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EXTRACTION OF ALUMINA FROM SELECTED NIGERIAN KAOLIN FOR INDUSTRIAL APPLICATIONS

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Abstract

The extraction of alumina from selected Nigerian kaolin was investigated to assess its viability as a raw material for industrial applications. Kaolin samples from four locations Okpella, Afowa (white and black), and Ado Ekiti, were analyzed for their chemical composition and subjected to calcination and acid-leaching processes. The clays were calcined at 900°C to produce metakaolin, a precursor favorable for alumina extraction. Various concentrations of nitric acid (HNO_3) and hydrochloric acid (HCl) were employed to leach alumina from the calcined samples. The leached alumina was precipitated as aluminum hydroxide and subsequently calcined at 900°C to yield alumina (Al_2O_3). The process efficiency was evaluated through XRF analysis, revealing recovery rates up to 92% under optimal conditions. This study demonstrates the potential of Nigerian kaolin as a sustainable source of alumina for industrial applications and highlights the influence of acid concentration and calcination on extraction efficiency.

Keywords: *Acrylic bone cement, Cement-bone interface, BHR Prostheses, SEM analysis.*

Introduction

Alumina (Al_2O_3) is a vital material with extensive applications across numerous industries, such as ceramics, refractories, and aluminum production. Its significance stems from its exceptional properties, including high thermal and chemical stability, corrosion resistance, and electrical insulation [1-3]. These attributes make alumina indispensable in manufacturing advanced ceramics, high-temperature resistant materials, and as a primary feedstock for aluminum production. The growing demand for alumina, driven by the rapid industrialization and technological advancements, has placed considerable emphasis on identifying sustainable and cost-effective raw materials to meet global needs.

Traditionally, bauxite has been the primary source of alumina [4]. However, the environmental challenges associated with bauxite mining and the increasing depletion of high-grade reserves have intensified the search for alternative alumina sources [5]. One such promising alternative is kaolin, a naturally occurring clay mineral composed predominantly of aluminum silicate [6, 7]. Kaolin's wide distribution, ease of access, and favorable mineralogical properties make it an attractive candidate for alumina extraction [8]. When subjected to thermal treatment, kaolin undergoes dehydroxylation to form metakaolin, a highly reactive phase that facilitates efficient alumina leaching [6].

Nigeria is endowed with substantial kaolin deposits, particularly in states like Edo and Ekiti, where these resources remain underexplored for industrial applications [9]. Harnessing these deposits for alumina production not only reduces reliance on imported materials but also offers significant economic and environmental benefits. Despite its potential, the industrial utilization

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of Nigerian kaolin faces several challenges, including the variability in clay composition, the need for efficient calcination, and optimization of the leaching process. Factors such as the type of acid used, its concentration, calcination temperature, and leaching duration significantly influence the yield and purity of extracted alumina [10]. Addressing these challenges requires a comprehensive understanding of the mineralogical characteristics of the clay and a systematic approach to refining the extraction process.

This study focuses on extracting alumina from kaolin samples sourced from specific locations in Nigeria, namely Okpella, Afowa (white and black), and Ado Ekiti. These samples were selected based on their high alumina content, identified through X-ray fluorescence (XRF) analysis. Employing acid-leaching techniques, the research evaluates the effects of varying process parameters on alumina recovery and purity. By analyzing the chemical composition and recovery efficiency, the study aims to determine the industrial suitability of these kaolin deposits. The findings will contribute significantly to the sustainable exploitation of Nigerian kaolin, promoting local resource utilization and advancing cost-effective methodologies for industrial alumina production.

