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Characterization of Minim-Martap Bauxite and Its Extracted Alumina

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ARTICLE DETAILS

Article history:
Received as 01 November 2018
Accepted 13 November 2018
Available online 27 November 2018

Keywords: Bauxite Alumina Bayer Process Mini-Martap

ABSTRACT

Due to their chemical and mineralogical properties bauxites are composites with multiple applications. In this work, the properties of bauxite collected from Minim-Martap and its extracted alumina were evaluated. Alumina was extracted by adapting the Bayer process at laboratory scale while the characteristics of bauxite and alumina were variably characterized by determining the particle size distribution, relative humidity, loss on ignition, pH, EC, CEC, PZC, XRD, IR, and DSC-TGA. These results show that Minim-Martap bauxite gray bauxite with easily extractable alumina. This bauxite is acidic with a pH of 6.05, PZC of 6.91 and CEC of 15 Meq/100 g which is closer to that of kaolinite clays. This result was confirm by XRD which shows two types of kaolinites: $Al_2Si_2O_5(OH)_4$ with 83.24% and $H_4Al_2Si_2O_9$ with 42.47%, mullite $Al_2.25Si_{0.75}O_{4.875}$ with 67.32%, gibbsite (H_2SiO_4) with 83.92%. The bauxite is hydroscopic as evident from its humidity results. The IR spectroscopy of bauxite and alumina shows the presence of Al-OH and Si-OH which increase with acid treatment. Minim-martap bauxite and its extracted alumina can be used as raw materials for many industrial processes because of the different properties they possess as revealed by this study.

1. Introduction

The knowledge of the quality of bauxite is important for its application in different industrial processes. Bauxites are more and more used in many domains such as, alumina production, cements, glass industry, water depollution, etc. For its application in alumina production it is important to determine the silica modulus [1] which represents the ratio between the Al₂O₃ concentration and the SiO₂ in the bauxite. The higher the silica modulus, the higher the quality of the bauxite. Bauxite of modulus greater than 10 is considered to be of high quality, those between 8 and 10 are intermediary viable while those with modulus less than 8 are not economically viable [2]. The bauxites of high quality are becomming more and more uncommon consequently the necessity for the intensive investigation of the mineralogical and chemical composition of bauxites which are available in large quantities and profitable for the production of alumina. Out of the intended compound present in bauxite, it also contain other elements notably oxides of iron, silicon, titanium, calcium and magnesium. It can also contain a series of minerals in trace amounts such as: Na, K, P, Cr, V, Ga, Zn, Pb, Cu, Ni, Mn, and Co, etc.

The bauxite constitutes the source of revenue for certain countries (Guinea Bissao, China, Austria, etc.). In Cameroun there are 7 deposits of bauxite, among these deposits those of Ngaoundal and Minim-Martap are found to be more important with 1 billion tons. In this work, the properties of bauxite collected from Minim-Martap and its extracted alumina are evaluated. Results of this study will contribute in promoting the utilization of local natural resources for some local industrial processes thereby creating more jobs, improving the economy and the environment.

2. Experimental Methods

2.1 Sampling

Bauxite used was collected at Tehabal Aleo (Fig. 1) with coordinates 06° 53'14" N and 12°57'55" E at 12 m 74 altitude. The collection was done in boreholes dug during the bauxite exploration phase [3]. Collected bauxite was crushed and grounded to fine powder particles. This is to increase the

material's surface area thereby, improving mineral extractability and increases Bayer process efficiency. The crushed bauxite was washed with distilled water to remove undesirable material such as residual clays, which have a deleterious effect on the efficiency of the Bayer process. The washed sample was then dried at $100\,^{\circ}\text{C}$ for 24 hours and used for characterization study and extraction of alumina.

