EVALUATION OF NIGERIAN ALUMINIUM ORES FOR MAKING ALUM

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ABSTRACT

The preparation of potassium aluminium sulphate dodecahydrate ($KAl(SO_4)_2.12H_2O$), potash alum, from some locally available ores in Bauchi and Plateau states of Nigeria is described. In a comparism of bauxite, kaolin, feldspar, muscovite and beryl, bauxite gave the highest yield (71%) of the potash alum crystals. The results indicate a non-quantitative relationship between the potash alum yields and aluminium content of the ores. The crystals were analysed for the parameters; assay, water of crystallisation, pH, aluminium, potassium and sulphate ions. These parameters were similar for alum samples prepared in this work and those obtained commercially.

INTRODUCTION

Nigeria has the potential of being self sufficient in the supply of certain raw materials if her natural resource base can be harnessed and utilised[1-3]. Potassium aluminium sulphate, dodecahydrate, potash alum, is a well known chemical that has a variety of uses, notable among them being water purification, dyeing, fire proofing fabrics, baking powders and fire extinguishers[4]. The technology of potash alum manufacture[5] is very simple and involves the use of relatively cheap raw materials.

This paper evaluates ores derived from Bauchi and Plateau states for the preparation of potash alum by a relatively simple process.

EXPERIMENTAL

Ore sample collection

Kaolin and beryl were collected from Alkaleri and Tafawa Balewa local government areas (LGAs) of Bauchi state respectively. Bauxite, feldspar and muscovite were obtained from Bassa LGA in Plateau state. Commercial potash alum was purchased from a local vendor of chemicals in Bauchi town.

Determination of ore solubility

28g of crushed and sieved ore was added to one litre of distilled water and warmed with stirring for 30 minutes. The mixture was then filtered and the residue dried and weighed; the exact amount of ore that dissolved was computed by subtracting the weight of dry residue from 28g to give the ore solubility in g/l. The results are presented in Table 1.

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Aluminium content of ore samples

Aluminium content of the ore was determined by adapting a literature procedure[6] based on complexiometric titration using EDTA. 28g of crushed and sieved ore was placed in a furnace at 800°C for 3hrs. On cooling, the residue was weighed and dissolved in 1 litre of distilled water. The mixture was then filtered and the residue dried and weighed; the exact amount of ore that dissolved was obtained by subtracting the weight of dry residue from 28g. 1cm³ of the ore solution was diluted to 100cm³; 25cm³ of this solution was titrated against standardized 0.01M EDTA solution with eriochrome black T as indicator. An additional 0.4cm³ of EDTA solution was added after each end point, and for each titration NH_4OH was used in making the solution alkaline. The excess EDTA in the ore solution was then titrated against 0.01M $ZnSO_4$. 1cm³ of 0.01M EDTA = 0.2698mg of Al: the Al content was calculated and reported as a percentage of the Al ore solution, and the results presented in Table 1.

Table 1: Selected characteristics of ores and alum samples derived

Al ore	Solubility of ore (gl^{-1})	Al content of ore (%)	Yield of potash alum (%)
Bauxite	27.05	31.75	70.75
Kaolin	27.01	20.98	69.98
muscovite	26.98	18.82	62.42
beryl	26.80	9.23	52.22
feldspar	26.74	7.49	51.98

Preparation of Potash Alum ($KAl(SO_4)_2$

The preparation was carried out by adapting a literature procedure[5]. 2.5g of the crushed and sieved ore was placed in a furnace at 800°C for 3 hrs. On cooling, the residue was treated with 250cm^3 of 2M KOH solution in a flask. The mixture was boiled with stirring for 2hr and filtered under vacum using a Buchner flask fitted with a glass funnel parked with glass wool. The ore that dissolved was determined by subtracting the weight of the dry residue from 2.5g. The filtrate was then treated with 9M H_2SO_4 until acid to litmus and boiled for 15 minutes to dissolve small white lumps of Al $(OH)_3$ produced. The mixture was concentrated to about 100cm^3 and allowed to cool (ice bath); then potash alum crystallised out. The supernatant liquid was decanted and the crystals recrystallised in 200cm^3 of water. A second crop of crystals was obtained by concentrating and cooling the decanted liquid. The combined crystals were recrystallised from absolute ethanol, dried in the oven at 75°C to obtain crystals with m.pt. 91-93°C. The yield was calculated and the resullts are presented in Table 1.

