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Production of Poly Aluminum Chloride (PAC) from Local Material (Kaolin)

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Abstract: Poly aluminum chloride (PAC) has many applications such as water treatment, separation of slurry in industrial processes. The basic raw material for PAC production is aluminum oxide ore such as Kaolin, Bauxite, Mica and Silliminite. In this study Kaolin was used and investigated carefully. Kaolin is abundant in the Sudan. The chemical composition is $SiO_2, Al_2O_3, Fe_2O_3, TiO_2, K_2O, P_2O_5$,CaO, MgO, Na_2O, L.O.I. For the production of liquid PAC, Kaolin was activated by calcination process to produce intermediate product (alumina, Al_2O_3). Then the alumina extracted from calcined Kaolin was leached with hydrochloric acid to produce aluminum chloride. Finally base is added to aluminum chloride to produce PAC. The effects of the various parameters of production process (calcination temperature, acid concentration, residence time, ratio Kaolin-acid W/W) were investigated and the optimum conditions were found. The results showed that the optimum calcination temperature for the samples used, is 1000˚C with 3 hour residence time with alumina extract of 28.76 ppm .The optimum leaching conditions were determined and found at: Kaolin grain size, 200 meshes, Temperature, 90 ˚C, Residence Time 30 min, Acid concentration of 8N (HCL), which yielded 59.89 ppm of alum extract.

Keywords: PAC; Kaolin; Hydrochloric Acid; Calcination; Residence Time; Extract.

1. INTRODUCTION

The aluminium sulphate (Alum) is considered as the most commonly used coagulant for water treatment [2]. The polymerized forms, such as polyaluminium chloride (PAC) or poly-aluminium sulphate (PAS) have gained favorable usage in domestic and industrial applications for treating the waste water. Alum is traditionally extracted from bauxite [1]. The main raw material for PAC production is aluminum oxide ore such as Kaolin [10], bauxite. mica and silliminite [6].

Kaolin is another aluminosilicate mineral which is found among different types of clays in Sudan [3][4]. Kaolin contains a high Aluminium (Al) concentrate when compared to other types of clays such as smectite, illite, and chlorite [12]. This hydrated Aluminum Silicate mineral has different colors ranging from white to red due to presence of iron oxide [13]. Different mineral acids have been used to extract Aluminium from Kaolin [14]. One method uses the hydrochloric acid for leaching alumina. Compared to other acids the hydrochloric acid revealed several advantages such as the convenient filtration of slurries, and the perfect removal of iron and insoluble titanium dioxide present among several types of clays [15].

The main use of PAC is water treatment beside other application such as Municipal & Industrial Waste Water Treatments, Sewage Water Treatments, Separation of Slurry in Industrial Processes, Sizing in Paper and Pulp Industry, Decolorization & Decontamination of dyes in Textile Industry and in the Petroleum Oil Refining Industry[5]. Experimental results revealed that approximately, the same amount of alumina leaching extract is released by the hydrochloric and sulphuric acids [16][17].

This work aims to produce liquid PAC from local clay, such as Kaolin, since the Kaolin has high alumina content and it's Abundant in Sudan. In-addition. The country doesn't produce any type of PAC products, and the annual consumption of this product is around 300 Ton. Kaolin is found in many states, in North Kurdofan, North Darfour, AL-Gadaref, Khartoum, River Nile and Red Sea [7]. The Kaolin used in this work is from "Bunda" area at North Sudan that contains Al_2O_3. [8]. To accomplish this objective, two main points were investigated the availability of raw material and optimum PAC production conditions.

MATERIALS AND METHODS

2.1 Materials

The main raw material used to produce liquid PAC are Kaolin, Hydrochloric acid and Sodium hydroxide.

Fig.1: Kaolin Clay – Natural samples [13].

2.2 Methods

The production of liquid PAC has four steps. Firstly grinding of Kaolin sample, Calcination of Kaolin to produce an intermediate product (alumina), then reaction with hydrochloric acid to produce aluminum chloride, and the last step is reaction of aluminum chloride with a base to produce PAC.

2.2.1 Grinding

The ground clay sample was placed on the sieve (200 meshes), and mechanically shacked for 5 min. The oversizes were further grounded followed by sieving on the same sieve. The procedures were repeated till the entire clay sample passed through the sieve.