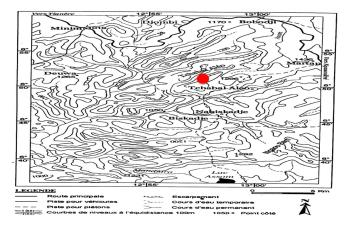


Fig. 1 Sampling zone

2.2 Production of Alumina

The method employed in the extraction of alumina from the bauxite was that adapted from the Bayer process [4] (Fig. 2). In which 20 g of finely crushed and grind activated bauxite (powder particles of diameter < 100 μm) were mixed with 40 mL solution 3 M NaOH in erlenmeyer flask and stirred (300 tr/min) for ten minutes. The reaction mixture was then heated at 80 °C for 20 minutes, allowed to cool down to laboratory temperature and filtered on the Whatmann filter paper No.1. 3 M solution of HCl was added to the filtrate collected in the becker to a pH between 5 and 6 (which is zone of stability for tetrahydroxyaluminates) to form a white precipitate. The precipitate obtained was filtered and dried overnight at 105 °C and calcined at 700 °C (in a muffle oven for 3 hours) to obtain alumina.

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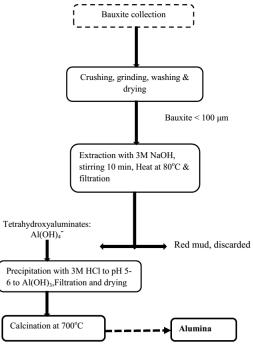


Fig. 2 Alumina extraction process employed

2.3 Characterization of Bauxite and Alumina

IR and X-ray diffraction technics were used to characterize the bauxite and alumina obtained. The bauxite was additionally characterized by the following methods. Sample was characterised by X-ray Fluorescence and the thermal stability using TGA-DSC analysis.

2.3.1 Particle Size Distribution

A master sizer HYDRO2000SM was used to study the particle size distribution of 50 and 100 μm samples.

2.3.2 Determination of Loss of Ignition and Relative Humidity

About 5 g of bauxite sample was dried at 105 °C until the mass became constant. To an empty crucible with mass M was added 5 g of bauxite sample with mass M1 and after drying at 105 °C a constant final mass M2 was obtained and relative humidity was calculated using Eq.(1). The crucible with mass M2 was then place in the furnace at 525 °C for 4 hour then weigh after cooling in a desiccator to obtain a constant mass M3 [13]. The loss of ignition was calculated using Eq.(2).

Relative humidity (%) =
$$\frac{(M_1 - M_2)X 100}{(M_1 - M)}$$
 (1)

Loss of ignition =
$$\frac{(M_2 - M_3)X100}{(M_2 - M)}$$
 (2)

2.3.3 pH, Electrical Conductivity (EC) and Point of Zero Charge

The pH and electrical conductivity (EC) of adsorbent were measured using a Hach HQ 40D multi pH meter. To measure these parameters, 0.5 g of the red mud was mixed with 10mL of distilled water and stirred (300 tr/min) for 20 minutes. After filtration, the pH and electrical conductivity of solution were measured [4]. The cationic exchange capacity (CEC) and the PZC were measured by acid titration [6]. The method used involves the titration of red mud using HCl to a neutral pH according to the following procedure: 1 g of red mud was mixed with 5 mL of distilled water and agitated for 15 minutes. This susupension was then titrated with a 0.1 M HCl solution to a neutral pH (pH = 7.0). The PZC was determined during titration, using zeta potential values measured by a Hach HQ 40D multipH meter. The equivalent gram of $\rm H_30^+$ necessary to neutralize the OH on red mud surface is the cationic exchange capacity. The pH at which zeta potential is zero equals the PZC.

3. Results and Discussion

3.1 Particle Size Distribution

Results of the particle size distribution of bauxite particles less than 50 and 100 microns are shown in Fig. 3. For the fraction of size less than 100

 μm , 10% has the size less than $6.139~\mu m$, 50% of particles has the size less than $79.536~\mu m$ and 90% less than $100~\mu m$. Their external surface is $0.51~m^2/g$. For the fractions less than $50~\mu m$, 10% of particles has the size less than $0.213~\mu$, 50% has the size less than $2.665~\mu m$ and 90% has the size less than $50~\mu m$. Their external surface is $7.85~m^2/g$. We note that the external surface of particles decreased as their size increased.

