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Decreasing Yield and Alumina Content of Red Mud by Optimization of the Bauxite Processing Process

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Abstract Bauxite produces 60-120 million tons of toxic red mud annually during its processing to alumina. Thus, in this work, the modeling of the bauxite processing process to reduce quantity of red mud produced and with low alumina content was envisaged. Bauxite from Minim-Martap was processed by adapting the Bayer process under laboratory conditions. This process was then optimized to obtain conditions that yield less red mud with reduced alumina content. This was done using the Doehlert experimental design with three factors: stirring time, sodium hydroxide concentration and temperature. X-ray diffraction (XRD) and X-ray fluorescence (XRF) were used to characterize obtained alumina, red mud and crushed bauxite. Model process equation obtained from optimization results shows that increasing the tested parameters lead to production of low amount of red mud (temperature and sodium hydroxide concentration were the main factors that influenced the process). The model obtained described the process adequately with a 92% coefficient of determination, low absolute average deviation of 0.004 and strong agreement between theoretical and experimental responses. The optimum conditions gave a red mud yield of 86% with 13% alumina compared to a non-optimized process with 98% red mud yield and 16% alumina content. The fact that increasing tested parameters reduced red mud yield however, implies high amount of alumina is produced, thus a significant economic and environmental advantage for the aluminum industry. It was concluded that optimization of bauxite processing process reduced red mud produced and its alumina content.

Keywords Bauxite, Bayer process, Experimental design, Minim-Martap, Red mud

1. Introduction

Red mud is the caustic by-product stream from alumina production via the Bayer process, often colloquially referred to as red mud or bauxite residue or Bayer process tailings [1, 2]. About 90% of raw bauxite ore goes into the waste as alkaline red mud slurry during processing [3] as for every ton of alumina produced, between 1-2 tons (dry weight) are produced depending on the bauxite source and alumina extraction efficiency [4, 5]. Globally, about 60-120 million tons are produced annually [6]. This may lead to serious pollution of the surrounding soil, air and groundwater due to its high pH (10-13) [7, 8]. Depending upon jurisdiction, untreated bauxite residue may be classified as hazardous primarily due to its alkalinity rather than heavy metal or naturally occurring radionuclide content [9]. However, red mud contains a number of valuable metals and minerals (from parent bauxite and those introduced during the Bayer process) like aluminium, iron, silica, calcium, titanium and some minor constituents namely: Na, K, Cr, V, Ni, Ba, Cu, Mn, Pb, Zn etc. The typical constituents of red mud (% w/w) are: Fe_2O_3 (30-60%), Al_2O_3 (10-20%), SiO_2 (3-5%), Na_2O (2-10%), CaO (2-8%), TiO_2 (trace-10%) [10, 11], depending on the type and quality of ore used and the process parameters. Red mud has been used for metal recovery, building material, ceramics production, catalysis, soil amendment, pigments and paints, water treatment [8-9,12-16] etc.

Bauxite the primary source of over 99% of world aluminium [17] is a naturally occurring mixture of minerals rich in hydrated aluminum oxides (40-60%). The major impurities of bauxite are the oxides of Fe, Si, and Ti and trace amounts of metals which constitute red mud [11]. The most important Al- containing bauxite minerals are gibbsite [$Al(OH)_3$], boehmite [γ-AlO(OH)], and diaspore [α-AlO(OH)]. Based on their mineralogy, bauxites can be divided into two types; Lateritic bauxites, are predominately gibbsite and to a lesser extent boehmite and comprise approximately 90% of the world's exploitable bauxite reserves while karst bauxites are principally boehmite, and diaspora [18]. Though alumina can be produced from bauxite under alkaline conditions using lime (Lime Sinter process), sodium carbonate (Deville Pechiney process), at high temperature in reducing environment with presence of coke and nitrogen (Serpeck process), the alkalinisation by

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the use of sodium hydroxide (Bayer process) is the most economical process which is employed for purification of bauxite if it contains considerable amount of Fe_2O_3 [19]. It is responsible for 90% of the world's alumina production from bauxite [20]. It is a high temperature and high pressure selective dissolution process extracting gibsitte and/or boehmite from bauxite by dissolving these constituent in hot concentrated NaOH and 106-240°C and at 1-6 atm pressure [9, 20-22]. After bauxite dissolution or digestion, the $NaAl(OH)_4^-$ rich solution is separated from the remaining less soluble materials such as iron oxide and silica, known in the industry as "red mud or bauxite residue" [20]. $NaAl(OH)_4^-$ is precipitated to give Al $(OH)_3$ which is calcined at 1,000–1,200°C to give Al_2O_3. Lateritic bauxites are easier to digest than karst bauxites using less severe conditions of caustic concentration, temperature and/or holding times [18].

