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Reduction of Sulphur Content of AGBAJA Iron Ore using Hydrochloric Acid (HCL)

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Abstract

Iron ores are used in blast furnace for the production of pig iron; AGBAJA Iron ore has an estimated reserve of over I billion metric tonnes. Unfortunately, this large reserve cannot be utilized for the production of pig iron due to its high sulphur contents. This work studied the reduction of sulphur content of AGBAJA iron ore. Acid leaching methods were used to reduce sulphur contents of the ore. Sulphuric acid of different concentrations were used at various leaching times, acid concentrations and particle sizes. Atomic Absorption Spectrophotometer, X-ray fluorescence spectrophotometer, Digital muffle furnace and Absorbance-concentration technique were used for experimentation and chemical analysis. The reduction of the sulphur content of AGBAJA Iron Ore using Acid leaching process experiments were carried out at the National Metallurgical Development Centre (NMDC), Jos in Plateau State of Nigeria. Sulphur is one of the main harmful elements in ferrous metallurgy and it affects the quality of iron and steel produced. At present, Nigeria has some large iron ore deposits including AGBAJA which bear tremendous iron ore with high sulphur content of 0.12%. Central composite design technique was applied to obtain optimum conditions of the processes. Surface response plots were obtained. The percentage degrees of reduction of sulphur content of AGBAJA Iron ore were found to increase with increase in acid concentration and leaching time and a decrease in particle size for the three acids. The experimental results for percentage removal of sulphur are 85.56% the optimum % removal of sulphur is 89.66%. The result of this work has shown that AGBAJA Iron Ore if properly processed can be used in our metallurgical plants and also can be exported since sulphur contents of the ore have been reduced drastically.

Keywords: Reduction; Sulphur content; AGBAJA; Iron ore and hydrochloric acid (HCL)

Introduction

The AGBAJA ore is a fairly lean, acidic iron ore with high sulphur content. The ore is an earthy, friable material containing magnetite and goethite, together with minor aluminosilicates and phosphates of iron and aluminium. The ore contains approximately 54% iron and shows thermal effects associated with the elimination of water. The texture and chemical composition of the AGBAJA ore suggests that despite its magnetic character it cannot be easily beneficiated for use in a direct, non-conventional iron making process. The various slag-forming constituents (silica, alumina, lime and magnesium oxide) are so closely associated with the iron-bearing constituents that separation is impossible to achieve by simple physical means. Furthermore, the high sulphur content (about 0.12%) would probably give rise to problems in steel production unless a conventional, oxidizing; liquid-metal process (such as basic oxygen steelmaking) is used following blast-furnace production of liquid iron. For the AGBAJA iron ore the basicity value is very low (approximately 0.035) and hence the ore would need significant additions of lime, limestone or a lime-rich ore to make a self-fluxing sinter or pellet suitable for iron production. The reduction/removal of the high sulphur content can be achieved through the process of leaching using nitric acid.

Statement of the problem

AGBAJA iron ore is the largest iron deposit in Nigeria with an estimated reserve of over 1 billion tonnes. This iron ore has high relative high sulphur content. Consequently, the iron ore deposit is abundant in both research work and exploitation. The high sulphur content in steel making cause brittleness or crackability depending on the type of Steel products. The sulphur content in the AGBAJA iron ore has a detrimental effect on the steel making process using the ore as raw materials in steel making. It is therefore, necessary to drastically remove/reduce the sulphur content.

Purpose and goals

The purpose of this work is to carry out experiments on the reduction of sulphur content of AGBAJA iron ore using acid leaching. Leaching of lean ores or complex ore in different acids has proved successful for several years. However, the leaching of sulphur contaminated iron ore has made a very limited progress. This underscores the ongoing intense research in the area for several decades. Depending upon the degree of association of sulphur with the minerals in the iron body, iron ore can therefore the sulphur content can be reduced using the following mineral acids which are often used as leaching agent of Hydrochloric acid (HCl). The purpose therefore is to use all the three mentioned acid leaching with a view to reducing/removing of sulphur content in order to making it useable for steel making process in the Blast Furnace.

In Nigeria today, it is a known fact that most of the steel industries are not functional due to lack of infrastructure. Despite all the endowment of the abundant mineral resources that are readily available due to natural blessings. This project work was carried out

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with a view to making use of this minerals in which the iorn ore falls into the materials used in the production of iron and steel. Nigeria is endowed with large deposits of iron ores; however, the ores are not suitable for the production of Direct Reduced Iron (DRI). The ore closest to specification in terms of sulphur content is the Itakpe iron ore deposit which has a reserve of about 300 million tonnes. The ore is beneficiated from its natural occurrence of 36.8% Fe and 45.8% acid gangue (SiO_2) + Al_2O_3 to 63% Fe and 8% acid gangue by the National Iron Ore Mining Company (NIOMCO). The latter parameters are only suitable for the Blast Furnace (BF) plant located at Ajaokuta Steel Company Limited which is yet to be completed. However a sister plant located at Aladja which has been in operation since 1982 requires some 1.5 million tonnes of high quality iron ore (66% Fe and <3.5% of SiO_2 + Al_2O_3).

The supply of the high grade ore has so far been met only through importation which imposes high financial constraint on the operation of the Aladja plant. It can be found that at a landing cost conservatively put at 104 dollar per tonne of the ore and at even 50% capacity utilization. More than 10 billion Naira will be expended on the procurement of the ore alone. In order to meet the challenges posed by the importation of ore to meet the local demands at the steel industries in Nigeria, it therefore becomes imperative to investigate the suitability of the AGBAJA Iron ore with a view to carry out the reduction of the sulphur content of AGBAJA iron ore using acid leaching. High sulphur content found in the AGBAJA iron ore has been verified to have adverse effects for use in the Blast Furnace of the Ajaokuta Steel Company Limited. Nigeria is one of the richest countries of the world in terms of mineral deposits. Among these deposits is iron ore, located at AGBAJA, Kogi State, Nigeria. AGBAJA iron ore deposit, the largest in Nigeria is about 1.3 billion tonnes. AGBAJA iron ore is of low silicon modulus (SiO_2/Al_2O_3), fine texture and contains about 1.4-2.0% sulphur.

