International Journal of Scientific Research in Chemical Sciences **Research Paper**
Vol.7, Issue.3, pp.01-04, June (2020) **E-ISSN:** 2455-3174

Characterization and Dissolution Application of Local Clay

Samuel C. Aniaka

Dept. of Chemical Engineering, Faculty of Engineering, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria

Author's Mail Id:samuelaniaka@gmail.com, Tel: +2348037971794

Available online at: www.isroset.org

Received: 30/Apr/2020, Accepted: 03/May/2020, Online: 30/June/2020

***Abstract*—** This research was carried out to characterize and investigate the use of local clay found in Delta state for the extraction of pure alumina by the use of HCl and H_2SO_4 acids. In order to ascertain the most suitable conditions, leaching experiments were conducted under several process variables such as varying; clay to acid ratio (5:100 and 10:100), particle size (600µm and 850µm), dissolution temperature (50˚C, 100˚C and 150˚C), calcination temperature (600˚C and 700˚C) and acid concentrations (0.5M, 1M and 2M). Alumina content was determined using FS 240 Varian Atomic Absorption Spectrophotometer (AAS). Minerals contained in the clay were sorted out by using 1.54056-CuKα1 for 2Θ arranged by X-Ray power Diffraction (XRD) obtained from International Centre for Diffraction Data.

***Keywords*—** Alumina Extraction, Calcination, Inorganic Acid Leaching, X-ray Diffraction (XRD)

I. INTRODUCTION

Chemical analysis such as XRD has shown that alumina and silica are major constituents of clay, while iron, calcium, magnesium oxide and other elements are in minor quantities. The study of clay minerals via x-ray diffraction methods involve evaluation of samples that have undergone various chemical or thermal treatments [1]. However, optimal conditions can be achieved by combining different inorganic acid concentrations, particle size, clay to acid ratio and calcination temperature.

This paper focuses on the extraction of alumina from the abundant local clay found in Ubulu-uku, a community in Delta State, Nigeria. The various segments of the paper are as follows: Section I contains the introduction of major constituents of clay obtained from XRD analysis. Section II contains analysis of previous literatures and related work. Section III presents the preparation of the clay sample, calcination temperature, particle sizes and acid concentration. Section IV discusses the resulting data obtained and section V finally concludes the paper.

II. RELATED WORK

Treatment of clay with strong inorganic acid is frequently called "acid dissolution" of clays [2]. In the works of P. Komadel and J. Madejova, emphasis was placed on the clay's structural units. P. K. Igbokwe, F. L. Olebunne and M. S. Nwakaudu worked on the effect of activation parameters in which the strength of the acid, activation duration and temperature on conversion in esterification reaction were studied using a montmorillonite clay [3].

Related works on clay sciences and mineral analysis have been reviewed from time to time. Reference [4] analyzed clay samples from the mines of Katni district of Madhya for its vibrational spectra using Fourier Transfer Infrared spectroscopy (FTIR); XRF (X-Ray Fluorescence) to analyze the chemical elements present in the clay sample, and XRD patterns obtained on a powder X-ray diffractometer on a CuKα radiation of 0.04°/s scan speed.

There are two main challenges of X-ray identification pointed out by [5] to include the multiplicity of lines when several components are present and poorly defined diagrams resulting from poor crystallinity or minute size of the crystals. While the former can be resolved experimentally, the latter is inherent in the problem. Hence, simplification of X-ray diagrams becomes feasible by acid and thermal treatments.

A closely related work was done by A. A. Al-Zahrani and M. H. Abdul-Majid where the research focused on extraction of alumina from local clays by only hydrochloric acid process [6]. Other works on inorganic acid extraction of alumina from clay were done by S. Ziegenbalg and G. Discher [7], Ibid [8], J.A. Eisele [9].