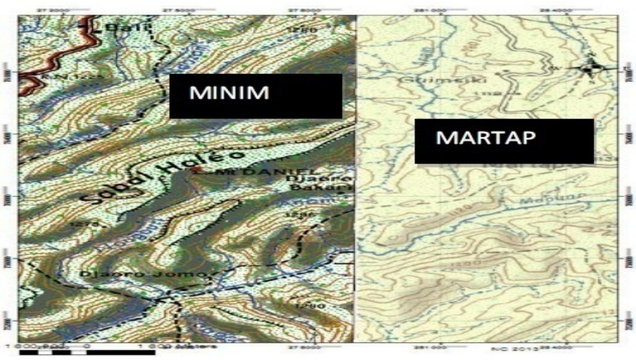
Bayer process is entirely a large scale industrial process, unfortunately the nature and scope of the information about it is owner and/or refinery specific and not consistent in either form or content. As, each refinery has unique operating details with respect to red mud technologies, management and engineering practices [23] thus, placing a severe limitation on the ability to collect systematize and interrogate information on the process. The above review shows that the process is influenced principally by the parameters; holding time, bauxite type, sodium hydroxide concentration and temperature and pressure. The improvement of process conditions can reduce the about 90% of raw bauxite ore that goes into the waste and yields more alumina.

Published information on the laboratory processing of bauxite in general and particularly bauxite from Cameroon as well as the optimization of the process is very scarce. It is estimated that Cameroon has the 6th world bauxite reserves [24], with approximately 1.8 billion tons from which 1 billion tons are estimated for the two groups of deposits situated in the Minim-Martap and Ngaoundal [25]. But there is no bauxite exploitation activity in Cameroon yet. This work is thus, aimed at adapting the Bayer process at laboratory level to process bauxite from Minim-Martap and optimizing the process parameters in view of identifying process conditions that yield less amount of red mud with improved properties (reduced alumina content) and help raise awareness on risk of bauxite processing in anticipation of lateritic bauxite [26] processing to start in Cameroon.

2. Materials and Methods

2.1. Sampling of Bauxite and Its Preparation for Processing

Samples, mainly red bauxite were collected at Sabal Haleo (06°27'27'' N 12° 59' 28'' E) figure1a from bore holes (about 10 meters deep) figure 1b, dug by Cameroon Alumina Limited (CAL) during exploration.



a)

b)

Figure 1. a) Map of the sampling zone b) Bore holes where bauxite was collected

Equal samples of bauxite taken at different positions were thoroughly mixed to obtain a representative starting material. It was then 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°C for 24 hours from where bauxite particles lower than 100 µm were prepared for further usage in bauxite processing.

2.2. Processing of Minim-Martap Bauxite

A trial extraction process was performed using the procedure described by Benhamou *et al.,* 2008 and Excoffier, 2009 [27, 28]. Results of the trial process (which gave 98% red mud yield, rich in alumina) oriented the optimization of the extraction. For trial process, 20 g of finely crushed and grinned bauxite were mixed with a prepared 120 mL solution of 3M NaOH in a 250 mL capped Erlenmeyer flask. This mixture was stirred (300tr/min) for 10 minutes and then, heated at 80°C for 20 minutes (on a Thermo Scientific Cimarec stirring hot plate, model: SP 131320-33). After this period, the slurry was allowed to cool down to laboratory temperature and the slurry was filtered over Whatmann filter paper N° 1. Red mud was collected on the filter paper while the filtrate was collected in a 500 mL Becker where a prepared 3M solution of HCl was added drop wise to the filtrate to a pH between 5 and 6 (which is the zone of stability for aluminium hydroxide), measured using a Hach HQ 40D multi pH meter. The precipitate obtained was filtered and dried overnight at 120°C and calcined at 1000°C (in a muffle oven for 3 hours) to obtain alumina. Red mud obtained was washed with distilled water several times and dried at 100°C overnight, stored at ambient temperatures and used without further treatment.