Iron is one of the most abundant elements in the earth's crust, being the fourth most abundant element at about 5% by weight [1]. Astrophysical and seisimic evidence indicate that iron is even more abundant in the interior of the earth and is apparently combined with nickel to make up the bulk of planets core. Iron ores are mainly composed of iron oxides, and oxyhydroxides, with other accessory gangue phases. These iron ores cannot be used in the production of steel in their raw states. For them to be maximally used in the production of quality steel, they must be upgraded or beneficiated.

Although the terms coarse-grained, intermediate size and fine grained are not assigned definite or specific dermacative values in mineral processing, a fine grained iron ore is often construed as one in which mineral matter is so finely disseminated within the gangue matrix that crushing and grinding, to effect liberation, produce minute particles that respond poorly to conventional beneficiation equipment and/ or processes (froth flotation, magnetic separation gravity separation etc.) [2]. Uwadiale [3] observed that the utilization of AGBAJA iron ore is hampered by its poor response to established industrial beneficiation techniques, this is as a result of fine grained texture of the iron ore.

Sulphur may be incorporated either into the crystal lattice of iron oxides or into the gangue minerals [4]. This element has a deleterious effect on the workability of steel. For that reason, in most places only premium low sulphur ores are extracted leaving many iron mines around the world enriched in which are unsaleable, high–sulphur iron ore.

If steel is produced with high level of sulphur, that steel will be brittle and therefore not ideal for industrial application hence the need for reduction. Depending on the degree of association of sulphur with the minerals in the ore body, iron ore can be reduced either physically or chemically [5,6].

Communition followed by wet magnetic separation or froth flotation is generally employed when the sulphates gangue minerals appear as discrete inclusions in the iron oxide matrix (primary mineralization) [5,6]. However, when sulphur is disseminated in the iron oxide structure, possibly forming cryptocrystalline phosphates or forming solid solutions with the iron oxide phases (secondary mineralization), the reduction can only be processed by chemical routes [4-6].

The chemical reduction involves the hydrometallurgical processing of the ore, that is, the selective leaching of sulphur in the ore with a reagent usually acid or base. Since early in the 19th century, suggested the use of sulphuric acid to remove sulphur compounds from lumps of iron ore. Nevertheless, a real scientific interest in hydrometallurgical processing of high sulphur iron ores can only be noticed after the last third of the 20th century, when several papers and patents were published [4-6]. Ever since, traditionally low prices of iron ore products had impeded the large-scale industrial application of chemical reduction. At the present time, an increase in world steel production has increased demand for iron ore with a consequent increase in the price for this commodity, making hydrometallurgical sulphate removal viable [7]. In the last eight years, the situation of iron ore markets has changed dramatically due to an increase in the world steel consumption, pushed up mainly by the economic growth of China and other Asian emerging markets.

The mechanism and process analysis of reduction of sulphur content of AGBAJA iron ore concentrate using powdered potassium trioxochlorate (v) ($KClO_3$) as an oxidant has been reported [8]. Concentrates were treated at a temperature range 500°C-800°C.

The prevalence of this amount of sulphur is a major setback to its utilization in the blast furnace or direct reduction process [9]. The removal of sulphur from iron and steel presents problems because of similarity of the standard free energies of formation of iron oxide and sulphur pentoxide [9]. Consequently, in the reducing conditions of the blast furnace to recover some 99.5% of the iron charged, near complete reduction of sulphur pentoxide from the acid blast furnace occurs. As the sulphur in the ore impregnates the pig iron, there occur two distinct processes of tackling the problem: pyrometallurgical route and hydrometallurgical route. The first route employs basic slag during the conversion to steel. This technique covers the activity coefficient of sulphur pentoxide in the slag [9,10]. The second route delves into ways of reducing sulphur in the iron at relatively low temperatures. Leaching of lean ores or complex ore in different acids has proved successful for several years. However, the leaching of sulphur contaminated iron ore has made a very limited progress. This underscores the ongoing intense research in the area for several decades. As is well known, smelting process is effective for dispersion but with very high cost, and it is still under fundamental research. For physical separation, communition followed by wet magnetic separation or froth flotation is generally employed when the sulphur in the gangue mineral appears as discrete inclusion in the iron bodymatrix(primary mineralization) [5-7]. Low sulphur extraction, high grinding cost and iron loss are the major disadvantages of the method. However, when sulphur is disseminated in the iron structure, possibly forming cryptocrystalline sulphates or solids solutions with the iron oxide phases (secondary mineralization), and the beneficiation can only proceed by chemical routes [5,7]. He investigated reduction with acid leaching. In their studies, the

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acid concentrations were very high and low sulphur extraction was obtained. In this study, the feasibility of reduction of sulphur content of AGBAJA iron ore by acid leaching at various dilution ratios, dwell time and particle size will be investigated. Also, the Design of Experiment for the evaluation of interactive factorial effects on the sulphur removal will be investigated.

AGBAJA iron ore deposit is the largest in Nigeria with a reserved deposit of One Billion metric tonnes. However, when compared with other iron ore, it is evidently shown that the sulphur content of the AGBAJA iron ore is high (0.12%) with low silicon modules of (SiO_2/Al_2O_3=0.89) and fine grain texture which constitute major problem for utilization in the Blast Furnace (BF) direct reduction process.