Materials and Methods

Extraction process of the alumina from the clays

From this research, four clays were selected for this experiment: Afowa black clay, Ado Ekiti clay (sourced from the satellite area), Afowa white clay and Okpella clay, as outlined in Table 1 because these clays have higher percentage of alumina. The higher the Al_2O_3 composition in a clay sample, the greater the potential for alumina leaching from the clay [11]. These samples were chosen based on their high alumina content, as identified through XRF analysis. The clays underwent thorough washing, were dried for one week, and then sieved to a particle size of 75 microns to achieve uniformity. Following this preparation, the samples were placed in a muffle kiln, where they were heated to 900°C and held at this temperature for three hours to complete the calcination process. The calcined samples were allowed to cool overnight before collection. This calcination step enhances the properties of kaolin by improving its whiteness, chemical inertness, and transforming it into metakaolin, a beneficial form for further applications [12]. Once fully cooled, the samples were carefully transferred into labeled plastic containers for storage.

Table 1. List of clays used for the extraction of alumina

S/N	Source	State
1	Okpella/ Edo state	Edo
2	Afowa white Edo state	Edo
3	Afowa black Edo State	Edo
4	Secretariat along satellite campus Ado Ekiti	Ekiti

In this experiment, nitric acid (HNO_3) and hydrochloric acid (HCl) were used to process the clay samples. Initially, 3M and 4M concentrations of HNO_3 were applied for leaching the samples, followed by the addition of HCl to precipitate aluminum hydroxide. In the subsequent phase, 3M and 4M concentrations of HCl were used both for the leaching process and the precipitation stage. This choice of acid concentrations and prolonged leaching times aimed to minimize the total acid required, thereby reducing production costs while enhancing extraction efficiency compared to other studies [7, 12].

For each trial, 10 grams of calcined clay was combined with 100 ml of 3M acid solution in a 250 mL round-bottom flask equipped with a condenser. The solution was preheated to 90°C, and the mixture was stirred at a speed of 500 rpm for 3hours. The resulting slurry was filtered using a Buchner funnel, and the residue was washed with distilled water and dried at 80°C. Next, a 5M

solution of sodium hydroxide (NaOH) was added to the filtrate to convert the dissolved alumina into sodium aluminate ($NaAlO_2$), allowing the separation of iron and magnesium hydroxides (as shown in Fig. 3). Following an additional filtration step, 4M HCl was introduced into the $NaAlO_2$ filtrate with continuous stirring to adjust the pH to 7, prompting the precipitation of aluminum hydroxide. This precipitate was aged overnight, filtered, thoroughly rinsed with distilled water, and dried at 110°C.

Finally, alumina (Al_2O_3) was obtained by calcining the aluminum hydroxide precipitate in a muffle furnace at 900°C for two hours. The percentage of aluminum recovered was calculated using Equation 1, with the recovery results presented. Additionally, the alumina samples underwent characterization using the EDX3600B X-ray fluorescence spectrometer (XRF), with the findings detailed and recorded.

$$\% Al_2O_3 = 100 \times (\text{final weight} / \text{initial weight}) \quad (1)$$

Result and Discussion

Results

A portion of the calcined kaolin samples was selected for chemical composition analysis using the EDX3600B X-ray fluorescence (XRF) spectrometer. Phase identification was conducted with a BRUKER AXS D8 Advanced X-ray diffractometer, employing Cu Kα radiation, and scanning within a 2θ range of 5° to 70°. Additionally, the microstructural and elemental analysis was performed using Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM/EDS) on a Phenom Pro X SEM system. The results from these analyses were thoroughly documented.

XRD result of the Kaolin samples

The XRD analysis results for the clay samples, depicted in Figures 1–4, provide a comprehensive overview of their mineralogical composition. These results are instrumental in identifying the crystalline phases present, highlighting both primary and secondary mineral components. Such detailed insights into the structural and chemical properties of the clays are essential for assessing their potential in various industrial applications, including ceramics production, alumina extraction, and other specialized uses. The diffraction patterns presented in the figures enable a thorough evaluation of the quality and suitability of the clay samples, serving as a critical foundation for determining their effectiveness in targeted applications.