2.2.1. Optimization of the Minim-Martap Bauxite Processing Process

The Doehlert design was used because of its numerous advantages over other designs [29]. Particularly, it permits to follow a sequential manner in studying a response surface of second degree. Also the matrix is flexible for adding new parameters or extending experimental domains, without restarting the experiments that has already been done. Moreover, polynomial equations with and without interaction are used as models. Then, the coefficient of determination (R^2) and absolute average deviation (AAD) are used to investigate the adequacy of the proposed models. The selection of the experimental design was based on the assumption that bauxite processing process is affected by 3 variables: stirring time (X_1: 10 - 60 min), sodium hydroxide concentration (X_2: 1 -3 mol/L), digestion temperarure (X_3: 80 - 150°C), Table 1.

Table 1. Factors and levels to be used in Doehlert Optimization design

Factors	Low level (−1)	Middle (0)	High level (+1)
Time of agitation (mins), X_1	10	35	60
Concentration NaOH (mol/L), X_2	1	2	3
Temperature (°C), X_3	80	115	150

Values in bracket are coded values

Based on the coded values of Doehlert experimental matrix, the real values all presented in Table 2 were calculated using the equations 1-3.

$$U_i = U_i^o + x_i \Delta u \quad (1)$$

Where U_i is the value of natural variable i, U_i^o is the central value of natural variable i, x_i is the coded value of variable i, Δu is the increment which could be calculated from equation

$$\Delta u = \frac{U_i - U_i^o}{x_i} \quad (2)$$

Then the real value in the domain, X_i is calculated from equation

$$X_i = U_i^o + x_i \Delta u \quad (3)$$

These transformations are those of Mathieu and Phan-Tan, 1995 [30].

A total of 17 different experiments (with constant mass of bauxite, 20g) were performed according to the experimental design for three parameters. The response ((Y) measured was: red mud yield in percentage. The analysis of variance (ANOVA) was generated and the effect and regression coefficients of individual, quadratic and interaction terms were determined. The regression coefficients were then used to make statistical calculations to generate response surface graph from the regression model. The experimental design obtained leads to the development of a model equation with calculated coefficients. This model is used in the surface response methodology in complete quadratic form, equation 4:

$$Y_j = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i=1}^{k=1} \sum_{j=i+1}^{k} b_{ij} X_i X \quad (4)$$

Where: Where Y is the response, b0 = constant term, bi = linear constant effect,

bii = quadratic constant effect, bij = interaction constant effect and Xi and Xj are the independent parameters. Using Statgraphics 16, the coefficients and the mathematical equation relating the response factors with the independent parameters were generated and used in the determination of the theoretical zones of optimal response.

2.3. Characterization of Bauxite, Alumina and Red Mud

The alumina obtained from trial process was analyzed by X-ray diffraction (XRD) to determine the efficiency of the extraction process. Chemical composition of red mud obtained under the two processing conditions (trial and optimum) was determined by X-ray Fluorescence (XRF) with aim of controlling red mud alumina content from the two conditions. The chemical composition of the bauxite used was also determined by XRF. The mineral composition of red mud was also characterized by XRD.

3. Results and Discussion

3.1. Modeling of the Process

The design matrix in coded and real values, together with the experimental values of the responses (red mud yield in %), theoretical yields of red mud generated from statgraphics and calculated values of ADD obtained from equation 5 below at different conditions, are presented in table 2.

$$AAD = \left\{ \left[\sum_{i=1}^{z} \left(\left| y_{i.exp} - y_{i.cal} \right| / y_{i.exp} \right) \right] \Big/ Z \right\} * 100 \quad (5)$$

Where Yi.exp and Yi.cal are the experimental and calculated responses respectively, Z is the number of experimental runs.

