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Production and Characterization of Water Treatment Coagulant from locally sourced Kaolin Clays

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ABSTRACT: Base on high cost in coagulant for treating both domestic and industrial water in the recent time. There is need to identify cheaper and efficient methods of removing contaminant as the demand for clean water increases. A study was carried out to investigate the use of locally sourced Kaolinite clay from Okefomo Agbarigidoma, Ilorin south Local Government of Kwara state, for the production of aluminum sulphate using sulphuric acid (H_2SO_4) solution. The clay sample was beneficiated and calcined at 700^oC for 7 h to obtain meta-kaolin, then later leached using sulphuric acid. The effect of leaching temperature (25-100^oC),period of activation (10-120 minutes),acid concentration(1-6M),particle sizes(50-200µm),acid to clay weight ratio on the leaching were investigated. The optimum leaching conditions for the calcined Kaolin clay were found to be particle size 100µm, acid concentration 5M, leaching temperature 110^oC and leaching time of 90 min. Under optimized condition 68.75% (*w/w*) aluminum sulphate (alum) was extracted. The result showed that sulphuric acid could be used on a large scale to extract alum from Kaolin clay. The extract alum showed similar structure and physical characteristics compared with commercial alum. A dosage of 38mg/L of the extract alum show effective coagulant properties with a great potential of treating Industrial water.

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The high increase in demand for portable water for domestic and industrial uses cannot be over emphasized (Abdulsalam *et al.,* 2013). Treatment of raw water to remove unwanted materials is a long practice and the commonly used chemical to achieve this purpose is Aluminum sulphate, otherwise called Alum (McCurdy *et al*., 2004; Renault *et al*., 2008). Alum is found naturally in the earth crust as a double salt with formula, $Al_2(SO_4)_3.14\text{-}27H_2O$. This compound is a coagulant that is commonly used in water treatment plants for safe domestic and industrial water (Koohestanian *et al*., 2008; Ismail, 2010; Arnoldsson *et al.,* 2011; Apostol et al., 2011). Traditionally, Alum is extracted from bauxite an alumino silicate mineral through bayer process (Pehliran et *al*., 2012). The bayer process involves grinding of bauxite mineral and pressure leaching with an alkali, such as sodium hydroxide to obtain an aluminate solution. Pure aluminum hydroxide is then precipitated from aluminate solution by seeding (Chingodo *et al*., 2015). Apparently, bauxite is often completely absent in known commercial quantity in most developing countries such as Nigeria (Aderemi *et al.,* 2009). Apart from bauxite usage, the use of kaolin for the production of aluminum sulphate has received great attention in recent time (Az-zahrani and Abduk-majid, 2004; Hosseini *et al*., 2011; Pauwska and keshir.2002). Nigeria was reported to have estimated two (2) billion metric tons of kaolin deposit scattered in different part of the country which needs to be properly exploit for economic and technological development of the country (Ekosse, 2010).

Kaolin is another aluminosilicate mineral which is found among different type of clays. It has a high Aluminum content compare to other type s of clay minerals in its category, which include; smectite, illite and chlorites (Abdulsalam *et al.* 2013; Madejoval, 2003; Murray, 2007). Aluminum has been extracted from kaolin clays using different mineral acids (Hosseini, 2004). However, the use of hydrochloric acid for leaching alumina compare to other mineral acids offer many advantages which ranges from; the ease of filtration of slurries, ease of iron removal , and the insolubility of titanium dioxide present in many clays (Dodson *et al.,* 1939). The problem of severe corrosion of equipment which is the only setback of using hydrochloric acid has been solved to a large extent. Therefore both hydrochloric acid and sulphuric acid extract approximately the same amount of alumina from same quantity of clay (Raghavan and Gajam, 1985; Schoenborn and Hofman, 1979).

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In addition, other aluminosilicate form such as fly ash can be adopted in the extraction of alumina. The use of alkaline treatment on desilicate coal fly ash was reported to extract 89-90% alumina (Bai *et al*., 2010; Su *et al*., 2011). Apparently, the use of both alkali and acid dissolution process on fly ash suffer high energy consumption drawback that are not economical at industrial scale level (Bai *et al*., 2010).

Currently, all water treatment plant across Nigeria import aluminum sulphate from other developed countries like, France, Newzealand, and Egypt, which attract huge amount in dollars. Base on this aforementioned, there is need to identify local source of coagulant to reduce or totally eliminate import cost. In this study, an investigation was carried out on the production of aluminum sulphate from locally sourced kaolin clay from Okefomo Agbarigidomo axis of Ilorin South Local Government Area of Kwara State, Nigeria. This study focused on production of water coagulant (alum) from locally abundant kaolin clay using sulphuric acid. All the optimizing conditions for project scale-up would be established and the efficiency of the product would be tested on industrial wastewater treatment practice.