III. METHODOLOGY

The clay samples were prepared for the leaching and calcination works. However, the preparation is similar for both cases. The clay was dried for 24 hours for the removal of moisture content. The clay was further reduced to smaller sizes by mechanical crushing using a mortar and pestle.

The clay was sieved into sizes of 600µm and 850µm. Some quantity of the clay was calcined while the rest were chemically activated. The clay was calcined at 600˚C and 700˚C, which was done in a Carbolite furnace under close observation. This process enhances the solubility of aluminium and further hardens the surface of the clay particles.

The calcined clay samples were leached using different concentrations (0.5M, 1M and 2M) of the two inorganic acids used in the study. The acid leaching was done for different times (30mins – 150mins), different temperatures (50˚C, 100˚C and 150˚C), different particle size and clay to acid ratio (5:100, 10:100).

For the XRD, the clay samples were sieved to a particulate size of 850µm, the acid activation was performed on the raw clay sample and the 700˚C calcined clay sample with an acid concentration of 50% for 2hours, where the clay to acid ratio was put at 50g to 100ml of acid solution. After the acid activation, the samples were washed to a neutral PH value and dried in constant temperature oven at 105˚C, after which the dried acid activated samples were grounded. XRD analysis was also carried out on the raw clay and chemically activated clay samples.

The resulting mixture was filtered to obtain a clear filtrate. The resulting solutions were diluted and analysed for alumina using Atomic Absorption Spectrophotometer. The effect of variables on alumina extraction was thus investigated.

IV. RESULTS AND DISCUSSION

XRD analysis primarily focuses on structural groups or aspects of the clay minerals. The X-ray diagrams of the raw clay sample, acid modified and thermally modified layered components are shown in figure 1 to figure 4. Several characteristic radiations are usually employed in X-ray diffraction, but CuKα is generally the most useful [10].

The XRD graphs below show the peak positions of the mineral element composition of the various clay samples. The plus signs (+) on the graph indicate the position of the active elements in the clay sample. From the scans below, there are poorly resolved diffraction peaks. This can be attributed to the small particle sizes used [11].

XRD helps to determine the mineralogical composition of the clay samples. The mineral occurrences in the clay were identified by comparing the ‘d’ values (from figure 1 to 4) to their possible minerals as obtained from International Centre for Diffraction Data (ICDD).

The full-width at half-maximum (FWHM) is obtained from the peak of the XRD pattern. It helps to calculate the crystal sizes of the clay samples.

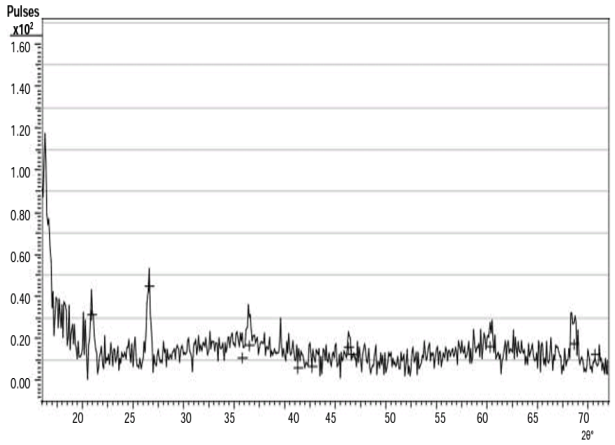

Figure 1. XRD scan for the raw clay samples (Exposure Time: 1200/1200sec. Radiation: CuKα, avg)

Table 1. The full-width at half-maximum (FWHM), d and the 2Θ values for the raw clay samples