Table 2. Effect of stirring time (X_1), sodium hydroxide concentration (X_2) and digestion temperature (X_3) on the processing of Minim-Martap

N° exp	Coded values			Reel values			Response		AAD
	X_1	X_2	X_3	X_1	X_2	X_3	Y_{ex} (%)	Y_{th} (%)	
1	0	0	0	35	2	115	80.50	81.20	0.05
2	1	0	0	60	2	115	74.10	72.53	0.13
3	0.5	0.866	0	47.5	2.866	115	79.10	77.65	0.11
4	0.5	0.289	0.816	47.5	2.289	143.56	64.10	67.13	0.28
5	-1	0	0	10	2	115	73.20	74.78	0.13
6	-0.5	-0.866	0	22.5	1.134	115	85.00	86.45	0.1
7	0.5	-0.866	0	47.5	1.134	115	85.50	85.93	0.03
8	-0.5	0.866	0	22.5	2.866	115	79.80	79.38	0.03
9	-0.5	-0.289	-0.816	22.5	1.711	86.44	92.50	89.48	0.19
10	0.5	-0.289	-0.816	47.5	1.711	86.44	91.40	92.55	0.07
11	-0.5	0.289	0.816	22.5	2.289	143.56	73.60	72.45	0.09
12	0	0.577	-0.816	35	2.577	86.44	95.00	96.88	0.12
13	0	-0.577	0.816	35	1.423	143.56	85.20	83.33	0.13
14	0	0	0	35	2	115	78.70	81.20	0.19
15	0	0	0	35	2	115	82.70	81.20	0 .11
16	0	0	0	35	2	115	78.80	81.20	0.18
17	0	0	0	35	2	115	85.30	81.20	0.28
				Average AAD					**0,004**

Y_{ex} = Experimental yield, **Y_{th}** = Theoretical yield, **AAD** = absolute average deviation, N^0 exp= experimental run

By applying ANOVA and regression analyses in stat graphics software using the experimental results in Table 2, the coefficient of determination (R^2) was determined and a polynomial model that explain the variations of the red mud yield was generated according to equation 6 and presented as

$$\textbf{Quantity of RM (\%)} = 81.2 - 0.95X_1 - 4.39X_2 - 11.31X_3 - 7.35X_1^2 + 3.98X_2^2 + 4.65X_3^2 - 0.69X_1X_2 - 4.53X_1X_3 - 9.51X_2X_3 \quad (6)$$

The negative value of the coefficient of the main factors (coefficients of X_1, X_2 and X_3) shows that increasing these parameters lead to production of low amount of red mud. This observation implies high amount of alumina is produced. However, the coefficient of X_3 is three times of magnitude greater than that of X_2 and is more than twelve times of magnitude greater than that of X_1. This means that temperature (X_3) is the main parameter and sodium hydroxide concentration(X_2) comes in the second position. Stirring time has little effect on red mud production.

The high value of negative X_1^2 coefficient (-7.35) means increasing the stirring time could lead to little decrease in the amount of red mud produced. At the same time, the quadratic effect of sodium hydroxide concentration (3.98) and that of temperature (4.65) implies that these two parameters should be kept in reasonable interval to have low yield of red mud. This observation is also valid considering the interaction factor between stirring time and temperature and between sodium hydroxide concentration and temperature. It is thus, obvious from these analyses that processing temperature is the key factor.

The R^2 statistic of 92.95% form ANOVA indicates that the fitted model explains 92.95% of the variability of quantity of red mud produced. Statistically adjusted R-squared, which is preferable to compare models with different numbers of independent variables, is 83.88%. Therefore, it can be assumed that the proposed model does not explain at least 7% of the experimental results.

3.1.1. Validation of the Model

The value of coefficient of determination for the red mud yield from Minim-Martap bauxite processing is 92.95%. The value of R^2 shows that the proposed model is adequate. Mendenhall, 1975 [31] reported that the closer the value of R^2 to the unity, the better the empirical models and the actual data. In fact, Joglekar and May, 1987 [32] suggested that, for a good fit of a model, R^2 should be at least 80.0%. On the other hand, the low absolute average deviation (AAD) value red mud yield from Minim-Martap bauxite processing (0,004) confirms the adequacy of the model as it must be as small as possible [33]. The validity of the model equations was also tested by drawing a regression line between experimental and theoretical responses for red mud yield (figure 2). The agreement (or low residues) between the responses also validates this model.

Therefore the model described the process adequately and could be used to generate surface response curves to explain the influence of the independent factors on the responses studied. This is aimed at visualization of the effects of stirring time (X_1), sodium hydxoxide concentration (X_2) and digestion temperature (X_3) on the yield of red mud from processing of Minim-Martap bauxite is presented in Fig. 3 using response surface graph.