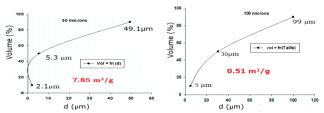


Fig. 3 Particle size distribution of bauxite (a) 50 microns and (b) 100 microns

3.2 Chemical Composition of Bauxite

Table 1 presents the result of chemical composition of natural Minim-Martap bauxite.

Table 1 Chemical composition of Minim-Martap bauxite

Const.	Al ₂ O ₃	Fe ₂ O ₃	SiO ₂	Ca0	K ₂ O	Na ₂ O	SO_3	TiO ₂	Mn_2O_3	P2O5	LOI*
Content (%)	31.69	34.25	0.42	0.45	0.03	-	0.31	7.85	0.05	0.27	24.78
*LOI= loss on ignition											

This analysis shows than the silica modulus (mSi) which is the factor of alumina versus silica oxide (SiO₂) is 75.45. The higher this ratio, the more precious the bauxite ore [7]. Minin-martap bauxite can thus be classified as gray bauxite characterized by very poor silica content (1% on average) and very rich iron oxide content (30% on average) [8].

3.3 pH, EC, CEC, Relative Humidity (Hr), PZC and LOI

Results of pH, cation exchange capacity, relative humidity, point of zero charge and loss on ignition are presented in Table 2. It is observed that although the bauxite is acidic, the surface is almost neutral (PZC = 6.91). Bauxite samples are mixture of aluminosilicates. The broken bonds on the surface of bauxite are covalent Al-O bonds and Si-O bonds which are ≡Si- 0^{-1} , \equiv Si-OH, \equiv Al- $0^{-1.25}$ functions [9], which can caused the absorption and electrostatic action with cationic reagents like ferric or aluminium ions.. The surface charge is positive when the pH of the medium is less than the pHzpc, and negative when the pH is above the pHzpc. The humidity of the bauxite is relatively high showing that Minim-martap bauxite is hygroscopic in character [10]. The CEC obtain is considered as high and it belongs to kaolins domain (between 12 and 14 méq/100g) (Al₂O₃.2SiO₂.2H₂O) [11]. The valency of silicion and oxygen in tetrahedral layer on one part, and of aluminium and oxygen in octahedral layer on another part are not saturated. To compensate these valences there are an introduction of water molecules and apparition of silanol group (Si-OH) or aluminol (Al-OH) which as a function of pH can pick up or liberate the protons. These protons can exchange with other cations. The number and the nature of charges is directly link to the pH. The loss on ignition which represents the total organic material and other impurities determined from Eq.(2) and X-ray fluorescence are basically the same.

Table 2 pH, EC, CEC, Relative humidity (Hr), PZC and LOI of Minim-Martap bauxite

рН	EC	CEC	Hr	LOI	PZC
6.05	0.86	15.00 Meg/100g	22.80	24.9	6.91

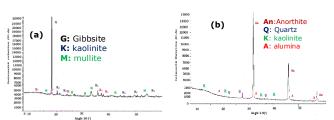


Fig. 4 XRD patterns of Minim-Martap bauxite (a) and extracted alumina (b)

3.4 Mineral Composition of Bauxite and Extracted Alumina

The X-ray diffraction reveal that the bauxite (Fig. 4a) is constituted principally of two types of kaolin, $(Al_2Si_2O_5(OH)_4$ (83.24%) and $H_4Al_2Si_2O_5$ (42.47%), mullite 67.32% and 83.92% of gibbsite (G). The extracted alumina (Fig. 4b) by Bayer process yielded 12.90% which shows the noncomplete extraction as well as low calcination temperature used [4]. This

shows that the alumina remain associated with kaolins $Al_2Si_2O_5(OH)_4$ (18.51%) and anorthites $CaAl_2Si_2O_8$ (An) (40.10%). The high mineral content of the bauxite and its extracted alumina shows they can be used for many industrial applications.

