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EXTRACTION OF PURE ALUMINA FROM KAOLIN: A REVIEW

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Abstract

This study demonstrated that high-purity alumina can be generated from kaolin using various methods. It examined different leaching techniques, considering the size and percentage recovery of alumina by weight in selected kaolin samples, the calcination temperature, and the molar concentration of acids over time. Additionally, it analyzed the characteristics of several kaolin sources in Nigeria with the aim of extracting alumina. The research indicates that Nigerian kaolinite clay typically contains 22–40% alumina by weight. However, leaching kaolin yielded alumina with a purity of 60–97 wt%, with particle sizes ranging from 16 to 177 nm. Alumina derived from kaolin is deemed beneficial for manufacturing refractory materials, water purification, and biomedical applications.

Keywords: *Alumina, kaolin, characterization of kaolin, extraction of alumina*

Introduction

Kaolin, commonly known as China clay, is a fine white clay mineral made up mostly of kaolinite and other minerals like quartz, feldspar, and mica. It is widely spread geologically and found in deposits throughout the continents, making it an easily accessible and economically lucrative resource [1]. Alumina derived from kaolin is extremely important in industry due to its versatile qualities and wide range of applications. As the principal precursor for aluminum manufacture, alumina is an essential component in the creation of aluminum metal, ceramics, catalysts, abrasives, and a variety of other products used in various industries [2]. The predominant raw material used in alumina production is Bauxite, and this is achieved through the Bayer's process [3]. However, the economic discovery of bauxite in substantial quantities is lacking in many regions, and the substantial personnel requirements for initiating bauxite processing plants open the door to exploring alternative methods for alumina production that do not rely on the Bayer process. According to a report by The Vanguard newspaper on November 26, 2018, the Republic of Guinea invested approximately $3 billion in establishing a facility for bauxite refining to obtain alumina, highlighting the high cost associated with this method. Consequently, there is a need to use kaolinite clay as an alternative to bauxite for alumina production [2, 4].

The extraction of alumina from kaolin involves several intricate steps, typically starting with beneficiation processes to remove impurities and enhance the purity of the kaolin feedstock. Subsequently, the kaolin undergoes a series of chemical treatments, often involving acid leaching or alkali digestion, to dissolve the alumina content selectively [5]. Following the dissolution of alumina from kaolin, the solution is subjected to precipitation techniques to recover the alumina as a solid precipitate. This precipitate is then washed,

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filtered, and subjected to calcination mostly ranging from (600-850^{o}C), a process of heating to high temperatures to transform the precipitated alumina hydrates into the desired alumina oxide form [2]. Alumina finds extensive utilization across diverse industries, ranging from the production of aluminum metal via electrolytic reduction to the manufacturing of ceramics, refractories, abrasives, and specialty chemicals. The efficient extraction of alumina from kaolin contributes significantly to industrial productivity, economic growth, and technological advancement. It is important to note that this method of alumina recovery has substantial economic benefits, including plentiful supply, low cost, environmental friendliness, high purity, and a variety of uses [6]. To achieve a high yield alumina with greater economic benefit, it is imperative to address environmental issues associated with waste management, mining, and processing. To guarantee the responsible use of kaolin resources while limiting environmental impact, it is imperative to implement sustainable practices, conserve resources, and engage in environmental stewardship

The Kaolin

Clays are abundant fine powders formed through the weathering and disintegration of granite and feldspathic rock, occurring in both plain and riverine areas [7] & [8]. They represent complex inorganic mixtures with widely varying compositions dependent on geographical location. These minerals are prolific in nature, continually generated on the earth's surface as a consequence of rock weathering [9] & [10]. Most clay minerals primarily comprise silica, alumina, and water of hydration, with varying proportions of organic or carbonaceous materials. Erosion, weathering agents, or the prolonged movement of feldspathic rock minerals contribute to the formation of clay over time. Examples of clay minerals include ball clay, kaolinites, bentonites, mullites, montmorillonite-smectite, and illite. Additionally, chlorite, vermiculite, talc, and pyrophyllite are sometimes categorized as clay minerals.

Kaolin, a soft and white clay, is primarily composed of kaolinite ($Al_4(OH)_8.(Si_4O_{10})$, and it may also contain other minerals like nacrite and dickite [11]. The name "kaolin" originates from a hill in China known as "kao-ling," where it has been mined for centuries [1] & [12]. Commonly referred to as China clay, kaolin is a mineral characterized by a crystal compound primarily made up of kaolinite ($Al_2O_3.2SiO_2.2H_2O$). Its notable characteristics include whiteness, softness, and the ability to disperse easily in water or other solutions [13].