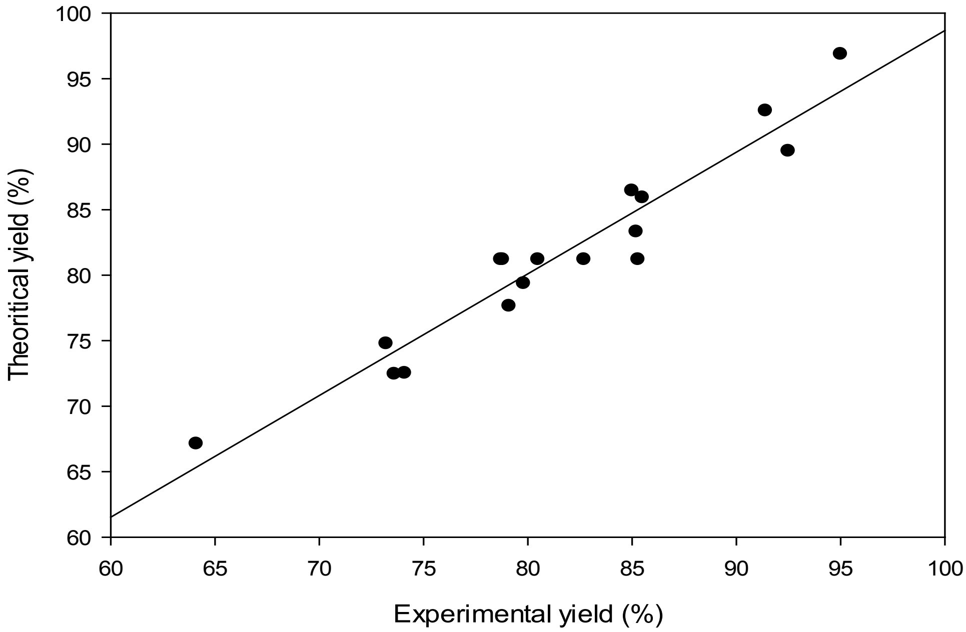


Figure 2. Correlation between experimental and theoretical yields (%) of red mud from Minim-Martap bauxite

When digestion temperature was kept constant, the response surface figure 3 indicated that the red mud yield increased with an increase in stirring time and sodium hydroxide concentration and then decreased dramatically. This decreased may be due to the partial dissolution of some components of red mud after prolonged contact with sodium hydroxide.

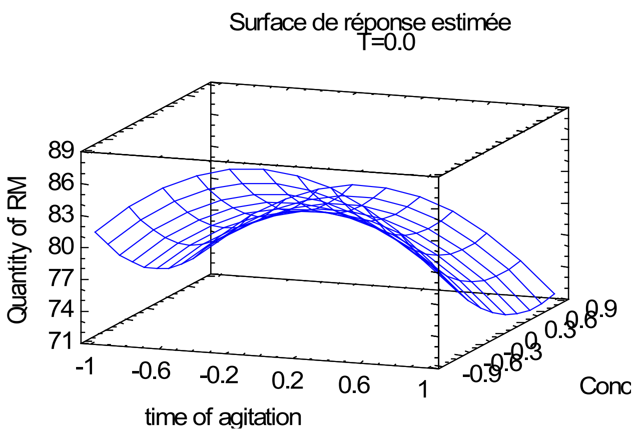


Figure 3. Response surface graph showing the variation of red mud yield from Minim-Martap bauxite processing as a function of stirring time and sodium hydroxide concentration

The response surface methodology was used in the optimization of the process. Using the numerical analysis and the model equations (equations 2 and 3), the theoretical optimum conditions were found as follows: If the domain of X_1, X_2 and X_3 are (10 - 60 min), (1 - 3 mol/L) and (80 - 150°C) respectively, then -0.075, 0.866 and -0.797 are the initial coded values of X_1, X_2 and X_3 respectively for optimum conditions corresponding to the given domains: $-1 \leq X_1 \leq 1$; $-0.866 \leq X_2 \leq 0.866$; $-0.816 \leq X_3 \leq 0.816$. Then, the optimum conditions depicted a stirring time of 33.12 minutes, hydroxide concentration of 3 mol/L and digestion temperature of 87.12°C respectively. The optimum red mud yield was found to be 86%.