Although it is Nigerian's largest known ore deposit estimated at over one billion tonnes, its utilization is hampered by its poor industrial beneficiation techniques. It is in the light of the above that the work seeks to find an alternative way of effectively reducing the sulphur content of AGBAJA iron ore apart from the conventional method of roasting sulphur. Sulphur which is one of the main harmful elements to ferrous metallurgy affects the quality of iron and steel products. At present Nigeria has some large iron ore deposits including AGBAJA which bear tremendous iron ore with high sulphur content. The removal of sulphur from iron ores involves smelting process, physical separation and chemical leaching. Smelting process is effective for reduction but very expensive and still under research. High sulphur extraction, high grinding cost and iron loss are the major disadvantages of the ore. The ore is leached with a suitable solution in a relatively simple process as it can directly treat the fines without strict requirements for the particles sizes. The cost of chemical leaching process mostly depends on the consumption of the acid leaching. In the study, the feasibility of reduction by acid leaching will be investigated. The study is to evaluate the effect acid leaching of Hydrochloric acid (HCL), this with a view of using aid leaching agent for the purpose of reducing the Sulphur content so that the ore could be used for the production of iron and steel through the process of the Blast Furnace located at the Ajaokuta Steel Company Limited. The experiment therefore to bring all these potentials to bear in the iron and steel industry.

General Description AGBAJA Iron Ores

The ore samples were compacted bonded crystalline. The AGBAJA sample consisted mainly of aggregates of brown, compact, fine – grained material with some larger, extremely friable particles. This ore is strongly magnetic. The lump ore samples were crushed mechanically and sieved to give particles in the size range 1-1.7 mm (16-10 mesh). Care was taken to ensure that the size fractions were the representative of the lump material. The ore particles were crushed further for certain experimental techniques. Analyses for calcium, magnesium, iron, aluminium, sulphur were made by atomic absorption spectrometry; silica was determined by a combination of gravimetric and colorimetric method, X-ray diffraction analysis that was performed (Figure 1).

Materials and Methods

The experiments on the research works were carried out in the course of studying the reduction of sulphur content of AGBAJA iron ore using acid leaching agents which includes HCl, for reduction. Mineral processing involves collection of ore samples, the prepared ore samples were carried out by communition and the chemical analysis of raw and scrubbed ore. Furthermore, modeling of the observed variables and data generated were used to carry out Central Composite Design (CCD) modeling technique.

Figure 1: Sample of the iron ore as obtained from Agbaja mines.

The materials and equipment that were used in this study include the following: Hand trowel, pan, mesh screens, blender, Electronic weighing balance, Mechanical shaker , porcelain pot, pH indicator device, Oleic acid, sodium silicate, distilled water, kerosene , beakers, crushers, x-ray fluorescence, spectrometer, atomic absorption spectrophotometer, LF6484A flotation machine, aero froth, cylinders, autoclave hot air oven, inoculating loop, petridishes, conical flask, binocular microscope, staining rack, glass, slides, test tubes, deionized water, and the acid leaching agents includes HCl, H_2SO_4 and HNO_3.

Reduction of sulphur content of AGBAJA iron ore using hydrochloric acid

30 kg of the raw ore sample was pulverized to 100 μ particle size for the remaining experiments. Scrubbing was carried out, where water and sodium silicate were both used. The iron ore was poured into a head pan and water was added to a reasonable level. The ore was washed and the water was decanted. This process was repeated for five times until clear water was observed. At this point 5 g of sodium silicate, and 25 drops of oleic acid was introduced and the washing continued for three more times.

After the scrubbing process, the iron ore was dried in the sun and subsequently analyzed chemically using X-ray fluorescence method. The scrubbed iron ore was pulverized and was sieved in order to obtain particle size of 10 μ, 20 μ, 40 μ, 60 μ and 80 μ. Hydrochloric acid solutions of different moles of 0.2, 0.4, 0.8, 1.0, 2.0, 4.0, 8.0, 12.0 and 16.0 were also prepared. 100 grams of particle size of 10 μ of the scrubbed iron ore was weighed and there after poured into a conical flask. 100 ml of 0.2 M of hydrochloric acid was poured into the conical flask containing the ore.

The mixture was stirred properly to ensure (uniform distribution of the particles) homogeneity. The contents were allowed to leach for 5 mins, 10 mins, 20 mins, 30 mins, 60 mins and 120 mins. After all these processes were achieved at the end of each period, the solutions were allowed to cool before filtering took place. The residues were collected, and washed which neutralizes the chemicals used as distilled water was introduced to air dry and oven dried. The experiments were repeated for 0.4, 0.8, 1.0, 2.0, 4.0, 8.0, 12.0 and 16.0 moles and particle size of 20 μ, 30 μ, 40 μ, 60 μ and 80 μ (Figures 2-5).

Hand specimen identification and specific gravity calculation

The physical analysis of the specimen indicates that the specimen has the following characteristics:

- Color………dark brown
- Streak …….brown

Figure 2: Weighing balance.

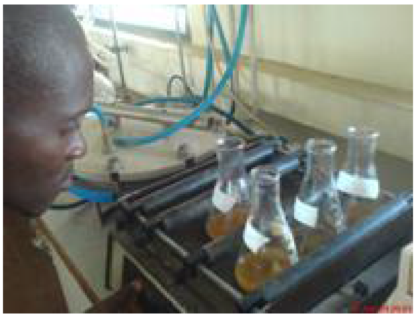

Figure 3: Mechanical shaker.

Figure 4: Blast furnace at Ajaokuta steel company limited.

Figure 5: Direct reduction iron (DRI) plant at Aladja delta steel.

- Specific gravity calculation

Mass of iron ore=297.58 g

Mass of equal volume of water=69.9 g

Sp. Gravity=Mass of substance/Mass of equal volume of water

=297.58 g/69 g

= 4.26

Specific gravity test: The specific gravity of a body is the ratio of the weight of the body to that of an equal volume of water.

