Production of Water Treatment Alum Using Ash and Aluminium Scraps

*[1]Birnin-Yauri U. A.; [1]Adetunji S. O. and [2]Alhaji, S. M.

[1]Dept. of Applied Chemistry, Usmanu Danfodiyo University, Sokoto. Nigeria

[2]Department of Chemistry, Sokoto State University, Sokoto, Nigeria

Abstract: *In this research, millet stalk, banana leaves and Prosopis africana (kirya) were burnt into ash. The alkaline leachate from the ash (lye) was mixed with sulphuric acid solution of aluminium scraps. The potassium alum ($KAlSO_4.12H_2O$) yield from millet stalk, banana leaves and Prosopis africana firewood had the yields of 51.95%, 49.36% and 29.83% respectively. Characterization of the synthesized alums confirmed the presence of Al:K:SO_4:H_2O in 1:1:12 mole ratio which corresponded with the theoretical mole ratio in potash alum. Optimum alum dosage of 150 mg/l of the three synthesized alums reduced the turbidity level of raw water by 99% with the pH changing from 6.80 in raw water to an average of 6.20 in the water treated with the alums. Synthesis of potash alum from such agrowastes and aluminium scrap is an interesting way of cleansing the environment and satisfying the demand of water industry for potash alum and an easy DIY process in the rural areas.*

Keywords: *Alum, coagulants, flocculants, turbidity, potash, lye*

INTRODUCTION

Water purification is an essential measure taken to ensure drinking water is safe to the health and welfare of a community and to ensure water quality meets drinking water standard (Casey, 1997; USEPA, 2016). Special considerations are placed on the softening of hard water, lowering of turbidity, removal of suspended solid and elimination of pathogens (Folkard, 1995). The efficiency of turbidity removal has been achieved by processes called coagulation and flocculation. (Bustanafruza *et. al* 2013). These are the processes applied to remove muddy or clayey particles dispersed in the water, suspended solid, microorganisms and minerals.

Coagulation is a chemical process by which destabilization of suspended particles takes place i.e., negatively charged colloidal particles in the water are neutralized and the cluster of colloidal particles come together into lumps or floc (Bahmann, 2014). Aggregation of small flocs into larger flocs is called flocculation. Coagulation and flocculation aid quick sedimentation and efficient filtration to get clean water (Hubbard, 2004; Bahmann, 2014). One of the earliest and still efficient coagulants is potash alum (potassium aluminium sulphate); it has been a standard raw material for water treatment in industry because of its ability to form multi charged polynuclear complexes with enhanced adsorption characteristics (Gregory and Duan, 2001).

Production of alum using chemical substances makes the cost of purchase a little bit higher especially where the raw materials are imported. The alternative way is to look for cheap sources so as to make the production easy and less expensive so that the cost of municipal water supply will be reduced and alum will be available at affordable price (Birnin-Yauri and Musa 2014). Synthesis of potash alum from aluminum scraps, ash of banana leaves, millet stalks and ash from the fire wood (hard wood) could solve problem of water purification and reduce the challenges of solid waste management. (Birnin-Yauri and Musa, 2014; Ekere *et* al., 2014).

EXPERIMENTAL

All the chemical reagents used were of analytical grade (A.R.). The water was doubly distilled and tightly capped when hot and allowed to cool before usage.

Leaching of Ash (Production of Lye)

The ash was collected and boiled for one hour in an earthen ware pot. It was allowed to cool to room temperature and then poured into a plastic bowl and left for 24 hours for maximum extraction. Activated charcoal was packed into a plastic bowl perforated at the base and the ash suspension was poured into the bowl. The bowl was placed on a plastic bucket as a receiver and water slowly added onto the suspension. The suspension was leached slowly. The activated charcoal was used to adsorb of some unwanted organic matters (Adejumo *et. al.,* 2016 and Babayemi *et al*., 2011). The lye obtained was filtered to remove any particles that may have leached out with the lye. The solution was then evaporated to dryness by heating at a low temperature.

Preparation of Alum

The method used was as explained by Birnin-Yauri and Musa, (2014); Ekere *et al*., (2014) and Liew *et al*. (2016). The aluminium beverage cans were cut-opened with scissors and flattened. The paint and the coating inside and outside the cans were scrubbed off using sand paper. The scraps were rinsed with distilled water, dried and cut into small strips. Then about 5.0g of clean aluminium strips were soaked into 80cm^3 of 8M H_2SO_4 in a 5-litre beaker. The mixture was stirred rigorously until the bubbles of hydrogen gas ceased. $2Al + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 3H_2$.