Analyses of samples

Means of triplicate determinations were taken for all the analyses reported. Potassium was determined by a standard procedure[6&7] on a Corning 400/4246 flame emission spectrophotometer. The pH determination was carried out by the electrometric method with the use of a glass electrode on a Phillips PW9718 pH meter, following a standard procedure[6]. Assay for $KAl(SO_4)_2.12H_2O$ was carried out by a literature procedure[8]. Sulphate and water of crystallisation were determined based on literature procedures[6]. Sulphate content of the potash alums was determined by back titrimetry with EDTA following a literature procedure[7] while water of crystallisation was determined also by titrimetry, using Karl Fisher procedure[7]. Results are included in Table 2.

Assay[8] for $KAl(SO_4)_2.12H_2O$

2g of potash alum was dissolved in 25cm^3 of water, followed by the addition of 50cm^3 of 0.1M EDTA and 10cm^3 of ammonium acetate-acetic acid buffer solution and the mixture heated on a steam bath for 20 minutes. The mixture was cooled and 85cm^3 of absolute methylated spirit added followed by 3cm^3 0.025% solution of dithizone in absolute methylated spirit. The excess EDTA was titrated with 0.1M zinc chloride solution until the colour changed from blue to reddish-purple. 1cm^3 of 0.1M EDTA = 0.04744g $AlK(SO_4)_2.12H_2O$. The results are presented in Table 2.

Table 2: Analysis of alum samples from indicated ores

Al ore	Assay (%)	SO_4^{2-} (%)	Al^{3+} (%)	K^+ (%)	H_2O (%)	**pH (units)**
Bauxite	98.68	39.98	5.32	7.98	44.97	3.3
kaolin	98.85	39.58	5.30	7.92	44.98	3.2
muscovite	98.76	99.56	5.26	7.94	45.00	3.3
beryl	97.81	39.58	5.31	7.93	44.99	3.3
feldspar	98.90	39.62	5.31	8.00	44.97	3.2
commercial alum	98.90	40.20	5.49	8.10	45.19	2.8

Aluminium content of potash alums

4.74g crystals of the potash allum was dissolved in 1 litre of water to give a 0.01M solution. The solution was titrated against standardized 0.01M EDTA solution with eriochrome black T as indicator. An additional 0.4cm^3 of EDTA solution was added after each end point, and for each titration NH_4OH was used in making the solution alkaline.

RESULTS AND DISCUSSION

Table 1 gives the aluminium content of each ore, solubility of the ore in water and respective yields of potash alum from the indicated ore. The results seem to indicate that the higher the aluminium content of the ore, the higher will be the yielld of potash alum, except that the yields are not exactly proportional. The aluminium content of bauxite and kaolin differ by about ten percent; this difference is however not reflected in the yield of potash alum from the two ores. Infact, both ores can be said to have approximately the same yields of potash alum while feldspar and beryl also have the same yields inspite of their aluminium content difference.

The solubility pattern of the ores is indicative of how readily accessible the ore is with respect to its conversion to potash alum. Analysed for the parameters % aluminium, % potassium, % sulphate, % water of crystallisation, pH, and assay ($KAl(SO_4)_2.12H_2O$), the potash alum crystals obtained from the ores compared closely with those of commercially available potash alum as shown in Table 2.

The assay for $KAl(SO_4)_2.12H_2O$ in the prepared potash alum falls within the range 97.81-98.90% while that of the commercial alum is 98.98% (Table 2). Though inferior to that recommended for analar reagent grade potash alums (99.5%)[8], this range is satisfactory for technical grade potash alums. The pH of the prepared potash alums is 3.2-3.3 against 2.8 for commercial potash alum, while the recommended value for analar reagent grade is 3.0[8].

CONCLUSION

The ores obtained in Bauchi and Plateau states are suitable raw materials for producing high quality potash alum using a simple existing procedure like the one applied in this work.

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ACKNOWLEDGEMENT

We are grateful to the Geology Programme, Abubakar Tafawa Balewa University for assistance with mineral ore identification.

received 3/10/96
accepted 18/6/97