Sieve analysis test

***Sample preparation*:** The samples were properly mixed and large residual sample lumps were broken up. The samples were reduced to test sizes by means of Roll Crusher. The test samples were dried to a constant weight in an oven regulated at 80 ± 5°C. The mass of the dry test sample was measured in gram.

***Procedure*:** The sieve chosen for the test were arranged in a stack, with the coarsest sieve on the top and the finest at the bottom. A tight-fitting pan or receiver was placed below the bottom sieve to receive the final undersize, and a lid (cover) was placed on top of the coarsest sieve to prevent escape of the sample.

The micrograph in Figure 6 shows the yellowish-brown colour indicates rings of iron mineral without clear grain boundaries at 250 magnifications, suggesting very fine grains. The visible bright yellow seen on deep brown patches of silica signifies the presence of sulphur in the ore. Moreover, other elements which are not visible from the micrograph occur probably in their apatite phase which makes them difficult to be seen visibly.

The above result when compared with standard values indicates that the color, the streak and the specific gravity showed that it is goethite FeO(OH) ore (Figure 6).

Also from the result obtained from the sieve analysis, a screen analysis graph of cumulative weight percentage retained against particle size was plotted. It can be observed that the 60 μm sieve retained the most quantity of iron ore particles. This shows that the AGBAJA iron ore has fine grain size.

Result of ED-XRF analysis

The chemical composition of AGBAJA iron ore as revealed by ED-X-ray florescence is shown in Table1 below. The ore was determined through XRF. The X-ray analysis indicates that the percentage of iron mineral is 89.40%. The minor elements in the ore are K, Ca, Ti, Mn, Mg, Al, Si, P, and S. A non-stoichiometry value was obtained from

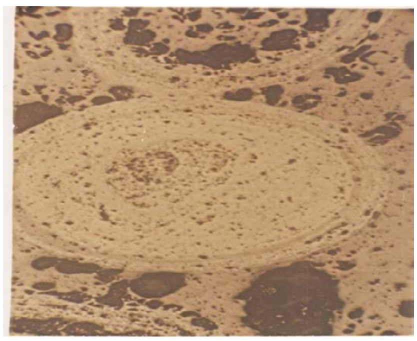

Figure 6: Micrograph of the ore as obtained after the thin section analysis.

Mineral	K_2O	CaO	TiO_2	MnO	Fe_2O_3	MgO	Al_2O_3	SiO_2	P_2O_5	S*	Al_2O_3
Composition	0.04	0.13	0.37	0.40	89.40	0.40	9.60	2.28	1.20	0.12	3.38

Table 1: Chemical composition of Agbaja Iron ore.

atomic ratio Fe: O, which implies that the ore is most likely to be a mixture of hemeatite and magnetite.

The various compositions of minerals have different effects on the ore; the presence of silicon promotes the formation of gray cast iron and as seen from Table 1. AGBAJA iron ore contains considerably high amount of silicon. It is noted that high sulphur causes hot shortness which is brittleness of steel at high temperatures. Moreover the aluminium in the ore does not pose any major problem; but makes it difficult to tap off the liquid slag. Also from the result obtained from the sieve analysis, a screen analysis graph of cumulative weight percentage retained against particles size was plotted. It can be observed that the 60 µm sieve retained the most quantity of iron ore with fine grain size.

Factorial design

Central composite design plan HCL

No. of variables=3

No. of star points=6

No. of center points=3

X_1=leaching time (5 mins – 120 mins)

X_2=Concentration (0.2 M – 16 M)

X_3=particle size (10 µ – 80 µ)

Lower level	Base level	Upper level
-1	0	+1
5 mins	60 mins	120 mins x_1
0.2 M	8 M	16 M x
10 µ	40 µ	80 µ x_3

Development of statistical multivariable models

The standard design of experiment (DOE) is an efficient procedure for planning experiments so that data obtained can be analyzed to yield valid objective conclusions.

Standard Central Composite Design (SCCD) with 2^3 and 2^2 full factorial designs will be employed. These were constructed from 2^{m-t} design for cube portion, which is augmented with center points and start points.

For 2^3 full factorial designs: number of experimental points for Central Composite Design (CCD).

$N = K^{m-t} + 2n + No$ (1)

Where,

K=Level of experiments=2

m=Total number of variables (3: x_1, x_2, x_3)

t =The degree of fractionality, t=0 for m<4

No=Center points added=3

Therefore,

$N=2^{3-0} + 2\ (3) + 3=17$ runs

For 2^2 full factorial designs:

$N=K^{m-t} + 2m + No$……… (3.2)

K=Level of experiments=2

m=Total number of variables (2: x_1, x_2)

T=The degree of fractionally, t=u for m<4

No=Center points added=3

Therefore,

$N=2^{2-0} + 2^{(2)} + 3=11$ runs

The model equation for the experiment is proposed as

$y_n=b_0 + b_1\ x_1 + b_2\ x_2 + b_3\ x_3 + b_{12}\ x_1\ x_2 + b_{13}\ x_1\ x_3 + b_{23}\ x_2 x_3 + b_{11}\ x_{12} + b_{22}\ x_{22} + b_{33} x_{32}$

Experimental Results and Discussion

Reduction of sulphur content of AGBAJA iron ore using HCL

Effect of HCL concentration on the percentage degree of reduction of sulphur content of AGBAJA iron ore: The effects of hydrochloric acid on the reduction of sulphur content of AGBAJA iron ore are shown in Figures 7-11. In Figure 7, it is shown that the percentage degree of reduction of sulphur content between the concentrations of 0.2 M to 0.8 M was fairly constant. It was also noted that as from 0.8 M there was a slight increase in percentage reduction of sulphur content until it got to 1 M. The experiment also showed that as from 1 M, the degree of reduction remained fairly constant. As the concentration was