According to [13], pure kaolin theoretically contains 46wt% silica, 40wt% alumina, and 14wt% water. It finds widespread use in the ceramic industry due to its plasticity, ease of shaping, smooth texture, quick drying, and production of a desirable white color after burning. Kaolin is also utilized in the rubber industry as fillers, developers, and amplifiers, as well as in the paper industry, among other applications. Its versatility extends to the paint industry, cosmetics, ink, medicine, crayon fillers, oil adsorbents, catalysts, and wine purifiers.

In Nigeria, kaolin deposits are distributed across the Anambra, Chad, and Sokoto Basins, the Benue Trough, and the Niger Delta Basin. In the northern part of the country, these deposits are found in Bauchi, Borno, Gombe, Katsina, Kaduna, and Kebbi States. In the southern part, they are present in Anambra, Enugu, Ondo, and Oyo States. Specific locations in Kebbi State include parts of Dakingari, Kaoje, Giro, and Koko [14]. An overview of kaolin deposits in Nigeria, as reported by the Raw Material Research and Development Council's report (1989), indicates that most Nigerian kaolin contains an average of 25 to 39% alumina. As discussed by [15], these deposits can be processed to extract the alumina content for industrial applications, including alumina production through leaching processes from kaolin clay. Table 1 below summarizes the locations and deposits of kaolin clay as reported by different authors.

Table 1. Kaolin clay samples from diverse locations in Nigeria have been characterized by various authors in the reviewed studies

S/N	State	Region	Silica wt%	Alumina wt%	Reference
1	Edo	Ajegbo2	32.540	29.834	[16]
2	Edo	Anegha	37.295	20.227	[16]
3	Osun	Ifon clay	63.35	22.42	[8]
4	Ondo	Igbara odo	56.636	25.737	[8])
5	Osun	Ipetumodu	59.482	25.02	[8]
6	Ekiti	Isan clay	54.657	23.98	[8]
7	Oyo	Iseyin clay	62.292	22.729	[8]
8	Edo	Ujogba	53.93	24.61	[17]
9	Edo	Ikpeshi (Akoko Edo)	53.18	30.67	[18]
10	Kebbi	Kasadi village	57.41	26.88	[18]
11	Osun	Alasan (iwoye junction)	49.30	33.10	[18]
12	Kebbi	Kaoje A	46.11	20.63	[19]
13	Kebbi	Kaoje B	40.68	26.3	[19]
14	Kebbi	Kaoje C	44.18	29.7	[19]
15	Ekiti	Ikere	54.752	40.302	Authors result
16	Edo	Auchi	60.769	27.319	Authors result
17	Osun	Ifon	63.35	22.42	[8]
18	Osun	Ipetumodu	59.4	25.03	[8]
19	Oyo	Iseyin	22.729	62.292	[8]
20	Ondo	Igbaraodo	25.737	56.636	[8]
21	Ondo	Ipinsa	43.05	53.91	[20]
22	Ekiti	Ijero	39.60	58.00	[20]
23	Delta company	Anara	38.67	44.50	[21]
24		Ire	54.00	39.35	[22]
		Ikere	44.93	38.31	
		Isan	46.50	30.01	
		Ado	46.10	33.26	
		ilawe	45.76	34.42	
25	Ondo	Onibode	39.30	42.30	[20]
26	Abuja	Sheda	40	41	[23]
		Abaji	43	37	
		Karimu	40	41	

The Alumina

Alumina, a crucial mineral employed across various industries as a catalyst, in abrasives, adsorbents, and refractories, is present in a stable form known as alpha alumina. Additionally, it exists in several meta-stable forms, including γ-, η-, δ-, θ-, κ-, and χ-alumina [24]. While alumina occurs naturally in its pure state as the mineral corundum, the primary natural ore for alumina is bauxite. Bauxites have been extensively utilized in industry for alumina production through the Bayer process, serving as the principal raw material for alumina production [3].

Alumina, found in stable form as alpha-alumina and various meta-stable forms like -alumina, holds significance as an industrial mineral. It serves purposes such as an abrasive material and an adsorbent [2]. Alumina is widely applicable in industrial and advanced technological settings, as well as in ceramics for producing high-strength materials due to its impressive mechanical strength. Its uses span the processing of high-quality insulators, micro-electronics, biofuels, and cell fuels, as well as in fireproof plastics, semiconductors, high-grade polishes, and refractory products.