3.2. Characterization

3.2.1. Mineral Composition by XRD

The XRD diffraction patterns of Al_2O_3 from the trial process is shown in figure 4 below, showing the presence of high amounts of alumino-silicate minerals (Kaolinite, $Al_2Si_2O_5(OH)_4$ (18.51%), Quartz, SiO_2 (19.27%), Anorthite, $CaAl_2Si_2O_8$ (40.10%), and only 12.90% of Al_2O_3.

Similarly, the XRD diffraction pattern of red mud obtained was rich in the following minerals: hematite, gibbsite, goethite, quartz, anatase, calcite and diaspore as presented in figure 5 below.

These XRD results confirm the high yield of 98% red mud obtained after filtering, washing and drying compared to Balomenos *et al.,* 2011 [34] and Deger and Gulfen, 2007 [35] findings that, the quantity of red mud generated varies between 55-65% of bauxite. However, this 98% yield is closer to studies of [3], that 90% of raw bauxite ore goes into the waste as alkaline red mud slurry during processing. The high red mud yield and low alumina yield contaminated with other minerals is likely due to the following:

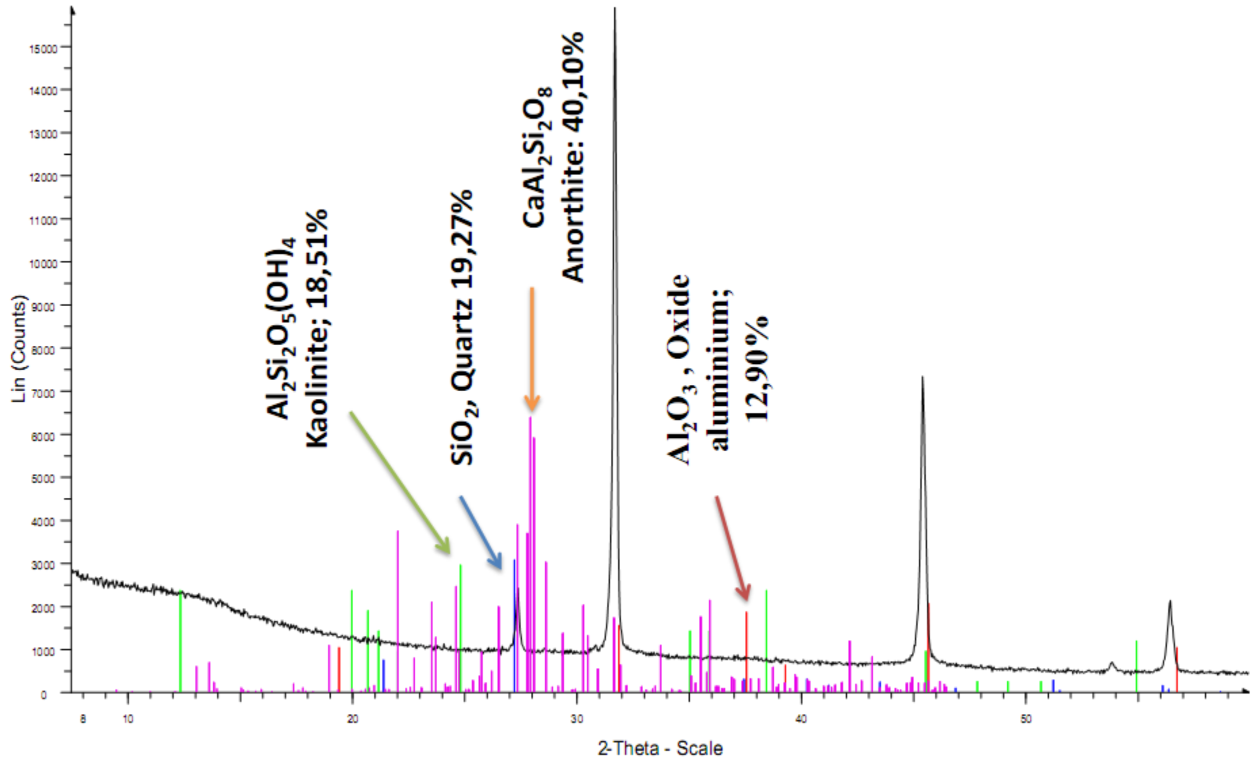