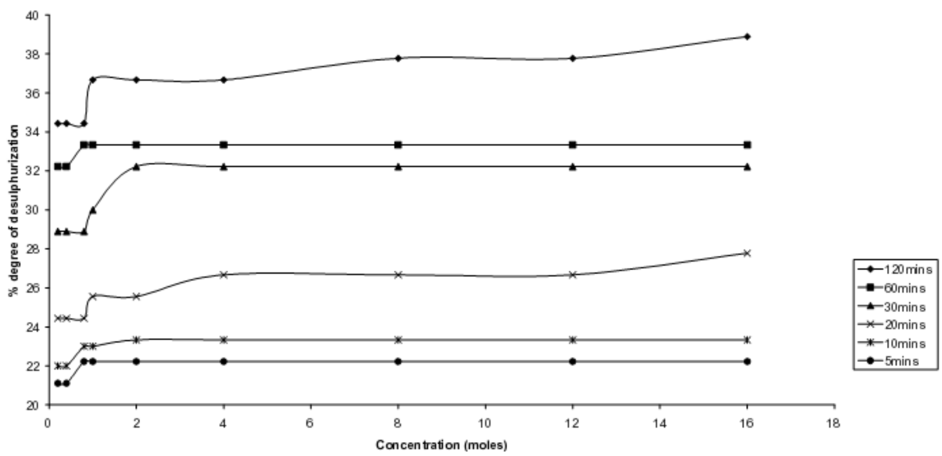

Figure 7: Effect of HCl Concentrations on the % degree of reduction of sulphur for 80 µ.

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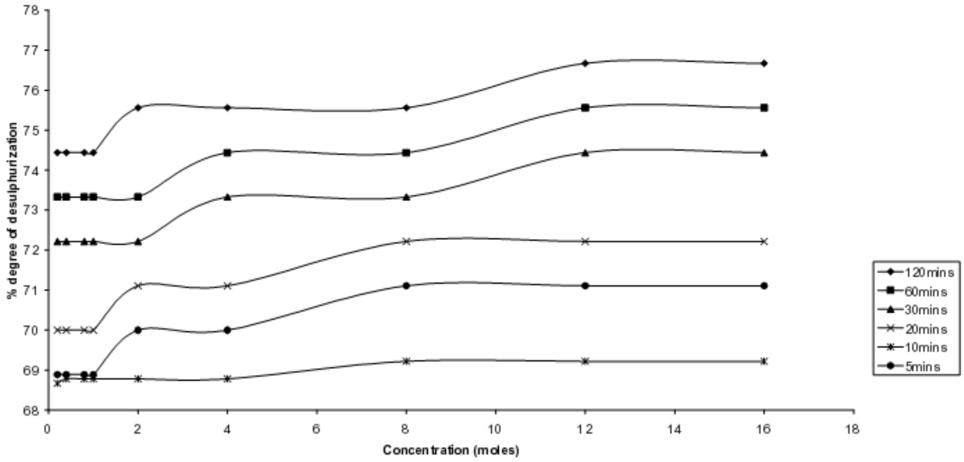

Figure 8: Effect of HCl concentration on the % degree of desulphurization for 60 µ.

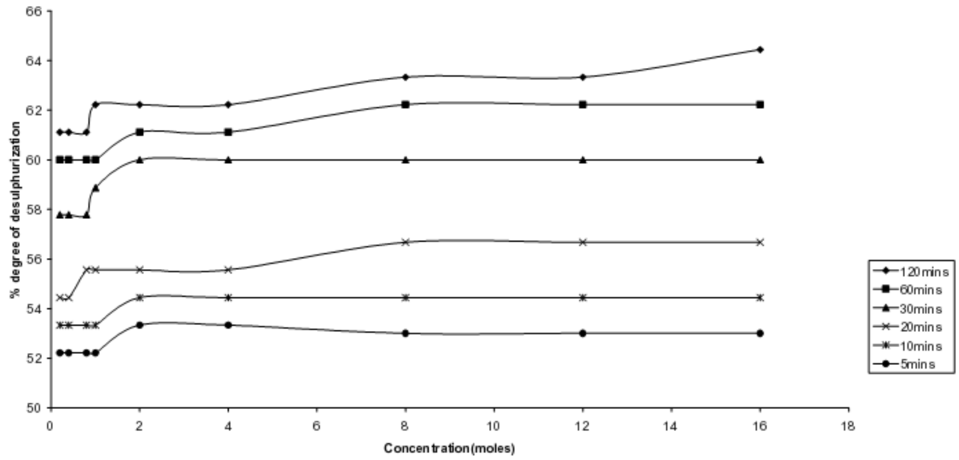

Figure 9: Effect of HCl concentration on the % degree of reduction of sulphur content for 40 µ.

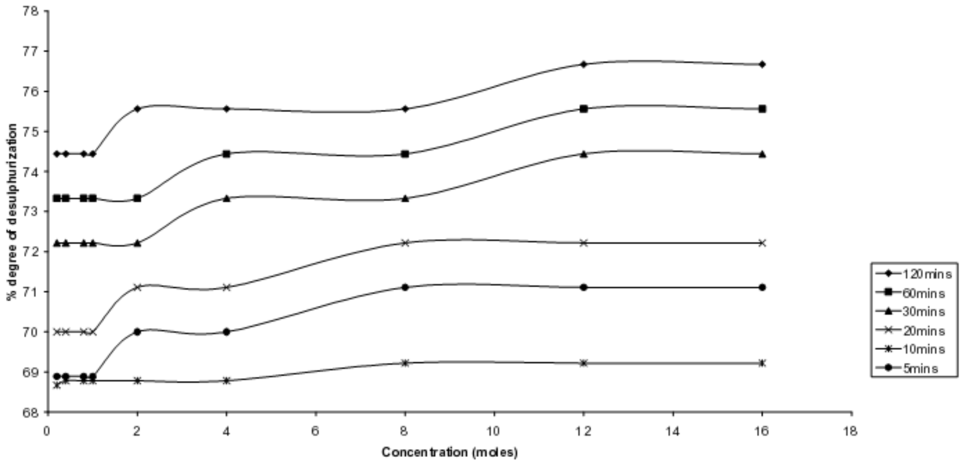

Figure 10: Effect of HCl concentrations on the % degree of reduction of sulphur content for 20 µ.

increased to 16 M for particle size of 80 μ, and leaching time of 120 minutes, the percentage degree of reduction of sulphur content was obtained at 38.89% while for 0.2 M at the same conditions, the rate of reduction of sulphur content was obtained at 34.44%.