Due to the limited discovery of bauxite in economically viable quantities in most countries, kaolinite clay has been processed as an alternative to bauxite for alumina production [2]. While bauxite ores are abundant in specific countries, non-bauxitic ores are widely distributed across many nations [25], [26] & [27]. One notable alternative source is Al-rich kaolin, a hydrous aluminum silicate ($Al_2Si_2O5(OH)4$) composed of the mineral kaolinite [15]. [5] asserts that the global surge in aluminum demand has prompted interest in developing alternative technologies for aluminum production from low-grade ores. Accordingly, extensive research and engineering endeavors have been undertaken in numerous countries to extract alumina from various naturally occurring non-bauxitic sources, particularly clays. Clays, abundant minerals primarily consisting of alumino-silicate minerals with traces of iron oxide and alkali-metal oxides [28] & [6], have been a focus in this pursuit.

Among the various clays, kaolin emerges as a particularly attractive candidate for alumina production due to its high aluminum content. The elevated aluminum content of kaolin (20–40%) renders it appealing as a substitute for bauxite in alumina production [3], [29] & [25] anticipates that, in the near future, a commercial process utilizing low-grade aluminum ore will be essential for aluminum production. He suggests that many clay minerals, especially kaolin, contain 30–40% alumina, and he further posits that kaolinite, with its high alumina content, stands out as the most plausible candidate to replace bauxite as an aluminum ore. Alumina is utilized in the manufacturing of a diverse range of ceramic products, including spark-plug insulators, integrated-circuit packages, bone and dental implants, laboratory ware, sandpaper grits and grinding wheels, as well as refractory linings for industrial furnaces and some specialized refractories.

Extraction of Alumina from Bauxite by Bayer's Process

Bauxite, the primary ore for alumina (Al_2O_3) used in aluminum (Al) production, consists of hydrated aluminum oxides, hydrated aluminosilicates, iron oxides, hydrated iron oxides, titanium oxide, and silica [30]. It contains various minerals such as gibbsite, boehmite, hematite, goethite, Al-goethite, anatase, rutile, ilmenite, kaolin, and quartz, and is derived from sedimentary, igneous, or metamorphic rocks [24].

According to Feeco International (2016), the Bayer process for obtaining alumina from bauxite is intricate, involving complex and lengthy chemical reaction processes. The process can be summarized as including crushing, washing, digestion, sedimentation, precipitation, and clarification. [30] supports this perspective, emphasizing the associated risks, including health, chemical, biological, ergonomic, and psychosocial risks, along with environmental concerns such as the red mud problem. Red mud, a by-product of the Bayer process, contains high alkaline content and poses a risk of water contamination when exposed to the environment, affecting both humans and aquatic animals.

It is noteworthy, as mentioned by [28] that economically viable bauxite for aluminum production must have an aluminum oxide content of 30% or higher. Only ores with high concentrations of minerals like gibbsite and boehmite, containing 65% to 85% alumina, are considered economically viable for processing.

Bauxite mining operations typically employ 500–1000 people for site, factory, or processing work.

The Processes of Extraction of Alumina from Kaolin

Hydrometallurgical Process

In the investigation conducted by [5] on the extraction of alumina from kaolin, it was concluded based on experimental results that the use of acids is more effective than bases in this process. Notably, oxalic acid emerged as a particularly attractive leaching agent for extracting alumina from calcined kaolin. The process involved a particle size of 250 microns before calcination. [5] explored the impact of varying calcination temperatures (650 °C, 750 °C, 850 °C, and 950 °C) and different durations (90, 120, and 180 minutes). The choice of acid solution used was a crucial factor, including options such as HCl, HNO_3, H_2SO_4, and $C_2H_2O_4$. According to his findings, the extraction of alumina from kaolin using HCl and H_2SO4 reached 82% and 76%, respectively, while HNO3 and oxalic acids achieved 86% and 79%. [31] expressed that, on a laboratory bench scale, leaching of alumina with diluted nitric acid exhibited high extractability, ranging between 60% and 86%. This was demonstrated through samples of micaceous residue and reference minerals, such as kaolinite and muscovite mica, underscoring the efficacy of HNO_3 in the extraction process.

Leaching with acid with varying concentration

The calcination temperature employed by [5] varied from 650 to 950 degrees Celsius, whereas [2] used a temperature range of 600 to 750 degrees Celsius for a duration of 2 hours. [2] cited opinions from [3] and [27] indicating that calcination of kaolin before leaching serves to eliminate organic materials, water, and chlorine. Additionally, it aids in activating an alumina layer within the kaolin structure during acid leaching in chemical treatment.