Figure 4. XRD pattern of alumina obtained from Minim-Martap bauxite

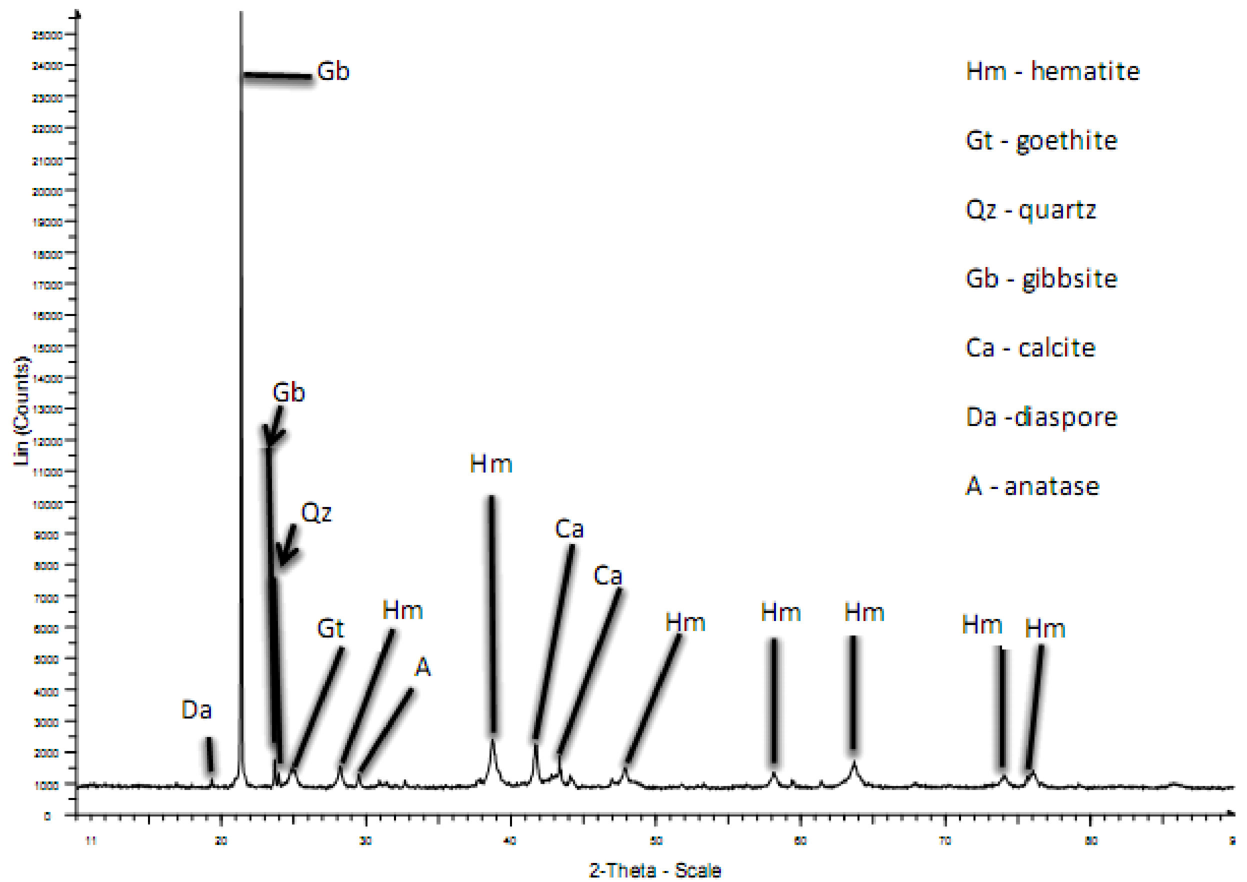


Figure 5. XRD pattern of red mud obtained from Minim-Martap bauxite

(i) Stirring which we considered as our predesilication phase was conducted for just 10 minutes instead of several hours [18]. This resulted in none or very low removal of silica from the aluminosilicates as predesilication is the removal of silica as desilication products (DSP) with chemical formula, $3(Na_2O.Al_2O_3.2SiO_2.nH_2O).Na_2X)$ n =0-2 and X = CO_3^{2-}, SO_4^{2-}, $2OH^-$, $2Cl^-$ [12] from alumino-silicate minerals prior to digestion particularly for bauxites with low reactive silica content [11]. This is confirmed by the results of XRD of Al_2O_3 that gave only 12.90% of Al_2O_3, the rest being the different aluminosilicates. This is also confirmed by the presence of sharp gibbsite peak, diaspore (AlOOH) and other alumino-silicate minerals on red mud. This significantly reduces the quantity of alumina produced. The reaction which takes place during predesilication is given by equations 7 and 8 that are:

$$3Al_2Si_2O_5(OH)_4 + 18NaOH \rightarrow 6Na_2SiO_3 + 6NaAl(OH)_4 + 3H_2O \quad (7)$$

$$6Na_2SiO_3 + 6NaAl(OH)_4 + Na_2X \rightarrow Na_6[Al_6Si_6O_{24}].Na_2X + 12NaOH + 6H_2O \quad (8)$$

X = CO_3^{2-}, SO_4^{2-}, $2OH^-$, $2Cl^-$ or a mixture of all [12, 18].

(ii) Secondly, digestion reactions (e.g. Gibbsite: $Al_2O_3.3H_2O+2NaOH \rightarrow 2NaAl(OH)_4^-$ + impurities) were done at 80°C against 100-150°C for gibbsite digestion [2] probably resulted in incomplete alumina extraction from different minerals in the bauxite. This is also confirmed by the presence of sharp gibbsite peak and diaspore (AlOOH) on red mud. Diaspore is extracted at very high temperature (200-245°C) and pressure [9]. Meyer, 2004 [36] report that, the bauxite of Minim-Martap is principally lateritic bauxite. However, the rich iron content of red mud confirms Briger, 2010 [37] findings that, the surface of Minim-Martap Bauxite is iron-rich.

3.2.2. Chemical Composition (Expressed as Oxide) by XRF

The chemical composition of bauxite (100 µm), red mud produced by trial process and under optimum conditions (using 100 µm bauxite particles) is shown in Table 3 below. While the quantities of trace elements remain almost the same after bauxite processing, those of Si, Al, Fe, Na and Ca show significant variations from those of the parent bauxite. It is also seen from this table that red mud obtain under optimum conditions has smaller amount of Al_2O_3 (13%) compared to 16% of under trial conditions and high amounts of Fe and Si oxides (56 and 4% respectively) compared to 48 and 2.76% respectively for trial process. Thus, optimization improves Bayer process by reducing alumina content in red mud which is the main goal of the process. Na_2O and Cao are only introduced in red mud during bauxite processing as it is absent in bauxite. It probably represents the DSP formed during predesilication.

4. Conclusions

The optimization of the processing of bauxite from Minim-Martap under laboratory conditions to obtain low quantity of red mud with reduced alumina content was investigated. From the results obtained, the following conclusions are drawn:

- Increasing the stirring time, temperature and sodium hydroxide concentration leads to production of low amount of red mud.
- The amount of red mud decreased from 98% in a non-optimized process to 86% with optimization.
- Red mud alumina content also decreased from 16% in a non-optimized process to 13% with optimization.
- Temperature and sodium hydroxide concentration are the main factors that influenced the process.
- The model obtained described the process adequately as evident from values of coefficient of determination (92%), low absolute average deviation (0.004) and strong agreement between experimental and theoretical responses.

Results of this study constitute the long-term objective on red mud management from production to disposal because each refinery has unique operating details with respect to red mud technologies, management and engineering practices thus, placing a severe limitation on the ability to collect systematize and interrogate information on the process.

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Table 3. Comparison of oxide composition of bauxite and red muds produced under different conditions from bauxite of Minim-Martap

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	TiO_2	Mn_2O_3	P_2O_5
Bauxite, 100 µm	0.54	27.41	29.96	…..	0.01	0.02	…..	0.26	4.70	0.03	0.12
RM, trial	2.76	16.41	48.59	0.53	0.08	0.07	4.23	0.12	3.76	0.03	0.11
RM, optimize	4.04	13.00	56.79	2.27	0.09	0.07	3.63	0.18	3.94	0.05	0.10

RM, trial: red mud obtained from Minim-Martap bauxite during trial studies or non-optimized studies
RM, optimize: red mud obtained from Minim-Martap bauxite during optimization studies

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