The percentage degree of reduction of sulphur content increased fairly between 0.2 M and 0.8 M, but from 0.8 M the increment was fairly steady. The highest value of reduction of sulphur content was shown in Figure 8 and was at 43.33% at concentration of 16 M for a leaching time of 120 minutes. Between concentrations of 0.2 M and 1.0 M, the rate of reduction of sulphur content was fairly constant. But as from 1.0 M to 2.0 M, there was an increase in the degree of reduction of sulphur content. From 2.0 M the increment was fairly constant. This was depicted in Figure 9. In Figures 10 and 11 a similar trend was obtained as shown in the figures described below. It could be inferred that as the concentration of hydrochloric acid increases, the percentage degree of reduction of sulphur content slightly increases.

Effect of leaching time on the percentage degree of reduction of sulphur content of AGBAJA iron ore: The time effect on the percentage degree of reduction of sulphur content is shown in Figures 12-16. Between 5 minutes and 30 minutes there was steady increase in the reduction of sulphur content as shown in Figure 12. As from 30 minutes, the rate of reduction of sulphur content was reduced. It was also observed that for 16 M, 22.22% of sulphur content was reduced in 5 minutes while 38.89% was reduced in 120 minutes. In Figure 13, there was a gradual reduction of sulphur content until it got to 60 minutes. As from 60 minutes, the percentage of reduction was increased. The highest value of 43.33% was obtained at leaching time of 120 minutes for 16 M concentration.

The percentage degree of reduction of sulphur content as shown in Figure 14 followed a similar trend; the percentage of reduction of sulphur content for 16 M at 120 minutes was obtained at 64.44% while at a time of 5 minutes it was observed that the reduction obtained was at 54.44%. It was generally shown that the longer the time of leaching the more reduction of sulphur content was achieved.

As the leaching time increases the degree of reduction of sulphur content increases as shown in Figures 15 and 16. The highest values of reduction for 16M at 120minutes were obtained at 76.67% and 84.44% respectively. As the analyses progresses it could be observed that as the leaching time increases the percentage degree of reduction of sulphur content increases. The results obtained from the experiments agreed with [9] which observed that the percentage reduction sulphur is directly proportional to leaching time.

Effect of particle size on the percentage degree of reduction of sulphur content: The effects of particle size on the percentage degree of reduction of sulphur content using hydrochloric acid are represented in Figures 17-22. In Figure 17, there was high percentage of reduction of sulphur content between 10 μ and 60 μ. From 60 μ to 80 μ the percentage reduction of sulphur content increased from 5.56% to 22.22%.

The highest percentage of reduction of sulphur content obtained was 89.22% for particle size of 10 μ while the least value obtained was 22.22% for particle size of 80 μ both at a concentration of 16 M for 5 minutes.

The percentage degree of reduction of sulphur content was quite significantly strong between 10 μ and 60 μ as depicted in Figure 18. From 60 μ, the percentage reduction was fairly moderate; an average percentage reduction at 27% was achieved. For 16 M concentration, and particle size of 10 μ, the highest reduction value of 89.22% was obtained.

Similar trend was obtained for Figure 19. The least value of percentage reduction was recorded at particle size of 80 μ. This was because the surface area that was exposed to leaching was less than that of 10 μ. This adduces the reason why the highest value of 83.33% was obtained using the particle size of 10 μ. The percentage degree of reduction of sulphur content was fairly moderate at particle size of 60 μ posting an average percentage reduction of 33.00%. Between 10 μ and 60 μ, an average percentage of 65.00% was achieved as shown in Figure 20. In Figures 21 and 22 it was observed that similar trend was obtained. The highest value of 84.44% was obtained in Figure 21 for particle size of 10 μ while the highest value of 85.56% was obtained for Figure 21 for the particle size of 10 μ for concentration of 16 M. It could be concluded that the percentage degree of reduction of sulphur content was inversely proportional to the average diameter of the particles. As the particle size decrease, the percentage of reduction of sulphur content increases as depicted in the works of Alafara [1].

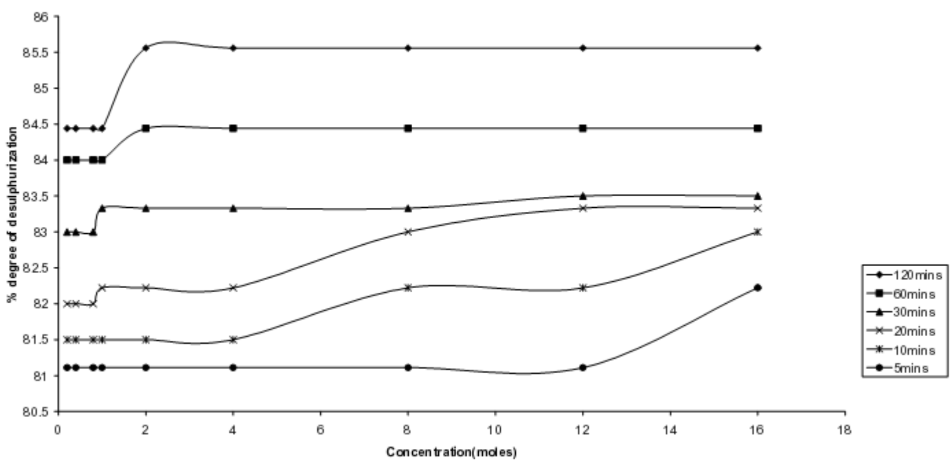

Figure 11: Effect of HCl concentrations on the % degree of reduction of sulphur content for 10 μ.