[2] experimented the leaching of alumina from kaolin involved particle sizes ranging from 125 to 38 micrometers. Hydrochloric acid with different pH values (ranging from 0.45 to 0.65) was used for leaching over 2 hours at temperatures of 30, 60, and 90 degrees. Sodium hydroxide was employed for treatment with NaOH. The study concluded that the leaching method resulted in a 95% extraction of alumina when analyzed using XRD. It was further emphasized that careful consideration of reaction temperature is essential, as an increase in reaction temperature correlates with a decrease in alumina content.

[32] suggested that achieving a higher percentage purity of alumina, up to 95%, requires a leaching time ranging from 30 to 180 minutes and a solid/liquid ratio of 0.1 g/ml when leaching with HCl or H_2SO_4. However, he affirmed that the percentage of leaching with HCl is higher than that of H_2SO_4.

Two ways leaching with acids

In his publication, [4] delved into the leaching of alumina from kaolin specifically with sulfuric acid and hydrochloric acid. He noted that [25] explored alumina extraction from kaolin using hydrochloric acid, sulfuric acid, and nitric acid. The findings suggested that the leaching rate of alumina was highest with hydrochloric acid, followed by sulfuric acid, and slowest with nitric acid.

In a subsequent work in 2019, [33] employed a modification process for alumina extraction, asserting that this approach reduces preparation costs by utilizing low-cost raw materials and enhances alumina extraction efficiency. Adopting a two-stage leaching method with acids, he considered experimental conditions involving 200ml of H_2SO_4 solution (40wt.%), 140°C, 3 hours of reaction time, 120ml of HCl for 0.5 hours, and NaOH solution (50wt.%). The conclusion drawn was that the alumina extraction rate reached 79.28%.

A notable distinction between the methods employed by [33] and [2] lies in the addition of NaCl salt to the kaolin prepared by [33] before calcination at a temperature of 700°C, despite both utilizing a two-stage leaching method.

Thermochemical activation using charcoal

The process for extracting pure alumina from kaolin using thermochemical methods has been significantly improved, as indicated by [34]. This method involves the calcination of kaolin along with limestone, accompanied by the addition of charcoal at a high temperature of 1360°C. [34] confirmed that, based on experimental results, the alumina percentage recovery rate increased from 77.7% to 87.04% with the addition of 1.5% charcoal. Consequently, it was concluded that charcoal serves as a cost-effective and energy-activated medium to enhance the recovery of alumina percentage from kaolin.

Pyro and hydro-metallurgical process

In 2019, [2] conducted an investigation into the pyro- and metallurgical processes for alumina extraction. The study utilized aqueous solutions of sodium carbonate as a leaching agent. [2]

suggested that both kaolin ore and limestone underwent a sintering process. This sintering process facilitated the dehydroxylation of kaolinite, the primary mineralogical phase in kaolin, and activated its transformation into metakaolin. According to the findings, metakaolin represents an amorphous Al-Si phase, allowing for easy leaching of alumina. The sintering occurred within the temperature range of 800-1400 °C for a duration of 1 hour. The investigation also explored the impact of briquetting pressure and sintering temperature on alumina recovery from kaolin [2] concluded that at 1360 °C and a briquetting pressure of 5 MPa, 87% of the alumina in the kaolin was successfully extracted.

Hydro thermal process

[35] conducted a study on alumina extraction from low-grade kaolin using lime and NaOH in a multi-stage hydrothermal process. The investigation involved the hydrothermal treatment of coal fly ash, focusing on the concentration of NaOH and the CaO/SiO_2 molar ratio, as discussed by [35], which was found to significantly influence alumina leaching.

[36] specifically examined the impact of NaOH concentration and CaO/SiO_2 molar ratio on alumina leaching from kaolin under fixed hydrothermal parameters of 180°C for 2 hours and a concentration of 10 g/L. The results justified that the leaching temperature, leaching time, and concentration of leaching agent in each stage were 180°C, 0.1 hours, and 20 g/L NaOH, respectively. Additionally, the molar ratio of CaO/SiO_2 was maintained at 0.7. The investigation confirmed the adequate decomposition of kaolinite, achieving a cumulative alumina leaching ratio of 72.09% after a three-stage hydrothermal process.

Solid state and phase transformation mechanism.