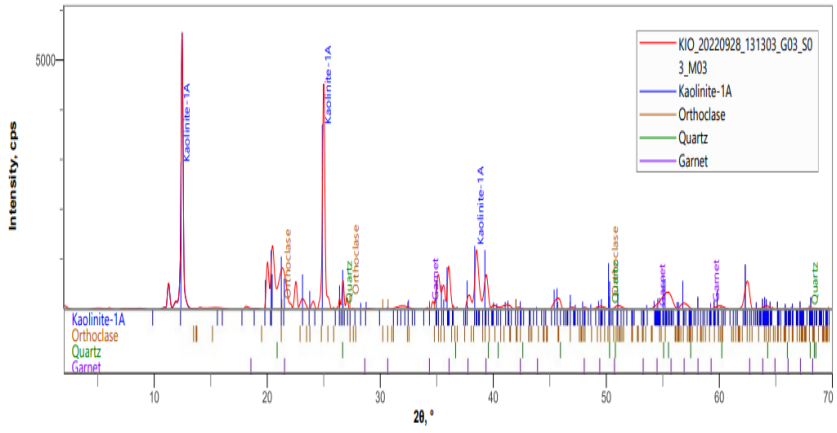

Fig. 1. XRD result of Okpella kaolin

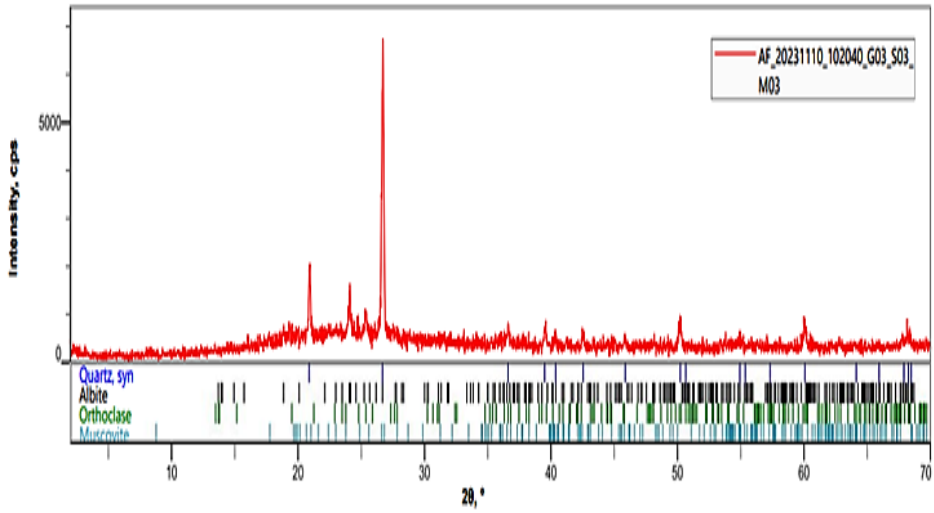

Fig. 2. XRD result of Afowa white kaolin

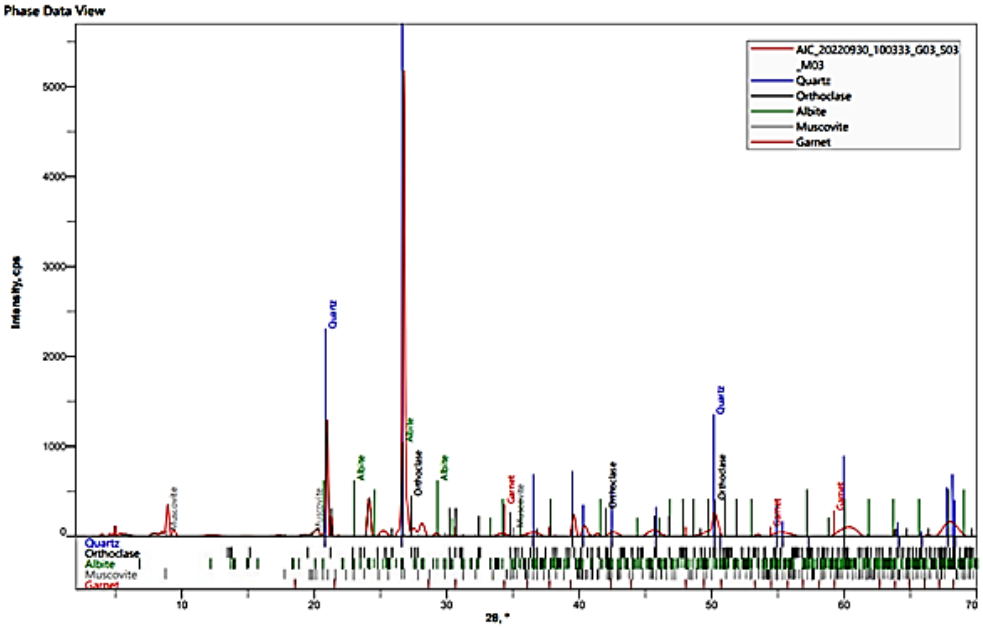

Fig. 3. XRD result of Afowa black kaolin

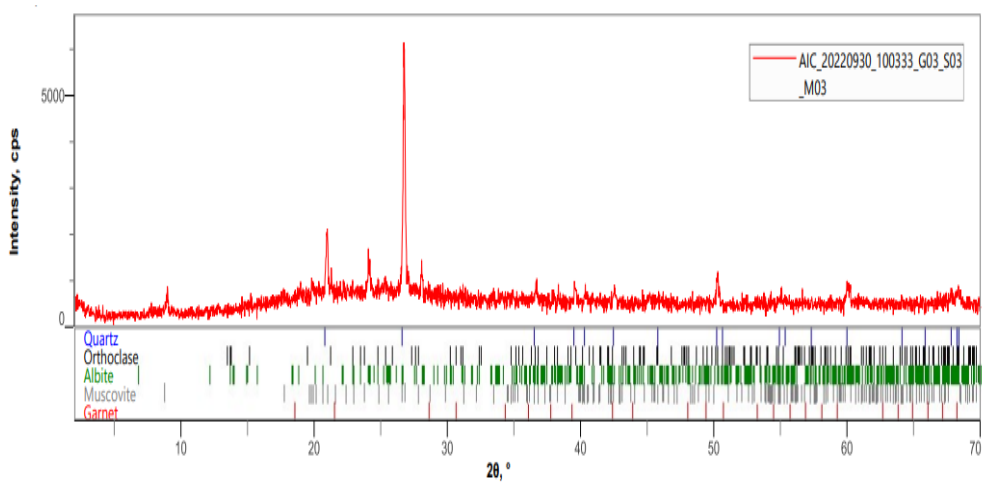

Fig. 4. XRD result of secretariat along satellite campus kaolin

XRF result of the kaolin samples

Elemental chemical analysis of clay samples is a critical process used to determine the chemical composition of clay, including the types and quantities of minerals it contains [13]. This analysis plays a vital role in various applications by identifying unknown materials, ensuring that a material complies with specific standards or requirements, and detecting the presence of potentially harmful chemicals. By providing detailed insights into the elemental and mineralogical makeup of the clay, this process supports quality control, environmental assessments, and the development of suitable industrial or construction materials. The elemental chemical analysis of the four clays were determined using XRF and the result presented in Table 2.

Table 2. Elemental Chemical analysis XRF result of the selected samples

Clay	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2
Okpella/ Edo state	52.613	41.323	0.950	0.093	3.670
Afowa white/ Edo state	54.147	35.412	1.787	0.100	6.306
Afowa black Edo State	54.476	40.637	1.291	0.035	0.236
Secretariat along satellite canpus Ado ekiti	55.258	36.217	4.232	0.233	2.090

Result of the percentage Alumina recovery from each sample of the clay

Table 3 presents the detailed result of the percentage of alumina recovery from the kaolin samples. This represents the amount of alumina extracted in percentages from each clay after leaching with different acids.