2.2.2 Calcination

The purpose of calcination is to remove moisture in the raw material lattice and destroy the crystal structure of gangue minerals [20], generate high surface activity Kaolinite, change Al_2Cl_3 into $y - Al_2Cl_3$ [9],which can react with hydrochloric acid or sulphuric acid rapidly and easily [18][19]. The leaching rate of $y - Al_2Cl_3$ can reach 90%. The process of calcined dehydration is as follows:

$$Al_2O_3.2SiO_2.2H_2O \longrightarrow Al_2O_3.2SiO_2+2H_2O \quad (1)$$

The nine samples were sieved and every sample had a weight of 15 gm. Thereafter the samples were fed to the furnace with a temperature range of (850°C to 1100°C). The calcination temperatures were (850-1000-1100)°C for all nine samples (see Table 1). The calcination times were (60-120-180) min. The optimum calcination temperature and time were determined, and the alumina ratio was determined carefully.

2.2.3 Acid Leaching

The optimum calcination temperature and time were used in the subsequent dissolution reactions for all the samples. Five grams of the calcined clay sample passing 200 mesh were leached [11]. The work used three acid concentrations which were (2-5-8) N with acid/Kaolin ratio (1:14) w/w for different periods of time (30-40-50 min) and at different leaching temperatures (50-70-90°C). A constant temperature (at a fixed shaking rate of 160 rpm) with boiling under reflux. By the end of leaching, the resulting slurry was filtered to separate undissolved materials, which were washed three times with 10 ml portions of distilled water. The filtrate and washings were continued to a constant volume of 50 ml in a volumetric flask. The resulting solutions (equation (2)), were diluted, analyzed for aluminum ion using Chlorophotometer.

$$Al_2O_3 + 6HCL \longrightarrow 2AlCl_3 + 3H_2O \quad (2)$$

2. RESULTS AND DISCUSSION

3.1 Calcination Temperature

The following Table(1), shows the resulting concentration of alumina (ppm) and the calcinated Kaolin at different temperatures and residence times.

Table 1. Calcinations effect on Kaolin.

Temperature	850 °C	1000°C	1100°C
Time	**Alumina**	**Concentration (ppm)**	
1hr	26.86	27.06	26.06
2hr	26.55	27.60	26.37
3hr	26.97	28.76	27.08

The results in Fig.2 shows that the calcination temperature was the most important parameter when preparing the extraction of the aluminum from Kaolin. The rise of the temperature leads to increase in alum extraction to an inversion temperature of 1000 °C, after which the increase of the calcination temperature terminates the extraction. The calcination temperature will normally increase the kinetic energy of the active material (Kaolin), resulting in more extraction. Fig.2 reveals that the optimum calcinations temperature is 1000°C, with 3 hour residence time. The higher concentration revealed a value of 28.76 ppm of alumina extract. The quantity of the extract increased with increasing calcination temperature from 26.06 ppm at 1100 °C to a maximum of 28.76 ppm at 1000°C. As expected the residence time increases the yield of extraction.

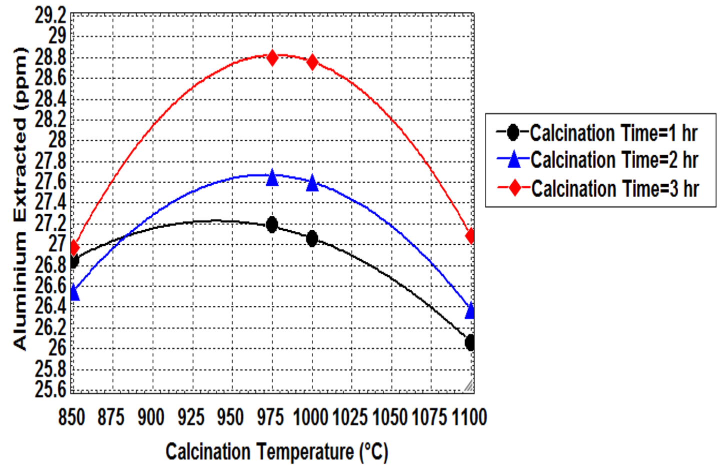

Fig.2: Effect of calcinations temperature on the extraction of the Aluminium.

Above the inversion temperature of 1000°C, a large decrease on aluminium extraction occurs due to complete dehydration of Kaolin clay, which influences the phase transformation of the material's structure, and disorderly reform metakaolin amorphous solid [11], that is less active to acid leaching.