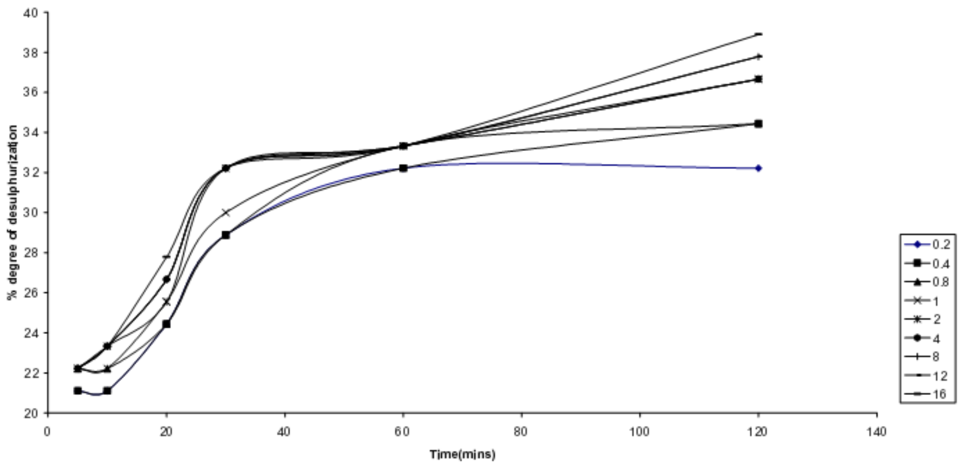

Figure 12: Effect of leaching time on the % degree of reduction sulphur for 80 μ using HCl.

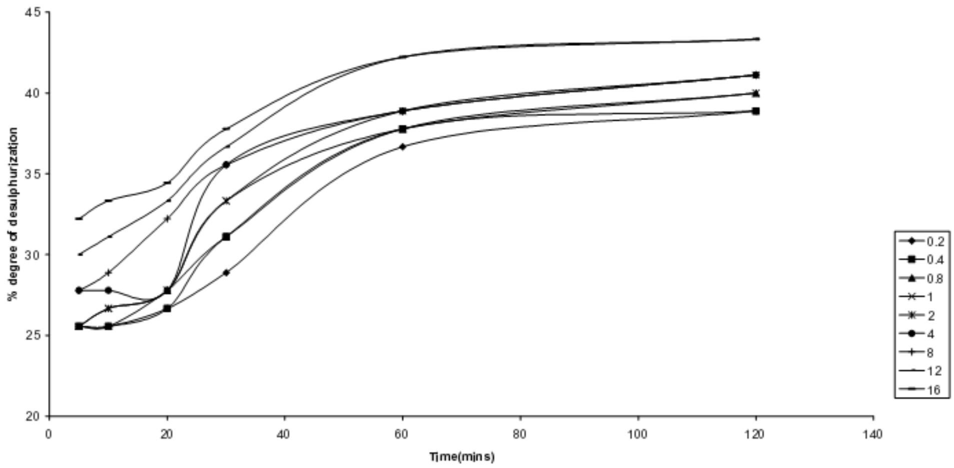

Figure 13: Effect of leaching time on the % degree of reduction of sulphur for 60 μ using HCl.

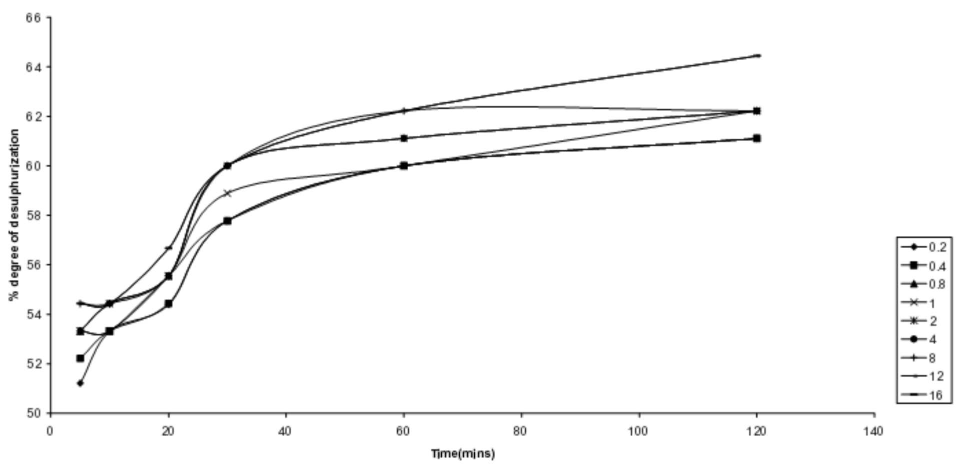

Figure 14: Effect of leaching time on the % degree of reduction sulphur for 40 μ using HCl.