MATERIAL AND METHODS

Study area: The study area is Okefomo Agbarigidoma, Ilorin, Kwara State, Nigeria with GPS reading N10″05″42.1″NC007″26″73″

*Sample collection***:** The clay sample selected for this study was kaolinite clay, collected from Okefomo Agbarigidoma axis, Ilorin South Local Government Area of Kwara state, Nigeria. The commercial aluminum sulphate used as a control and sulphuric acid used in this study are analytical grades and double distilled water was used to prepare all aqueous solutions.

*Sample treatment***:** *Beneficiation of raw sample***:** Beneficiation process involves the pretreatment of raw clay to remove impurities. About 200g of the clay sample was crushed, ground and washed with distilled water to remove soluble impurities. Already washed sample was subsequently suspended in water to get rid of intermediate to coarse associated mineral particles. The fine kaolin clay suspension was allowed to stand for six days to allow proper separation of the solid and liquid into two layers under the action of gravity. The upper layer was decanted and the solid layer was further dewatered to a thick mass in a clay bed under the action of pressure. The thick mass of clay resulted was dried overnight in an oven at temperature 80°Cand then crushed and ball milled into fine powder (Wu *et al*.,2012; Abdulsalam *et al*., 2013).

Sample Preparation and Activation: The clay samples were grounded using a ball mill to particle size 300µm. the ground clay sample was place on the sieved, and then mechanically shake for 5 min. The oversize was further ground followed by sieving on the same sieve. The procedure was repeated till the entire clay sample pass through the sieve. 25g of grounded clay sample of different particle sizes (20-200 µm) were put in the crucible and subjected to high temperature in a muffle furnace(carbolite-LP 124) for activation process (Bai *et al.,* 2010; Park, 2000). The calcination temperatures were varied between 500 and 900 for a period of 1.5 hours.

Sample / Product characterization: The elemental composition of powdered clay and the synthesized product was determined using x-ray fluorescence (XRF) machine model: MINIPAL-LX123 and the metal organic bonding of the kaolin clay surface and the product were significantly determined by Fourier transform infrared spectroscopy (FTIR) [Model-JENWAY 430] analysis respectively. A 1.0g clay samples was calcinated at 900^{o}C in a muffle furnace to determine the Loss on Ignition (LOI) and to reduce the impurities present.

*Production of alum***:** All leaching experiment were performed in triplicate as detailed in (Chigodo *et al*.,2015; Ilic, 2010; Park *et al.,* 1992). The calcinated clay sample 5g of different sizes (20-200 µm) were leached in a 250 mL glass reactor equipped with magnetic stirrer using wide ranging concentration of sulphuric acid (1-6M) over varying duration (10-120 min),at different temperature between 25-110^{o}C and various acid-clay ratio(4:1 to 7:1) *w/w* under reflux and constant stirring (120 rpm). At the end of leaching process the resulting residues was filtered to separate undissolved materials. The residue was washed thrice with 10 mL portion of distilled water in order to remove excess acid and the final product was dried and characterized using FTIR technique. The separating funnel was used to add slowly 100 mL of ethanol into the acid leach liquor and continuous stirring to precipitate aluminum sulphate. After precipitation, the precipitate was filtered using whatman filter paper and dried in the oven at temperature 90^{o}C for 1 hour.

*Comparative Evaluation between Commercial Alum and Locally Produced Coagulant:*The product form and the commercially purchased aluminum sulphate (Alum) were used in coagulation test. Four beakers (250 mL each) were filled with 100mL of Industrial wastewater from Tuyil Pharmaceuticals Industry, Ilorin, Nigeria. Different dosages (10-40 ppm) of the extracted products and commercially purchased aluminum sulphate were separately added to the wastewater. The beakers were stirred continuously for 10 min before allow to settle for 20 min. The

coagulated particles was filter and the Turbidity, conductivity, total dissolved solid and the pH of the filtrate were determined using detail procedure by Abdulsalam *et al.,*(2013).