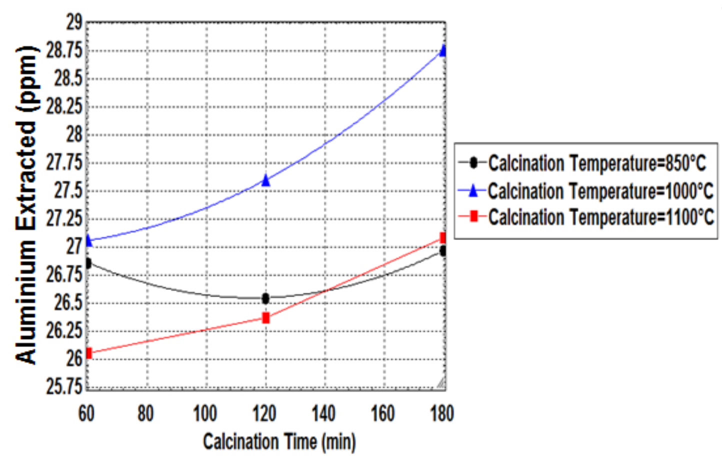

Fig.3: Effect of calcination time on the extraction of the Aluminium.

Fig.3, shows the effect of calcination time on the yield of alum. Higher calcinations time of 180 min, revealed an alum extraction of 28.76 ppm at a calcination temperature of 1000°C. The alum extracted increased up to 120 minutes. The results show that, low calcination times resulted in insufficient thermal treatment of the Kaolin sample.

3.2 Acid Leaching of Aluminium Chloride

Tables (2),(3), and (4), show the effect of (HCL) acid concentration (2-5-8)N, residence time (30-40-50) min, and leaching temperature (50-70-90) °C for the treated Kaolin samples at fixed acid/Kaolin ratio (14:1) W/W.

Table 2. Acid leaching of Kaolin, HCL Con=2N.

Exp.	HCL Con. (N)	Residence. Time (min)	Reaction Temperature (°C)	Al Con. (ppm)
1	2	30	50	37.32
2	2	40	50	49.19
3	2	50	50	40.38
4	2	30	70	38.02
5	2	40	70	42.51
6	2	50	70	40.37
7	2	30	90	44.61
8	2	40	90	45.41
9	2	50	90	42.96

Table 3. Acid leaching of Kaolin, HCL Con=5N.

Exp.	HCL Con. (N)	Residence. Time (min)	Reaction Temperature (°C)	Al Con. (ppm)
10	5	30	50	43.41
11	5	40	50	44.13
12	5	50	50	39.16
13	5	30	70	40.29
14	5	40	70	48.41
15	5	50	70	44.71
16	5	30	90	35.97
17	5	40	90	50.29
18	5	50	90	53.67

Table 4. Acid leaching of Kaolin, HCL Con=8N.

Exp.	HCL Con. (N)	Residence. Time (min)	Reaction Temperature (°C)	Al Con. (ppm)
19	8	30	50	51.23
20	8	40	50	48.59
21	8	50	50	43.68
22	8	30	70	48.21
23	8	40	70	54.32
24	8	50	70	53.71
25	8	30	90	59.89
26	8	40	90	47.81
27	8	50	90	55.67

According to the data of Table (2),(3) and (4), the effect of leaching time on the aluminum chloride extract is shown in Fig.(4),(5), and (6).The higher leaching time leads to higher extract of aluminum chloride. The results revealed that there is an optimum leaching time for each acid concentrates. The increase of the leaching time thereafter will drop the extract amount, due to excessive amount of the acid penetration. The adequate leaching time will enhance the interaction between the acid and the Kaolin molecules. Upon analyzing in Tables (2),(3), and (4), and Fig.4, it was found that the optimum condition for the aluminum chloride extract obtained at (59.89 ppm), is the leaching time of 30 minute, acid concentration of 8N (HCL), and leaching temperature of 70°C. The aluminum chloride extract was titrated by different concentrations of NaOH (1M, 3M, 5M), and the results are shown in table (5). The adequate time of acid contact and the optimum leaching temperature increases the kinetic energy of the Kaolin-acid solution due to the increase of collision frequency [12].