2Θ	d	I int	I max	I rel	I corr	FWHM
20.85	4.26075	607	29.0	68.6	77.6	0.27975
26.52	3.36079	885	42.3	100.0	100.0	0.28891
35.72	2.51366	181	8.7	20.4	18.5	0.28764
36.42	2.46677	296	14.2	33.5	30.1	0.28735
41.24	2.18885	70	3.4	8.0	6.9	0.28379
41.29	2.18646	75	3.6	8.4	7.4	0.28374
42.65	2.11959	91	4.4	10.3	8.9	0.28235
46.25	1.96278	276	13.2	31.2	26.6	0.27752
46.58	1.94954	214	10.3	24.2	20.6	0.27712
60.28	1.53540	293	14.0	33.1	27.1	0.28242
68.52	1.36932	311	14.9	35.2	28.4	0.27975
70.67	1.33282	206	9.8	23.2	18.7	0.27975

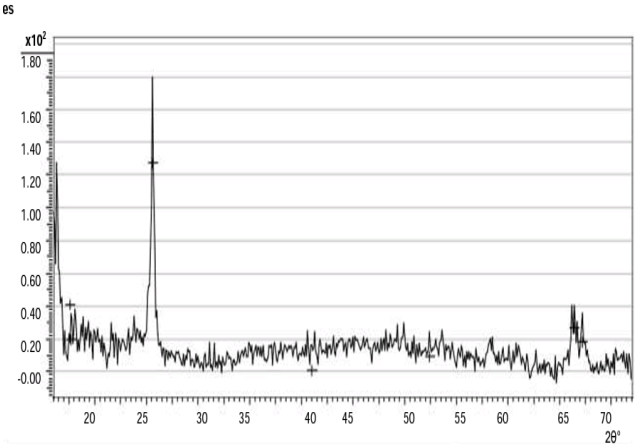

Figure 2. XRD scan for HCl acid activated clay sample (Exposure Time: 1200/1200sec. Radiation: CuKα, avg)

Table 2. The full-width at half-maximum (FWHM), d and the 2Θ values for the HCl acid activated sample

2Θ	d	I int	I max	I rel	I corr	FWHM
17.58	5.04544	902	43.2	33.4	41.9	0.27975
17.67	5.01831	466	22.3	17.2	21.5	0.27975
25.53	3.48855	2705	129.5	100.0	100.0	0.28244
40.96	2.20306	62	3.0	2.3	2.0	0.28404
52.33	1.74807	246	11.8	9.1	7.5	0.27262
66.34	1.40896	606	29.0	22.4	17.8	0.28070
67.14	1.39407	438	21.0	16.2	12.9	0.28234

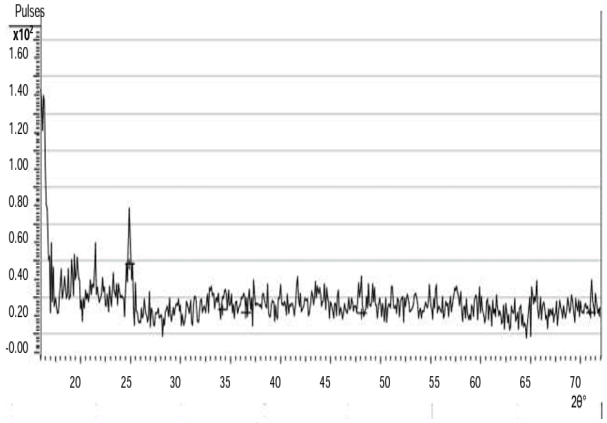

Figure 3. XRD scan of H_2SO_4 acid activated clay sample (Exposure Time: 1200/1200sec. Radiation: CuKα, avg)

Table 3. The full-width at half-maximum (FWHM), d and the 2Θ values for the H_2SO_4 sample

2Θ	d	I int	I max	I rel	I corr	FWHM
24.83	3.58519	896	42.9	100.0	100.0	0.27975
34.17	2.62424	379	18.1	42.3	37.6	0.28816
36.48	2.46276	341	16.3	38.0	33.2	0.28732
48.01	1.89502	335	16.0	37.3	30.7	0.27559
70.91	1.32894	342	16.4	38.2	29.8	0.27975