RESULTS AND DISCUSSION

Analysis of raw clay sample by XRF: The chemical composition of the clay sample used in this study is depicted in table 1. Alumina and silica are the major component of the clay. The percentage composition of the alumina (25.34%) was higher than the value previously reported (Chigodo *et al.,* 2015; Wu *etal*., 2011). In some other studies, clay of alumina content 29.40% was used to achieve an extraction of 32% $Al_2(SO_4)_3$ (Anderson and Prentice, 1999). Lori *et al*., (2004) reported sample containing approximately 66% SiO_2. The higher in silica content tends to form greater part of the insoluble residues after acid treatment (Chigodo *et al*., 2015). Other metallic and non-metallic oxide such as sodium, manganese, phosphorous and sulphur occur in trace below 1.70%. The calculated Loss on Ignition (LOI) value was 7.97%. The calculated value is attributed to bond hydroxyl ion in calcined kaolin clay (Greenwood and Earnshow, 1997)

FTIR Result of Clay sample before and after acid treatment

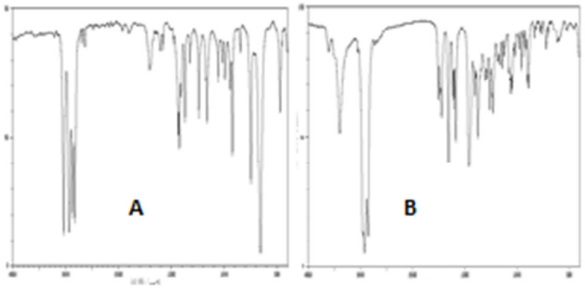

Fig 2: FTIR spectra of Okefomo Agbarigidoma kaolin clay (a) before leaching (b) after leaching.

Table 1: Elemental composition of raw kaolin clay sample

Compounds	Composition (*wt %*)
Al_2O_3	25.36
CaO	1.29
SiO_2	54.37
FeO_3	5.75
K_2O	1.67
MgO	1.87
Mn_2O_3	1.67
Na_2O	0.98
P_2O_5	0.13
SO_3	0.35
LOI	7.97

The FTIR spectra of clay sample used for this study before and after acid leaching are shown in figure 2a and b.

Figure 2a shows a spectrum band at 3210 $cm^{-1}$and 3941cm^{-1}which are assign to hydroxyl stretch. The band is attributed to the water hydroxyl group situated at kaolin surface. The peak can be traced to the hydrogen proton and oxygen atoms coordination aluminum ion in the octahedral structure layer (Chigodo *et al.,* 2015). The Si-O-Si band appeared at 457,463 and 473 cm^{-1} this is attributed to silica phase of the clay. After leaching, the OH stretching of inner surface hydroxyl group in Al-OH in octahedral layer of kaolin bands was observed at 3608 and 3396 cm^{-1} respectively. Appearance of new bands at 932, and 936 cm^{-1} was suspected to be deformation of hydroxyl group in the bonding of Al-Al-OH octahedral sheet (Az-zahrani and Abdul-majid, 2004).

*Effect of calcination temperature***:** Calcinations is an important parameter when investigating the extraction of aluminum from kaolin clay. The process thermally activates the kaolin to more reactive form (Greenwood and Earnshow, 1997). The effect of calcinations temperature on the extraction of aluminum sulphate is shown is figure

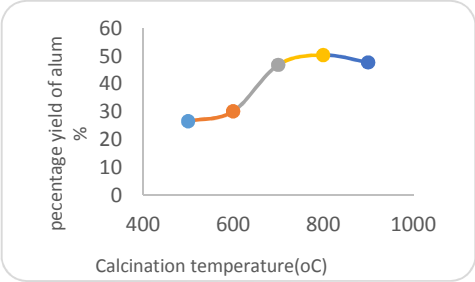

Fig 2: Effect of calcination temperature on the extraction of alum (time: 1.5 h, particle size: 100 µm)

The amount of alumina extracted increase with increasing calcinations temperature from 24.5% at 500°C to maximum of 50.2% at 800°C above 800°C the quantity of alumina decreased. Thermal treatment lead to loss of water molecules within the kaolin clay structure, which indicates that above 800°C, total dehydration of the kaolin clay occurs resulting in phase transformation and disruption of kaolin to meta-kaolin amorphous solid that is less prone to acid attack (Chigodo *et al.,* 2015; Panda *et al.,*2010). This process can be represented stochiometrically as:

$3SiAl_4O_{14} \rightarrow 2SiAl_6O_{13} + SiO_2$ (1)

Similar works (Lori et al., 2007; Ajemba and Onukwuli, 2012) showed that calcined clay at 750°C exhibited more alumina dissolution rate to those at lower temperature. Studies reported by Ibrahim *et al*.,(2013) also gave maximum extraction at 800°C.