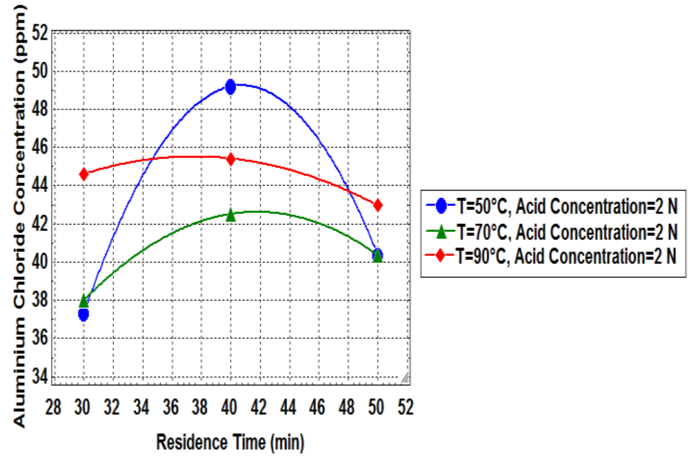

Fig.4: Effect of leaching time, temperature and acid concentration on the aluminum chloride extract.

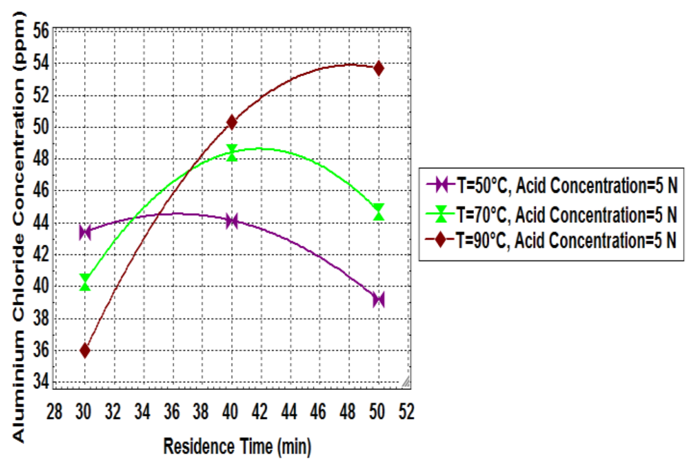

Fig.5: Effect of leaching time, temperature and acid concentration on the aluminum chloride extract.

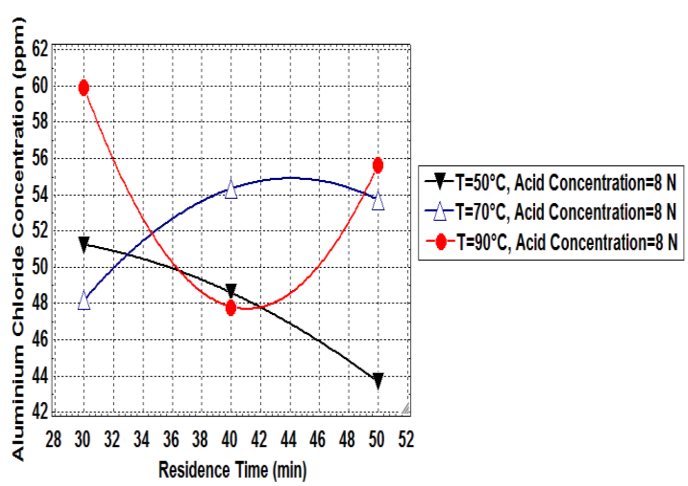

Fig.6: Effect of leaching time, temperature and acid concentration on the aluminum chloride extract.

Table 5. Titration of aluminum chloride extract.

Concentration of NaOH	Volume of Al (ml)	Volume of NaOH (ml)
1M	20	16
3M	20	8
5M	20	7.6

3.3 Jar Test Result

Different dosages of the PAC extracts were added to a sample of raw water. The raw water turbidity was 39.6 NTU after PAC was introduced, the turbidity revealed a value of 10.1 NTU.

3. CONCLUSIONS

The work has investigated an effective procedure to extract the PAC from local Kaolin. It was found that the maximum alumina extracted from a raw Kaolin clay under a calcination process with particle size 200 Tyler mesh at 1000ºC, and residence time 3 hours is 28.76 ppm. The Kaolin that contained the maximum amount of aluminium was reacted with hydrochloric acid at different parameters (acid concentration (2-5-8)N, residence time (30-40-50)min, temperature (50-70-90)˚C and fixed acid/Kaolin ratio (14:1) W/W. The results revealed an optimum aluminium extract of 59.89 ppm at leaching time of 30 minutes, acid concentration of 8N (HCL), and leaching temperature of 90°C. Finally the product was titrated by different concentrations of NaOH (1M, 3M, 5M,) to produce the final PAC.

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