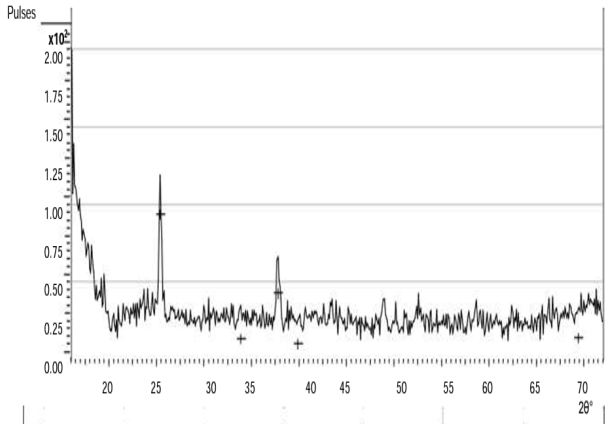

Figure 4. XRD scan of calcined clay sample (Exposure Time: 1200/1200sec. Radiation: CuKα, avg)

Table 4. The full-width at half-maximum (FWHM), d and the 2Θ values for calcined clay sample

2Θ	d	I int	I max	I rel	I corr	FWHM
25.34	3.51522	1844	88.3	100.0	100.0	0.27975
33.85	2.64809	170	8.1	9.2	8.3	0.28826
37.72	2.38479	781	37.4	42.3	36.9	0.28653
39.89	2.26000	107	5.1	5.8	5.0	0.28498
69.32	1.35556	184	8.8	10.0	7.9	0.27975

Effect of varying HCl concentration:

The figure 5 shows the conversion recorded at 100˚C from the clay sample activated with different Molarity of HCl acid. From the graph, clay activated with 0.5M acid gave a low concentration of about 3.90g/Lit. At 1.0M acid, conversion improved remarkably with a yield of 12.21g/Lit. However, when the acid strength was doubled, there was a decline in conversion with a value of 6.89g/Lit. Therefore, this result shows that conversion is optimum at 1M.

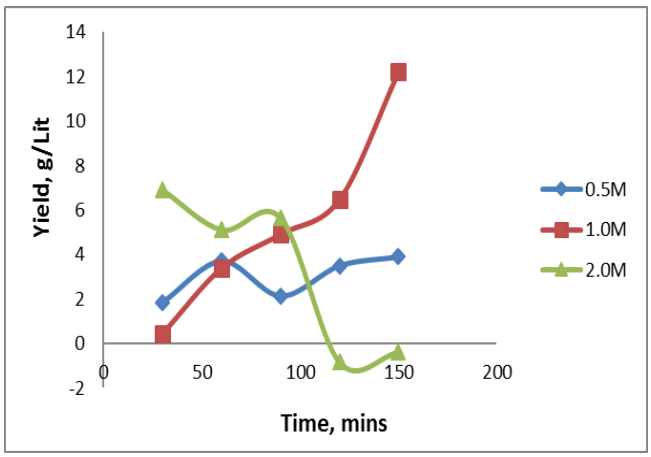

Figure 5. Effect of varying HCl concentration in the extraction of alumina

Effect of varying H_2SO_4 concentration:

From the graph, clay activated with 0.5M acid gave a low concentration of about 8.56g/Lit. At 1.0M acid, conversion improved remarkably with a yield of 14.83g/Lit. However, when the acid strength was doubled, there was a decline in conversion with a value of 11.70g/Lit. Therefore, this result shows that conversion is optimum with 1M at 90mins.

Comparing the two acids, H_2SO_4 acid gave a higher extraction of alumina 14.83g/Lit than HCl which gave 12.21g/Lit both in the same 1M concentration.