[37] investigated the mechanism of alumina extraction from kaolin through solid-state and phase transformation, involving the sintering of kaolin with limestone. The process utilized kaolin, sodium carbonate, and limestone. He observed that the optimal sintering temperature for inducing the solid-state reaction between clay and carbonate components, resulting in the highest alumina recovery of 80.49%, was 1360 °C. Under these optimal sintering conditions, kaolinite underwent dehydroxylation and transformation into metakaolinite, while calcite decomposed into calcium oxide. The ensuing reaction between these transformed components led to the formation of calcium aluminates, recognized as highly effective and generative precursor phases in the alumina extraction process. Table 2 presents the various leaching methods adopted by various researchers for the extraction of alumina from kaolin clay.

Table 2. An overview of various leaching methods for the extraction of alumna from kaolin

S/N	Extraction method	% alumina extracted by weight	Reference
1	Pyro and hydro metallurgical process	87	[6]
2	Hydrothermal process	72.09	[35]
3	Solid state and phase transformation	80.49	[37]
4	Thermochemical activation	77.7 - 87.04	[37]
5	Two way acid leaching method	79.28	[4]
6	Leaching	95-97	[2]
7	Hydrometallugical process	76-86	[5]

Industrial application of Alumina

Different authors have reviewed the importance and uses of alumina in various disciplines, and it has become necessary to evaluate the uses for industrial applications. This is presented and shown in Table 3.

Table 3. Different uses of alumina in different fields

S/N	Uses	Field	Properties	Reference
1	Bone replacement, Dental application, bone spacer, bone implant	Biomedical Application	Biodegradeable, bio inert and bio active, high density	[38]
2	Adsorbent	Water purification	Higher surface area and high thermal stability	[39]
3	Burner, thermocouple sheath, furnace lining, kiln furniture	Ceramics	refractoriness	[40]
4	Cups, plates, sheet glass,	Glass	High Mechanical strength	[41]
5	Spark plugs	Automotive	High di electrical strength	[41]
6	Abrasive, brake pads, ballistic armour, wear resistance	Structural alumina ceramics	Fracture toughness, elastic modulus, hardness	[42]
7	Porcelains	High voltage resistance	Hardness,	[41]

Conclusion

From the studies, it is observed that the fundamental structure of kaolinite comprises individual tetrahedral silica and octahedral alumina sheets linked by hydrogen bonding. In extractive metallurgy, the leaching process is widely employed due to its cost-effectiveness, environmentally friendly nature, low energy requirements, and its efficacy in treating low-grade ores [3]. The three common processes for extracting alumina from clays are: acid leaching with sulfuric acid, hydrochloric acid, or nitric acid. To extract alumina from calcined clay, sulfatization involving sintering clay with ammonium sulfate, followed by alumina extraction through leaching with hot water and alkali roasting through sintering clay with lime or soda, followed by alumina and silica extraction via leaching with hot water. Acids have been demonstrated to be more effective than bases in aluminum extraction [32].

From an industrial perspective, leaching calcined kaolin clay in hydrochloric acid offers notable advantages, including low silica solubility, the potential for selective crystallization of $AlCl_3$•$6H_2O_{25}$, and the recovery of hydrochloric acid from waste products for reuse in the process [43]. Recent research has concentrated on the extraction of alumina from kaolin through acid leaching, with extraction rates not surpassing 90%. However, efficient alumina extraction from kaolin by acid leaching faces challenges due to the stable structures of Al-O-Si and Si-O-Si , as well as the formation of hydrated silica during leaching reactions hindering the contact between the leaching agent and the leaching nucleus [32].

To enhance alumina extraction, kaolin is typically calcined in the temperature range of 500-850°C before leaching, increasing particle reactivity by rendering aluminum more soluble and removing organic material from the pores [32]. The kinetics of aluminum dissolution from calcined kaolin by hydrochloric acid are controlled by a liquid-film diffusion process [44]. Hydrothermal leaching of calcined kaolin by hydrochloric acid has been found to significantly enhance the alumina extraction rate, reaching approximately 98%, as it mitigates resistances caused by diffusion in the presence of hydrated silica [32]. Furthermore, the leaching of calcined kaolin in hydrochloric acid with fluoride ions present allows for higher extractions at lower roasting and leaching temperatures [45]. Above all, the extraction of alumina from calcined kaolin by leaching with hydrochloric acid depends on numerous variables, including kaolin fineness, calcination period and temperature, hydrochloric acid concentration, liquid/solid ratio, as well as leaching period and temperature [6]. It is therefore of most important for researchers to dive into the extraction of alumina from various kaolin because of its high extraction rate, purity, simplicity and efficiency together with its commercial viability.

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