The solution was filtered. Each of the lye extracted was added to the colorless filtrate in a beaker and stirred. Initially a thick white gelatinous precipitate of $Al(OH)_3$ was formed but as few drops of H_2SO_4 were added the precipitate disappeared. The solution was boiled to evaporate excess water and then placed in an ice bath for five hours for the crystallization of alum. When the crystals were delayed to form the wall of the flask was scratched with stirring rod to aid crystallization. $Al_2(SO_4)_3 + 6KOH \rightarrow 2Al(OH)_3 + 3K_2SO_4$

$K_2SO_4 + Al_2(SO_4)_3 + 24H_2O \rightarrow 2\,[KAl(SO_4)_2.12H_2O]$

While the alum crystal was forming, vacuum filtration system (Buchner funnel) was set up. When a good crop of alum crystals has formed, it was filtered from the cold solution, transferring as much of the solid product as possible to the funnel. The remaining crystals from the beaker were rinsed into the funnel with chilled 50% alcohol/water solution. This was repeated several times to completely remove any excess water or sulphuric acid. The crystals were allowed to dry for five minutes on the filter paper with full suction. The vacuum pump was turned off and the alum crystals were carefully poured into a 500cm^3 beakers, dissolved with distilled water for recrystallisation to obtain pure alum. The alum crystals formed were dried and weighed and the percent yield was calculated.

Characterization of the Synthesized Alum Crystals

Sulphate contents were determined by turbidimetry (APHA, 1998)

The metallic oxides in the alums were determined using Energy Dispersive x-ray Fluorescence (X-Supreme 8000) (Kishimura *et al*., 2014 and Chingodo *et al*., 2015).

Coagulating Efficiency of the Synthesized Alum (Jar Test)

Jar test method was employed to determine the optimum dosage and performance of synthesized potash alum in the water treatment. The method adopted was as explained in APHA (2012)

PHYSICOCHMEMICAL PARAMETERS OF WATER

pH Measurement

The calibrated pH meter was and dipped into sample solution and kept until stable pH reading was obtained.

Turbidity Measurement

The sample cell was rinsed with distilled water and filled with the sample solution to the volume. The cell was inserted into a pre-calibrated turbidity meter and the turbidity reading from the scale was noted for each sample.

RESULTS AND DISCUSSION

The results of the percentage composition the mole proportion of the constituents of the synthesized alums are shown in the Table 1.

Table 1 Chemical Composition of the Synthesized Alums (% wt).

Sample	Aluminium	Potassium	Sulphate	Water	Mole Ratio Al : K : SO_4 : H_2O
M_A	3.75	6.20	30.82	30.58	1.00 : 1.14 : 2.31 : 12.22
B_A	3.10	5.66	26.21	24.63	1.00 : 1.26 : 2.38 : 11.93
P_A	3.96	4.85	28.03	27.46	1.18 : 1.00 : 2.35 : 12.28

As shown in Table 1, the chemical compositions of the prepared alums were in agreement with the theoritical mole propotion of potash alum i,e. $Al:K:SO_4:H_2O \sim 1:1:2:12$, which is similar to the combination ratio of the theoretical alum. The formula of the alum produced can be written as $KAl(SO_4)_2.12H_2O$. The Al^{3+} ions become surrounded by 6 water molecules, arranged around the aluminum ion in xyz axis. Potassium ions are also surrounded by 6 water molecules. The formula indicates that these 12 water molecules are intimately bonded in the crystal.

Oxide Contents

Table 2 *Oxide compositions of aluminium cans and synthesized alums (wt %)*

Oxide	Al scraps	M_A	B_A	P_A
Na_2O	n.d.	1.697	1.445	1.489
Al_2O_3	98.004	16.153	16.047	16.392
SiO_2	n.d.	6.094	7.316	6.081
P_2O_5	n.d.	0.703	0.530	0.518
SO_3	n.d.	58.600	55.494	56.470
K_2O	n.d.	18.004	17.749	17.70
CaO	n.d.	0.505	0.660	0.604
MgO	1.007	0.779	0.637	0.626
Cr_2O_3	0.067	0.002	0.003	0.002
Mn_2O_3	0.045	0.002	0.003	0.003
Fe_2O_3	0.834	0.010	0.022	0.021
ZnO	0.001	0.030	n.d	n.d
TiO_2	0.002	n.d	0.003	0.006
% purity	98.004	92.837	89.292	90.556

n.d = not detected

X-ray fluorescence results of the synthesized alums as shown in Table 2 and it shows that the dominant oxides present in the alums are Al_2O_3, K_2O and SO_3. The SO_3 was found to be in preponderance in all the samples. Considering the Stoichiometry equation of the reaction of Al_2O_3, K_2O and SO_3 in aqueous solution to give potash alum $KAl(SO_4).12H_2O$. This is represented in the equation below:

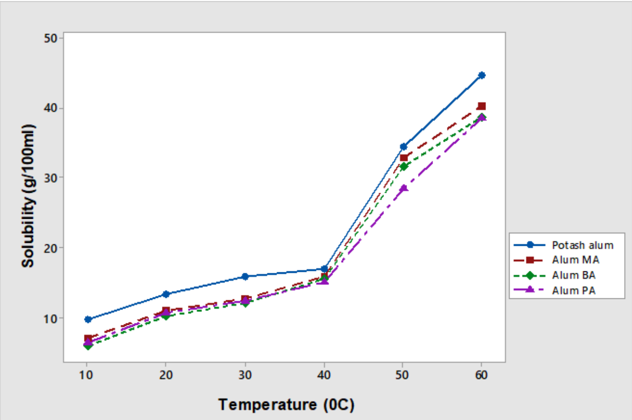
$$Al_2O_3 + K_2O + 4SO_3 + aq. \rightarrow 2KAl(SO_4)_2.12H_2O$$

The presence of other oxides apart from Al_2O_3, K_2O and SO_3 are impurities from the raw materials used in the synthesis. Na_2O, SiO_2, P_2O_3, MgO and CaO could be from the ash extracts. Similarly the other impurities: MgO, Cr_2O_3, Mn_2O_3, Fe_2O_3, ZnO and TiO_2 are the impurities from the aluminium scraps. This is asserted by the result of the X-ray fluorescence as shown in Table 2. The percentage purity of the alum M_A, B_A and P_A based on the sum of percentages of Al_2O_3, K_2O and SO_3 composition are 92.837%, 89.292% and 90.556% respectively.

Temperature Effects on the Solubility of the Potash Alums

The effect of temperature on the soulbility of the alums are shown in tFigure 1 .

Figure 1 Temperature Effects on the Solubility of the Alums



The plot indicates gradual increase in solubility of the alum from ambient temperature to about 42°C. There is rapid increase in solubility from 42°C to 60°C. The phenomenon could be explained that at temperatures below 40°C, the dissolution of alum is:

$$KAl(SO4)_2.12H_2O + aq. \rightarrow [Al(H_2O)_5(OH)_2]^+ + 2SO_4^{2-} + K^+ + 2H_3O^+$$

However, with the elevation of temperature above 40°C, the released H_3O^+ will, in accordance to Le Chatlier's principle, reverse the self ionization of water

$2H_2O \rightarrow H_3O^+ + OH^-$. This means that once $H3O^+$ ions are released from the hydrolysis of the alum, they are neutralized by OH^- released form self ionization of water: $H_3O^+ + OH^- \rightarrow 2H_2O$. This neutralization reaction enhanced the solubility of the alum.

Efficiency of Synthesized Alums on Turbidity Removal

The results of effects of alum dosage on percentage turbidity reduction of the commercial alums and the synthesized alums are shown in Figure 3