3.5 Infra-Red Spectroscopy

To better understand the superficial properties of natural bauxite and extracted alumina surfaces, they were all stirred in 200 mL solution of 2 N HCl for 20 minutes and filtered using Whatmann filter paper No.1. The IR spectra of natural and activated bauxite are shown in Fig. 5a while the IR spectra of extracted and its acid treated alumina sample is presented in Fig. 5b. Broad bands are observed between 3000 and 3600 cm⁻¹ for all the samples. This is attributed to valence vibration of OH bond of Al-OH group. Equally the observed bands at 1010, 965, 742 cm⁻¹ are cause by tetrahedral group of Si-O-Si and deformation vibration of Si-O link. However, the IR spectra of alumina show a more smooth pattern (Fig. 5b) with less number of peaks showing the elimination of many other groups compared to the bauxite sample (Fig. 5a). The peaks appear more intense with acid treated bauxite and acid treated alumina bauxite than their parent samples indicating the elimination of impurities and more purification of the material with acid treatment.

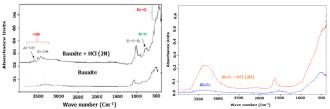


Fig. 5 IR curves of bauxite (a) and alumina (b)

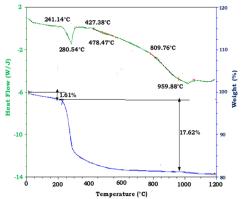


Fig. 6 DSC-TGA curves of bauxite (green is DSC and blue is TGA)

3.6 DSC-TGA Analysis

The TGA (Thermal gravimetric analysis) diagram of bauxite sample (Fig. 6) shows three steps for the weight loss. The first curve has occurred in the range of 20 to 280 °C (Weight loss about 1% of total weight), corresponding to loss of surface water. The $2^{\rm nd}$ and the $3^{\rm rd}$ breakings occurred in the range of 427-478 °C and 809-959 °C respectively corresponding to a total weight loss of about 17%. This is attributed to loss

of chemically adsorbed water. At 280.54 °C chemically bound water is removed in the form of OH ions from gibbsite, at 478.47 °C from kaolinite to form $\gamma\text{-Al}_2O_3$ and irregular metakaolinite Al $_2\text{Si}_2O_7$ [12] and at 959.88 °C to form mullite.

4. Conclusion

The results show that Minim-Martap bauxite has gray bauxite with less than 1% silica content and thus having a high silica modulus (mSi) index. It therefore has high commercially value. Alumina can easily be obtained from this bauxite. It has a pH of 6.05 and PZC of 6.91 and the CEC of 15 $M_{\rm eq}/100$ g which is closer to that of kaolinite clays. This result was confirm by XRD which shows two types of kaolinites ${\rm Al}_2{\rm Si}_2{\rm O}_5({\rm OH})_4$ with 83.24%, (H4Al2Si2O9) with 42.47%, mullite ${\rm Al}_{2.25}{\rm Si}_{0.75}{\rm O}_{4.875}$ with 67.32%, gibbsite with 83.92%. The IR spectroscopy of bauxite and alumina revealed the presence of the groups OH of Al-OH and Si-OH at 3690 cm-1 and 3619 cm-1 respectively. The bauxite as well as its extracted alumina has many properties that can favour their different applications.

Acknowledgement

The authors express their special gratitude to the European membrane institute, France for bauxite ore analysis, the Laboratory of Analytic Chemistry University of Yaounde I for IR analysis, Cimencam Cameroon where XRF analysis were conducted. We also thank the inhabitants of Minim and Martap who guided us through the collection of the bauxite.

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