Table 3. Percentage by weight of alumina extracted from the clay

Percentage Recovery	HNO_3 (3M) %	HNO_3 (4M) %	HCL (3M) %	HCL (4M %
Afonwa black clay	42.4	42.7	41.5	41.9
SAT Ado Ekiti	42.8	43.2	42.5	43.2
Afonwa white clay	40.4	41.2	40.3	40.9
Okpella	42.3	43.5	42.3	42.8

Table 4. presents the XRF result of the extracted calcined alumina from the kaolin. The alumina extraction results from the clay samples are summarized in Table 4, which details the

quantity of alumina recovered during the extraction process. This data highlights the efficiency and effectiveness of the methods employed, providing valuable insights into the potential of the clay as a raw material for alumina production. It also aids in evaluating the clay's suitability for various industrial applications. Additionally, Fig. 5 presents a bar chart illustrating the calcined alumina extraction results from the clays, as determined by XRF analysis.

Table 4: XRF result of the calcined alumina at 900^{O}C

Clay	Composition	HNO_3 (3M) 3hrs 500rpm	HNO_3 (4M) 3hrs 500rpm	HCL (3M) 3hrs 500rpm	HCL (4M) 3hrs 500rpm
Afonwa black clay	SiO_2 -	11.817	3.241	12.840	11.077
	Al_2O_3 -	82.292	92.979	78.355	83.379
	Fe_2O_3 -	0.152	0.152	1.781	0.0743
	Cl -	2.058	2.058	3.398	2.483
SAT Ado Ekiti	SiO_2 -	9.134	3.165	7.491	5.212
	Al_2O_3 -	86.786	92.021	79.966	86.803
	Fe_2O_3 -	0.0792	0.155	0.642	0.359
	Cl -	1.743	1.821	6.482	6.151
Afonwa white	SiO_2 -	9.52	4.532	5.728	10.894
	Al_2O_3 -	84.060	86.063	73.915	82.019
	Fe_2O_3 -	0.647	0.296	0.258	0.867
	Cl -	0.438	4.857	10.971	3.8412