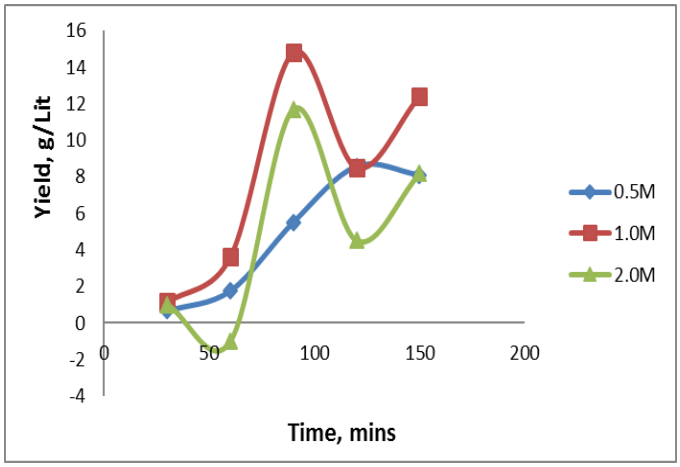

Figure 6. Effect of varying H_2SO_4 concentration in the extraction of alumina

Effect of varying clay-acid ratio:

From the graph below, clay-acid ratio of 10:100 gave a higher yield than 5:100. In ratio 5:100, the amount of clay was small compared to the volume of the acid, which then led to high leaching of the alumina content. However, in using clay to acid ratio of 10:100, a high yield of alumina (10.51g/Lit) was obtained using HCl acid at 120minutes leaching time.

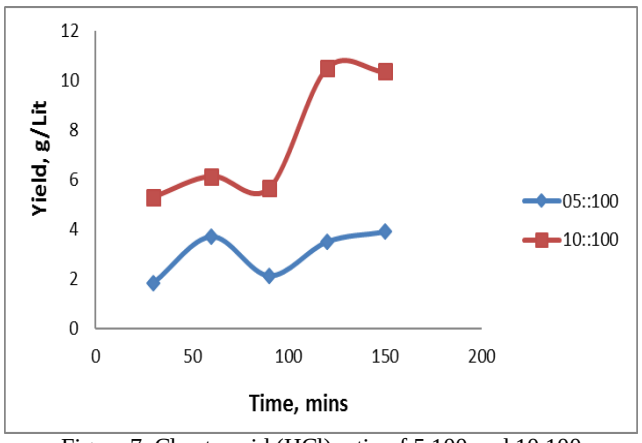

Figure 7. Clay to acid (HCl) ratio of 5:100 and 10:100

Here, clay to acid ratio of 10:100 still gave a high alumina yield (14.19g/Lit). We can deduce that sulphuric acid gave a higher conversion than hydrochloric acid.

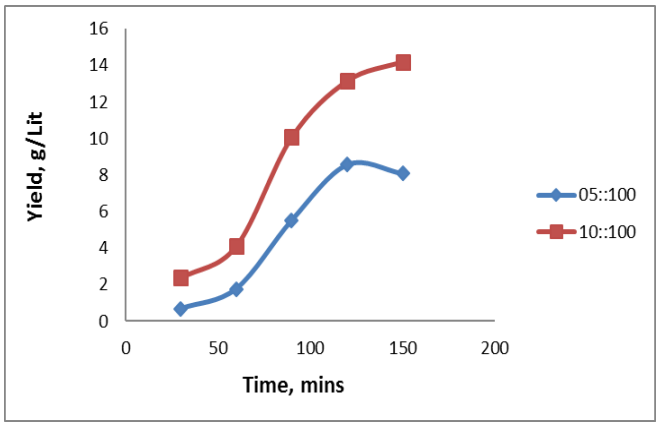

Figure 8. Clay to Acid(H_2SO_4) Ratio of 5:100 and 10:100

V. CONCLUSION AND FUTURE SCOPE

The XRD diagrams have shown there are poorly resolved diffraction peaks. It is recommended that the samples are thick enough for a significant amount of the diffracted X-ray intensity comes from the samples at all 2θ angles. Occurrence of any mineral in the samples can further be confirmed by FTIR study.