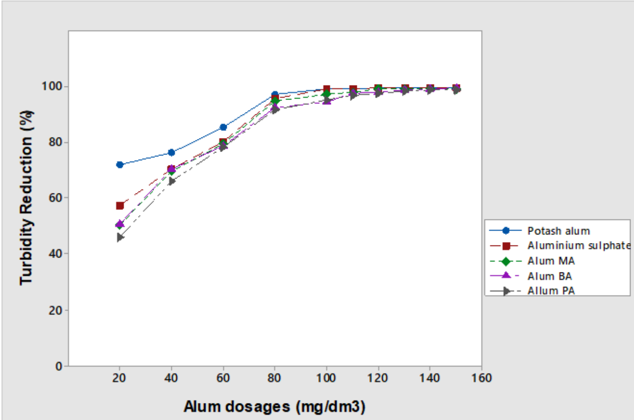


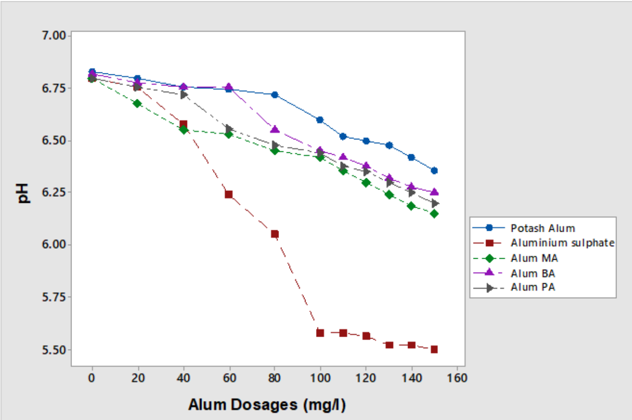
Figure 2 Effect of Alum Dosage on Percentage Turbidity Reduction

Figure 2 shows the effects of the alum dosage on turbidity removal of the raw water. Over 95% turbidity removal was achieved with maximum dosage of $150mg/dm^3$. Turbidity removal of 99.24% 99.37% and 98.72% (Fig. 1) with residual turbidity of 17, 23 and 36 NTU for synthesized alums M_A, B_A and P_A were respectively achieved with maximum dosage of 150 mg/dm^3 of the alums. These values are similar to that of commercial potash alum (99.67%, 9NTU),) All the alum samples showed increase in turbidity removal with increase in alum dosage. The increase was rapid at lower dosage up to $80mg/dm^3$, this agrees with the report of Gregoire and Wenquian, (2007) and Ekere *et al.*, (2014) that dosage of 80-120mg/l of potash alum removed 90-92% of water turbidity. This also indicates that the synthesized alums are good coagulating agents and are effective in water treatment. The coagulating property is due to the presence of large cations:

$KAl(SO_4)_2.12H_2O + aq. \rightarrow [Al(OH)_6]^{3+} + 2SO_4^{2-} + K^+$. Then the cation $[Al(OH)_6]^{3+}$ further hydrolyses in water: $[Al(OH)_6]^{3+} \rightarrow Al(H_2O)_4(OH)_2]^+ + 2H^+$.

The large positively charged ions neutralize the negatively charged clayey particles present in the water. This neutralization reaction, (coagulation) causes destabilization of suspended and colloidal particles, adsorb natural organic matter (NOM) to particles and create flocs of particles that enmesh other particles which then settle at the bottom of the container. (Gregoire and Weiqian 2007).

Fig. 3 Effect of dosage of Synthesized Alums on pH of the Water



The pH of the water after dosage of different amount of alum was represented in Figure 3. All alum samples showed decrease in pH as the concentration of each dosage increased. The rate at which pH reduce on addition of aluminium sulphate is more than that of the potash alums. Comparing the pH values of the water on dosage of 150mg/l of synthesized alums with commercial potash; alum M_A gave pH of 6.15, alum B_A and alum P_A gave values of 6.25 and 6.20 respectively. Alum B_A gave the highest pH value, these values are closed to the value giving by commercial potash alum which is 6.36 When considered their pH with turbidity removal at dosage of 100mg/l the alum M_A (97.33%, 6.42) alum B_A (94.33%, 6.45) and alum P_A (95.24%, 6.44) and also at dosage of 80mg/l the alum M_A (94.66%, 6.45) alum B_A (92.65%, 6.55) and alum P_A (91.48%, 6.48). The percentage turbidity reduction at this dosage could be due to mixing time and flocculation time which. Increasing mixing time and flocculation time will increase turbidity removal so as to meet the standard and acceptable pH (6.5 - 7.5) of drinking water (SON, 2011, WHO 2017 and APHA 2012). Comparing the pH values of the synthesized alums with those of the aluminium sulphate used in water board, the pH of the water treated with synthesized alum are higher i.e. less acidic than alum treated with aluminium sulphate even with lower dosage like 80mg/l aluminium sulphate gave pH of 6.05.

CONCLUSION

Potassium alum crystals have been a sought after chemical because of its usefulness in water purification and other industrial application, new discoveries of its usefulness has increase the demand. The results obtained from this studies revealed that preparation of potash alum from local waste materials is feasible, The alums synthesized are very effective in coagulating the suspend and colloid particles in the water which means they are active in reducing turbidity of water and adjust the pH to less acidic level. Although, all the alums are active, but in terms of the percentage purity alum from millet extract is the most efficient one. Synthesis of alum from waste material is more efficient and more economical.

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