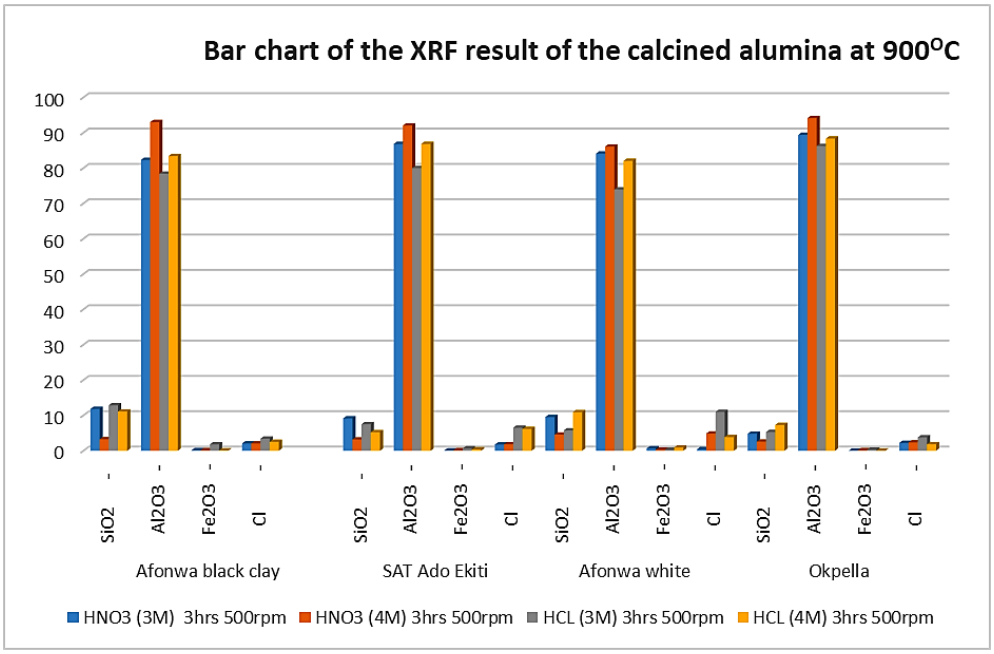

Fig. 5. Bar chat of the XRF result of the calcined alumina at 900°C

Discussion

XRD result

The XRD analysis results for the kaolin samples, as shown in Figures 1-4, reveal significant mineralogical characteristics that are closely aligned with their potential for alumina extraction. The diffraction patterns indicate the presence of kaolinite as a primary mineral phase, along with other minor crystalline components. Kaolinite, an aluminum silicate, is highly desirable for alumina extraction due to its rich aluminum content and relatively simple structure, which

facilitates chemical reactivity during calcination and leaching processes [6]. In the case of the Okpella kaolin (Fig. 1), the clay indicates a well-defined diffraction at 2-theta of kaolinite at 12.5º and 25º, with traces of orthoclase, quartz and garnet which confirm a dominant kaolinite phase with minimal impurities, underscoring its suitability for alumina recovery. Similarly, the Afowa white kaolin (Fig. 2) indicates a well-defined diffraction at 2-theta of kaolinite at 27 º, with traces of albite, orthoclase and muscovite while Afowa black kaolin (Fig. 3) exhibit strong kaolinite signatures indicates a well-defined diffraction at 2-theta of 21.5º and 27 º, with traces of albite, orthoclase, garnet and muscovite , with slight variations in secondary mineral phases , which could influence their reactivity during extraction. The satellite campus kaolin Ado Ekiti sample (Fig. 4) also demonstrates a kaolinite-rich composition diffraction at 2-theta of 27º, with traces of albite, orthoclase, Muscovite and garnet , further validating its potential as a raw material for alumina production. The identification of secondary phases, such as quartz and traces of iron- and titanium-bearing minerals, provides additional insights into the clays' mineralogical composition [15]. While quartz is inert in alumina extraction processes, the presence of iron and titanium compounds, though minor, may require attention during the purification stages to ensure the quality of the extracted alumina.

XRF result of the kaolin samples

The chemical composition analysis of the four clay samples, determined by XRF, highlights their potential for alumina extraction. The SiO_2 content across the samples ranges from 52.613% in Okpella clay to 55.258% in Ado Ekiti clay. While a high silica concentration is characteristic of kaolin clays and provides stability during processing, it may dilute the aluminum content and pose challenges for alumina recovery.

The alumina (Al_2O_3) content, a critical factor for industrial applications, varies between 35.412% in Afowa white clay and 41.323% in Okpella clay. The higher alumina concentrations in Okpella and Afowa black clays indicate their strong potential as raw materials for alumina production. These results align with the selection criteria for clays rich in aluminum content [4].

The iron oxide (Fe_2O_3) content is relatively low, ranging from 0.950% in Okpella clay to 4.232% in Ado Ekiti clay. While low iron levels are ideal for producing high-purity alumina, Ado Ekiti clay may require additional purification steps to meet industrial standards. Minor components such as calcium oxide (CaO) and titanium dioxide (TiO_2) are present in small amounts. The CaO content in Okpella clay, the lowest among the samples at 0.093%, enhances its suitability for alumina extraction by minimizing contamination risks.

Table 3 presents the Alumina Recovery Efficiency of the leaching process

The percentage of alumina recovered from the clay samples during acid leaching (Table 3) demonstrates the effectiveness of the extraction methods and provides a comparative evaluation of the clays:

Influence of Acid Type and Concentration:

HNO_3 (3M and 4M): Alumina recovery ranges from 40.4% to 43.5%, with higher concentrations generally yielding slightly better recovery rates. Okpella clay exhibits the highest recovery efficiency (43.5% with 4M HNO_3), followed by Ado Ekiti clay.