Effect of calcinations time: The effect of calcinations time was investigated on the yield of aluminum sulphate as presented in figure.3. The percentage of alum extracted up to 90 minutes. Beyond this 90 minutes the amount dropped by 8.79%. low calcinations time could result in insufficient thermal treatment of kaolin clay sample. Therefore, increasing the time of calcinations ensure that kaolin clay sample are adequately expose to calcination

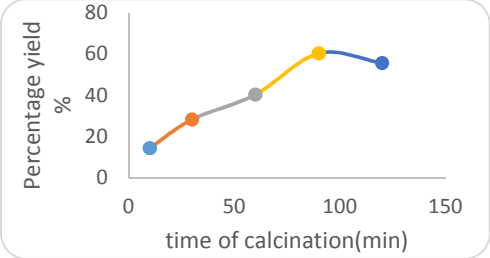

Fig 3: Effect of calcinations time on the extraction of alum (particle size: 100 µm, temperature: 800°C).

Effect of particle sizes: Figure 4 shows the effect of kaolin particle size on the quantity of alum extracted. At the beginning, the quantity of aluminum sulphate extracted increases with increasing particle size but later drop at 100 µm. The maximum extraction yield of 40.25% was observed at this point. Above 100 µm particle size, a decrease in the quantity of aluminum sulphate extracted was observed. The reason could be attributed to decrease in clay surface area expose to acid attack. The particle size of 100 µm were selected for further leaching experiment.

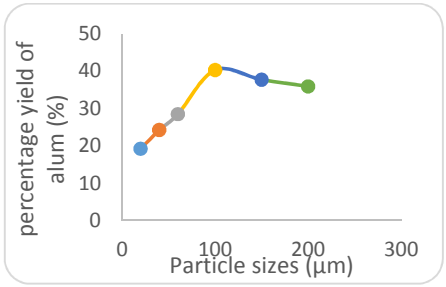

Fig 4: Effect of particle size on the extraction of alum (leaching time: 1.5 h, temperature: 80°C; acid concentration: 4M).

*Effect of leaching temperature***:** The yield obtained in a conventional aluminum sulphate extraction depends on the bath temperature. Figure 5 shows the effect of temperature on aluminum sulphate extraction. Increase in leaching temperature results in increasing yield of aluminum sulphate. An increased collision frequency occurred as a result of increase in kinetic energy of the kaolin acid solution due to increase in leaching time. The highest yield was obtained at temperature 120°C. Chingodo *et al*.,(2015) reported lower temperature. Other research works (Ajemba and Onukwuli, 2012; Park *et al*., 2000) concluded that kaolin leaching require high temperature.

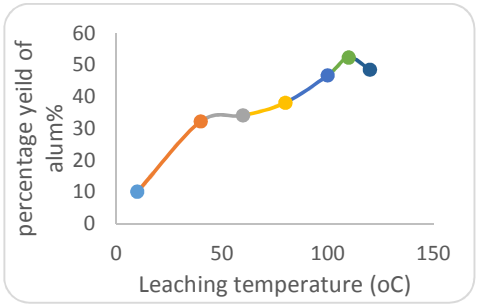

Fig 5: Effect of leaching temperature on the extraction of the alum (leaching time: 1.5 h; acid concentration: 4M; particle size: 100 µm,)

Effect of leaching time: The effect of leaching time on the extraction process is shown in figure 6. The yield of aluminum sulphate increased with increasing leaching time. Prolong leaching time allowed ample time of interaction between the acid and kaolin particles. The highest yield of 68.34% was achieved at 120 min. This result is a bit higher compare to the result reported by Numluk and Cheisea, (2014).

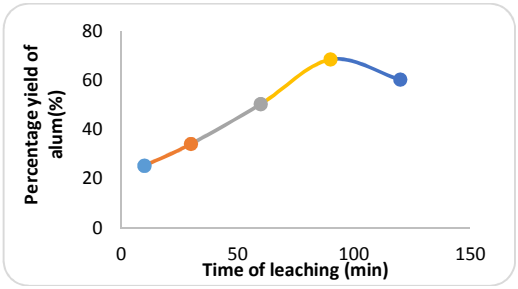

Fig 6: Effect of leaching time on the extraction of alum (acid concentration: 5M; particle size: 100 µm; temperature: 110°C)

*Effect of liquid-solid ratio***:** The effect of acid to kaolin clay ratio on aluminum sulphate extraction is presented in figure 7.