For the dissolution, clay to acid ratio of 10:100, using H_2SO_4 acid at 150minutes gave the optimum yield of alumina (14.19g/Lit). One of the factors affecting leaching is the solvent used. The solvent chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely. H_2SO_4 showed a better solvent ability as can be seen from the graphs in comparison with HCl acid.

ACKNOWLEDGMENT

I would like to thank Engr. Richard Chijioke Stephen for his friendly support. I also want to express my sincere gratitude to Associate Prof. R.O. Ajemba Department of Chemical Engineering, Nnamdi Azikiwe University, Awka, Anambra State, for guiding me with resources and ideas.

REFERENCES

[1] I.H. Milne, C.M. Warshaw, "Methods of Preparation and Control of Clay Mineral Specimens in X-ray Diffraction Analysis," *Gulf Research and Development Company, Pittsburgh, Pennsylvania*, Vol. **4**, pp. **22 - 30, 1955**.

[2] P. Komadel, J. Madejova, "Acid Activation of Clay Minerals," *Handbook of Clay Science*, Vol. **1**, pp. **263 - 287, 2006**.

[3] P.K. Igbokwe, F.L. Olebunne, M.S. Nwakaudu, "Effect of Activation Parameters on Conversion in Clay – Catalysed Esterification of Acetic Acid," *International Journal of Basic & Applied Sciences*, Vol. **11**, No. **05, 2011**.

[4] Preeti Sagar Nayak, B.K. Singh, "Instrumental Characterization of Clay by XRF, XRD and FTIR," *Indian Academy of Sciences*, Vol. **30**, No. **3**, pp. **235 - 238, 2007**.

[5] G.W. Brindley, "Identification of Clay Minerals by X-Ray Diffraction Analysis," *Clays and Clay Minerals*, Vol. **1**, pp.**119-129, 1952**. doi:10.1346/CCMN.1952.0010116.

[6] A.A. Al-Zahrani, M.H. Abdul-Majid, "Extraction of Alumina from Local Clays by Hydrochloric Acid Process," *Engineering Science Journal of King Abdulaziz Uni, Saudi Arabia*, Vol. **20**, No. **2**, pp. **29-41, 2009**.

[7] S. Ziegenbalg, G. Discher, "Extraction of Alumina from Clay by Hydrochloric Acid Process," *Freiberger Forschungshefte (Reihe) A*: pp. **7-25, 1983**.

[8] Ibid, "Investigations into the Alumina Extraction from Clay by Hydrochloric Acid and Sulfuric Acid Leaching," *Light Metals: Proceedings of Sessions, AIME Annual Meeting (Warrendale, Pennsylvania)*, **1119-1143, 1983**.

[9] J.A. Eisele, "Producing Alumina from Clay by the Hydrochloric Acid Process, A Bench-Scale Study," *U.S Bureau of Mines* R.I, No **8476, 1980**.

[10] B.D. Cullity, "Elements of X-Ray Diffraction," *Addison-Wesley Publishing Company*, **Philippines**, pp. **161-187, 1978**.

[11] Katherine Ozalas, Benjamin F. Hajek, "X-Ray Diffraction Analysis of Thin Clay Films from Dilute Suspensions using Glancing Incidence Diffraction," *Clays and Clay Minerals*, Vol. **44**, No. **6**, pp.**811-817,1996**.

Authors Profile

Engr. Samuel Chekwube Aniaka holds a B.Eng in Chemical Engineering from Nnamdi Azikiwe University, and is currently an M.Eng student of Industrial Engineering, University of Benin. He is a recipient of several scholarship awards for both first and second degrees. He is a Registered Engineer under the Council for the Regulation of Engineering in Nigeria (COREN), a member of various professional bodies such as the Nigerian Society of Engineers (NSE), the Nigerian Society of Chemical Engineers (NSChE), the Nigerian Institution of Safety Engineers (NISafetyE) and has over five years working experience working in the Nigerian power industry.