HCl (3M and 4M): Similar trends are observed, with recovery rates reaching up to 42.8% for Okpella clay and Ado Ekiti clay. The consistent performance of these samples suggests their adaptability to various acid treatments.

Sample Comparisons

Okpella Clay: Demonstrates superior recovery efficiency across all acid concentrations, confirming its high suitability for industrial alumina extraction while Ado Ekiti Clay: Achieves comparable recovery to Okpella, indicating its viability despite higher Fe_2O_3 content. Afowa

Clays: Show slightly lower recovery rates, likely due to their lower Al_2O_3 content compared to Okpella clay.

Table 4 presents the XRF result of the calcined alumina at 900OC

The XRF analysis of the calcined alumina extracted at 900°C reveals critical insights into the composition and suitability of the different clay samples for alumina production. The results focus on key oxides (SiO_2, Al_2O_3, Fe_2O_3) and residual chlorine (Cl), highlighting the effects of acid type, concentration, and clay source on extraction efficiency.

Al_2O_3 Content

The alumina content (Al_2O_3) ranges from 73.915% (Afonwa white clay, 3M HCl) to 94.11% (Okpella clay, 4M HNO_3). Higher acid concentration (4M) significantly enhances alumina purity across all samples, indicating improved leaching efficiency. For instance, Afonwa black clay's alumina content increases from 82.292% (3M HNO_3) to 92.979% (4M HNO_3). Among all the clays, Okpella consistently achieves the highest alumina purity, establishing it as the most efficient raw material for alumina production out of the four samples.

SiO_2 Content

Silica content (SiO_2) varies widely, with the lowest levels in Okpella clay (2.574% under 4M HNO_3) and higher values in clays like Afonwa black (12.84% under 3M HCl). HNO_3 treatments generally result in lower silica levels compared to HCl, highlighting its superior ability to remove siliceous impurities. Low SiO_2 content is critical for producing industrial-grade alumina, as excessive silica can limit its application, particularly in refractory materials.

Fe_2O_3 Content

Iron oxide (Fe_2O_3) is consistently low across the samples, ranging from 0.006% (Okpella, 4M HCl) to 1.781% (Afonwa black, 3M HCl). Clays like Okpella and SAT Ado Ekiti exhibit minimal iron levels, making them ideal for applications requiring high-purity alumina. HCl tends to leave slightly higher Fe_2O_3 content, potentially due to its lower oxidizing capacity compared to HNO_3.

Residual Chlorine (Cl)

Residual chlorine (Cl) content ranges from 0.438% (Afonwa white, 3M HNO_3) to 10.971% (Afonwa white, 3M HCl). HCl treatments, especially at lower concentrations, result in higher residual chlorine levels, indicating incomplete removal during washing . HNO_3 consistently produces lower chlorine residues, demonstrating better impurity control. The molarity of the acids is kept below 4M o as to reduce cost as against [7] who uses 6M leaching concentration of the acid.

Implications

Effect of Acid Type and Concentration:

HNO_3 is more efficient than HCl, yielding higher alumina purity with lower SiO_2, Fe_2O_3, and Cl levels. Higher acid concentrations (4M) further improve alumina purity and impurity removal, enhancing the overall efficiency of the extraction process.

Clay Type Performance

Okpella clay demonstrates the best performance, achieving the highest alumina purity and lowest impurity levels, making it the top candidate for industrial alumina production. SAT Ado Ekiti clay performs well, with alumina purity exceeding 86% in most conditions, though its SiO_2 and Cl levels are slightly higher than those of Okpella clay. Afonwa black and Afonwa white clays show relatively higher impurity levels (SiO_2 and Fe_2O_3) and lower alumina content, especially under HCl treatments, making them less efficient compared to the other clays.

Conclusion

Based on this research, the following conclusions can be made:

The results demonstrate the feasibility of extracting high-purity alumina from Nigerian kaolin clays using acid-leaching techniques.