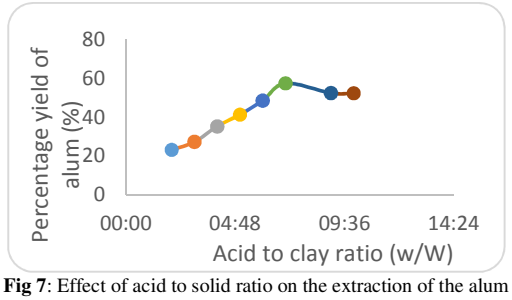

Fig 7: Effect of acid to solid ratio on the extraction of the alum (leaching time: 1.5 h; temperature: 110°C; particle size: 100µm; acid concentration: 5M)

This parameter indicates the actual amount of acid needed to be in contact with solid for optimum extraction. There was observable increase in

percentage of aluminum sulphate extracted as the acid- solid ratio increased. 57.37% yield was achieved at an acid-solid ratio of 7:1 and maintain till 9:1. Above 9:1 acid to solid ratio, the alum yield dropped to 52.31%. This might be probably due to a saturated of availability clay with hydrogen ion. Other studies showed that increasing the ratio from 4:1-10:1 increase the quantity of alum sulphate extracted (Park *et al*., 1992; Ibrahim *et al*., 2013).

*Effect of H_2SO_4concentration:*The effects of acid concentration were investigated on the extraction of aluminum sulphate as presented in Figure 8. The degree of extraction increased with increase in acid concentration. The percentage yield of alum increases from 24.1% at 1M to 68.75% at 5M. Above 5M H_2SO_4 concentration, there is decrease in aluminum sulphate extracted. An increase in acid strength increases the diffusion of H^+ ion into the octahedral layer of kaolin resulting in the dissolution of Al^{3+}. There is a collapse in the structure of the sample at a very high acid concentration which in turn lead to decrease in the Aluminum ion dissolution (Chigondo *et al*., 2015).

Comparative study of physical properties of extracted alum with commercially purchased alum: The melting point, boiling point, pH and colour of the extracted and commercially purchased aluminum sulphate are compared closely as depicted in table 2.

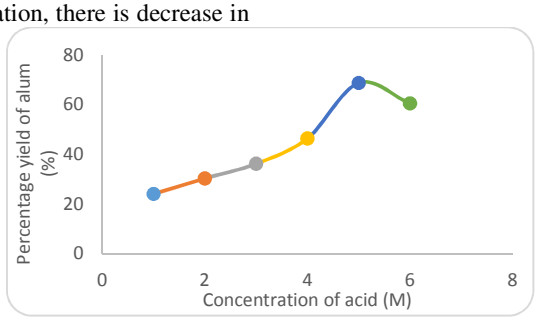

Fig 8: Effect of H_2SO_4concentration on the extraction of alum (leaching time:1.5 h; temperature:110^{o}C; particle size: 100 µm)

Table 2: Shows different in physical properties of the product and commercially purchased alum.

Property	Commercial alum	Produced alum
Appearance	White	Off-white
pH	7.3	7.20 (highest yield)
Melting point (^{o}C)	87.0	91.0
Boiling point (^{o}C)	185	194
Solubility	CD	CD

*CD: Completely dissolved at room temperature

Coagulation Test: The results obtain for the quality parameter test were presented in table 4. It can be seen that all parameters obtain for industrial wastewater indicates that it was not portable for drinking and hence treatment was required. The investigation revealed that parameter value for all the treated samples were lower than those obtained for commercial alum. In addition to this, the result obtained for the entire sample investigated fell below or within the limit specified by World Health Organization (WHO). The clear picture of this claim is summarized in table 6.

For Okefomo Agbarigidoma kaolin clay, the sample produce using 5M H_2SO_4 concentration at 110^{o}C gave the best result 68.75%. Therefore, these optimum conditions can be used for commercial production of aluminum sulphate from Okefomo Agbarigidoma kaolin clay within the limit of experimental procedure used in this investigation.

Table 3: Some Coagulant Properties of Water using Alum Produced from Okefomo Agbarigidoma Kaolin Clay Sample

Parameter	WHO values	Raw water	Commercial alum	Alum extracted at various [H_2SO_4]. 1M	2M	3M	4M	5M
pH	6.5-8.5	4.76	7.30	6.90	6.85	7.20	7.20	6.90
TDS (mg/L)	500 max.	86.30	124.00	126	130	160	126	128
Hardness(mg/L)	150 max.	60.10	61.00	63.0	50.0	53.0	56.0	56.0
Turbidity(NTU)	0-5	6.10	0.500	0.97	0.73	0 .83	0.90	0.65
Conductivity(µs/cm)	1000 max.	132.90	215.00	212	216	212	216	216
Colour(ptco)	15 max.	26.00	10.30	0.00	0.00	0.00	0.00	0.00

*Conclusion***:** Base on the results obtained from this study, it is demonstrated that aluminum sulphate can be efficiently extracted from locally sourced Okefomo Agbarigidoma kaolin clay because the product has shown great potential in removing pollutant from Industrial wastewater.

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