_{8.00}$:96-101-1178),Nitratine($Na_{6.00}N_{6.00}O_{18}$: 96-900-7556).

As it could be observed on the spectrum, the prominent peaks is that of Alum which covers 95% of the total spectrum with JCPDS NO: 96-101-1178, the 5% nitratine observed was as a result of impurities obtained during the analysis. The most prominent peak was observed at 2θ of 21.5 and 32.5° respectively, this is similar to the work of Adekola et al (2017) who also obtained similar result.

Also the XRF analysis of the produced alum was also carried out, the table below indicates the amount in percent of the components of the produced alum.

Table 1.0: X-ray elemental composition of raw wolframite ore by XRF

compund	Al_20_3	SiO_2	P_2O_5	SO_3	K_2O	Fe_2O_3	Ga_2O_3	Rb_2O
% Conc.	14.825	0.674	0.825	58.526	25.126	0.17	Traces	Traces

T

he x- ray fluorescence analysis of the synthesized alum crystal is reported in Table (1.0). The main compounds identified on the table in % are Al_2O_3 (14.825), SO_3 (58.526), and K_2O (25.126), these oxides forms the major component of the alum. This as a result of potassium hydroxide that was used in dissolving the aluminium can at the initial stage, while the sulphate oxide was as a result of the sulphuric acid used in precipitatin the alum out thus making it potassium aluminium sulphate.

This corresponds with the XRD analysis which reveals the name of the compound as alum with formular ($K_{4.00}Al_{4.00}S_{8.00}$). Other compounds observed either in small quantities or traces which may be as a result of impurities introduced during the analysis.

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FTIR analysis of the synthesized alum

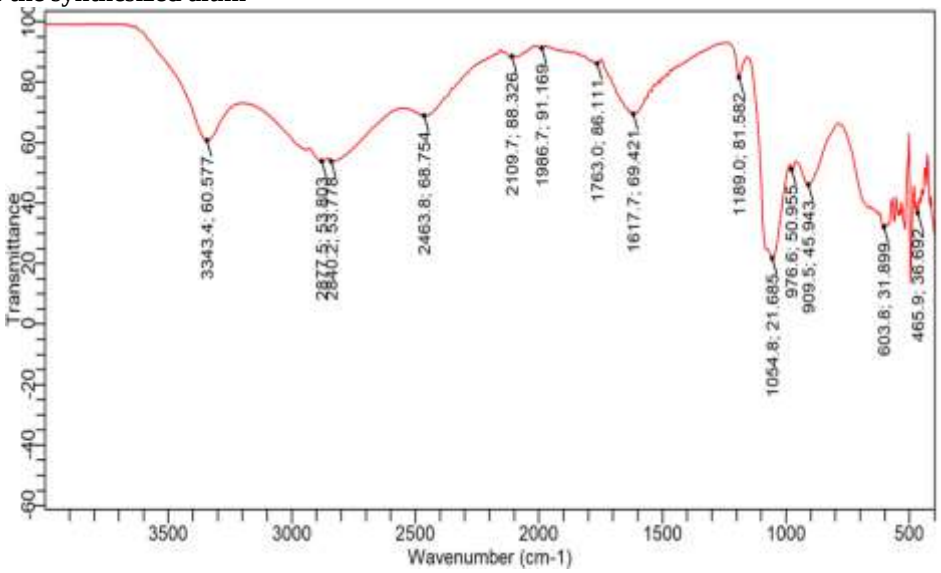

Fig.4 : FTIR spectrum of synthesized alum

The main peaks at 465.9 and 603.8 cm^{-1} can be assigned to the Al–O stretching mode in octahedral structure; the band around1054.8 cm^{-1} are related toAl–O stretching mode in tetrahedron and symmetric bending of Al–O–H, respectively(Peng et al., 2011; Shen et al. 2012 and Shek et al 1997).

Qualitative Chemical Tests for Aluminum ion (Al^{3+})

To a prepared alum solution, two drops of diluted potassium hydroxide was added, this was followed with the addition of Sulfuric acid in drops and then in excess. A thick white gelatinous precipitate was formed, which was insoluble in drops but soluble in excess sulfuric acid. This indicates the presence of aluminum ion in the synthesized alum crystal (Xu et al ,2016). To confirm the presence of potassium ions (K^+) in the synthesized alum. The crystal was held in a flame for 20 seconds until the red flame turned pale purple which is an indication that potassium ion is present in the produced alum (Klug, H. P., and Alexander, L. 1940)

Confirmation for sulfate ion (SO_4^{2-})

One gram of the grinded alum crystal was added to a testube half filled with water, the mixture was stirred thorougly till a homogenous solution is obtained. This was followed with the addition of two drops of aqueous barium chloride solution, a white precipitate was observed which remain insoluble in excess barium chloride indicating the presence of SO_4^{2-} ion (Li, F., and Yuan, G. 2005).

Melting point Determination of Alum crystal

Capillary tube was packed with alum crystal and fastened with a thermometer. Thereafter auniversal clamp and cork stopper were used to hold the thermometer to a retort stand. The capillary tube along with the thermometer were immeresed in a beaker containing paraffin which is already heated. The point at which the alum crystal melts wes observed and recorded accordingly.

Table 2.0: Qualitative analysis of ions present in the synthesized alum crystal

Test	Observation	Inference
Alum solution + Aqueous $Bacl_2$ solution	White precipitate formed and insoluble	SO_4^{2-}
Solid Alum Crystal + heat	Red flame turned to pale purple	K^+ confirmed
Aluminate ion solution + H_2SO_4 in drop and then, in excess	Thick, white gelatinous precipitate formed, insoluble in drop but soluble in excess	Al^{3+} confirmed

IV. CONCLUSION

The following conclusions can be drawn from this study:

1. Aluminium can wastes which litters the enviroment were effectively recycled via chemical treatment to synthesize alum crystals
2. The synthesized alum were characterized with XRF, XRD and FTIR respectively to identify the formation of the alum crystal.
3. Physico-chemical test were carried out on the synthesized alum
4. The synthesized alum succesfully serve as coagulant in water purification.

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