Okpella and Ado Ekiti clays exhibit superior performance in terms of alumina content, recovery efficiency, and purity, making them the most promising candidates for industrial applications.

The findings underscore the potential of utilizing local resources to meet growing industrial demand for alumina, reducing reliance on imported materials, and fostering economic development.

Further studies should focus on process optimization, including acid recovery and energy efficiency, to enhance the sustainability and cost-effectiveness of alumina production from kaolin.

References

[1] Da Fonseca, J. G. P. (2023). Refractory Ceramics in the Context of the Fabrication of Rocket Nozzles: Conventional Alumina and Industrial Waste Alumina-Silicate **Refractory Compositions**. Universidade da Beira Interior (Portugal).

[2] Sengupta, P. (2020). **Refractories for the cement industry**. Springer

[3] Smith, P., & Power, G. (2022). High purity alumina–current and future production. **Mineral Processing and Extractive Metallurgy Review**, 43(6), 747–756

[4] Raahauge, B. E., & Williams, F. S. (2022). Introduction: Primary Aluminum–Alumina–Bauxite. In Smelter Grade Alumina from Bauxite: **History, Best Practices, and Future Challenges** (pp. 1–17). Springer

[5] Lamidi, Y. D., Kashim, I. B., Adelabu, O. S., & Oluwasina, O. O. (2024). Extraction of pure alumina from kaolin: a review. **European journal of materials science and engineering**, Volume 9, Issue 3, 2024: 191-200 |

[6] Kyriakogona, K., Giannopoulou, I., & Panias, D. (2017). Extraction of aluminium from kaolin: A comparative study of hydrometallurgical processes. **Proceedings of the 3rd World Congress on Mechanical, Chemical, and Material Engineering,** 17, 8–10.

[7] Ibrahim, K. M., Moumani, M. K., & Mohammad, S. K. (2018). Extraction of γ-alumina from low-cost kaolin**. Resources**, 7(4), 63.

[8] Ali, A., AL-Taie, M., & Ayoob, I. (2019). The Extraction of Alumina from Kaolin. **Engineering and Technology Journal**, 37(4A), 133–139. https://doi.org/10.30684/etj.37.4a.4

[9] Ogunnowo, R. O. (2022). An evaluation of natural resources extraction and host communities' reaction in Nigeria and South Africa 2005-2015. North-West University (South Africa).

[10] Eldeeb, A. B., Brichkin, V. N., Martin Bertau, Savinova, Y. A., & Kurtenkov, R. V. (2020). Solid-state and phase transformation mechanism of kaolin sintered with limestone for alumina extraction**. Applied Clay Science**, 196.

[11] Dewi, R., Agusnar, H., Alfian, Z., & Tamrin (2018). Characterisation of technical kaolin using XRF, SEM, XRD, FTIR and its potential as industrial raw materials; **international conference on science and technology**.

[12] Tantawy, M. A., & Ali Alomari, A. (2019). Extraction of Alumina from Nawan Kaolin by Acid Leaching. **Oriental Journal of Chemistry**, 35(3), 1013–1021. https://doi.org/10.13005/ojc/350313

[13] Kumari, N., & Mohan, C. (2021). **Basics of clay minerals and their characteristic properties**. Clay Clay Miner, 24(1).

[14] Eldeeb, A. B., Brichkin, V. N., Kurtenkov, R. V., & Bormotov, I. S. (2019). Extraction of alumina from kaolin by a combination of pyro- and hydro-metallurgical processes. **Applied Clay Science**, 172, 146-154.

[15] Yunusa, A., Hong, H., Salim, A., Amam, T., Liu, C., Xu, Y., Zuo, X., & Li, Z. (2024). Mineralogical Characterization and Geochemical Signatures of Supergene Kaolinitic Clay Deposits**: Insight of Ropp Complex Kaolins**, Northcentral Nigeria. Minerals, 14(9), 869.

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