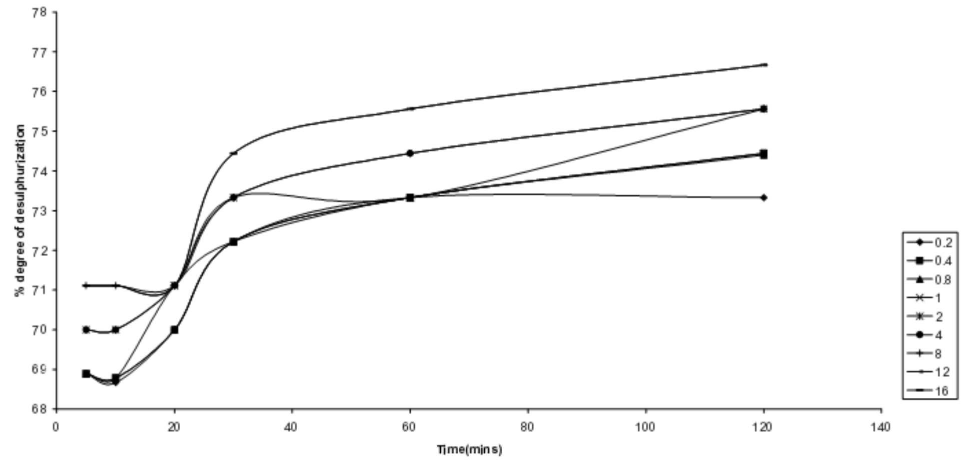

Figure 15: Effect of leaching time on the % degree of reduction of sulphur for 20 μ using HCl.

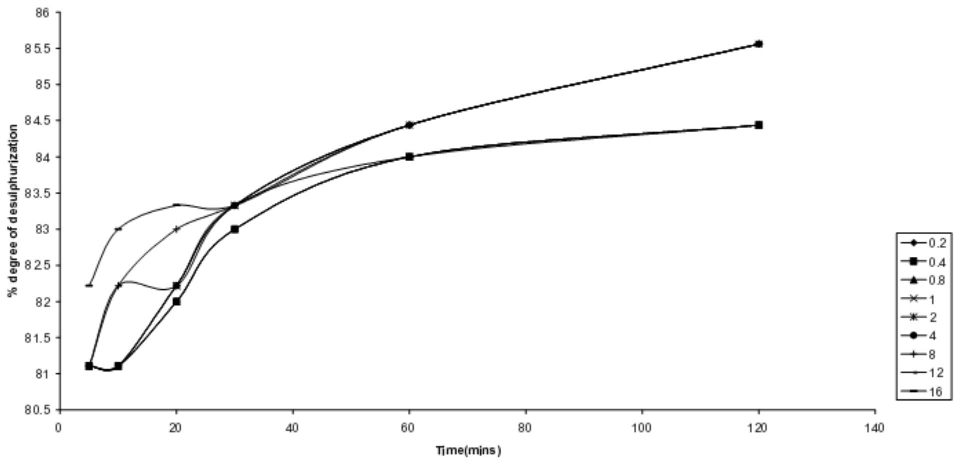

Figure 16: Effect of leaching time on the % degree of reduction of sulphur for 10 μ using HCl.

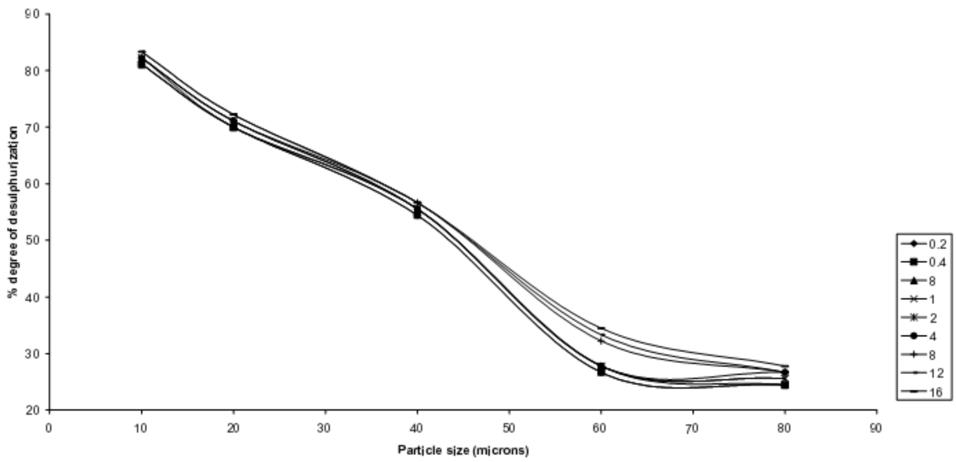

Figure 17: Effect of particle size on the % degree of reduction of sulphur for 5 mins using HCl.

Citation: Ocheri C, Mbah AC (2016) Reduction of Sulphur Content of AGBAJA Iron Ore using Hydrochloric Acid (HCL). J Material Sci Eng 5: 292. doi:10.4172/2169-0022.1000292

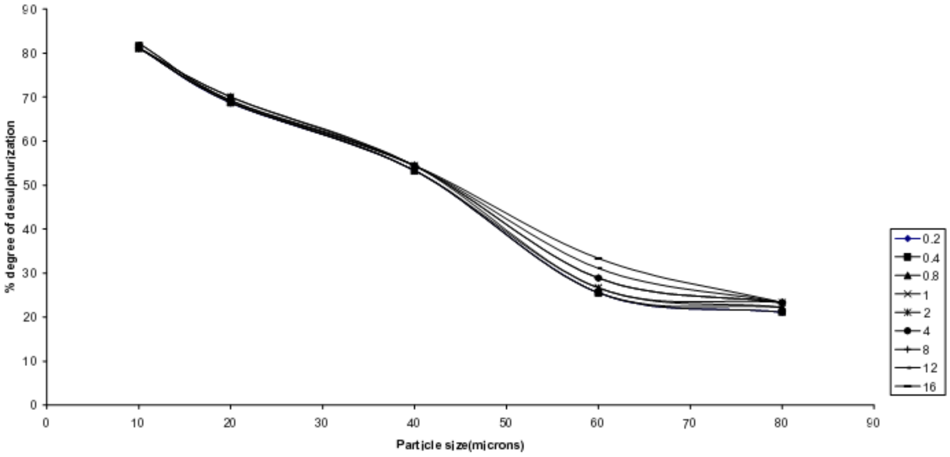

Figure 18: Effect of particle size on the % degree of reduction of sulphur for 10 mins using HCl.

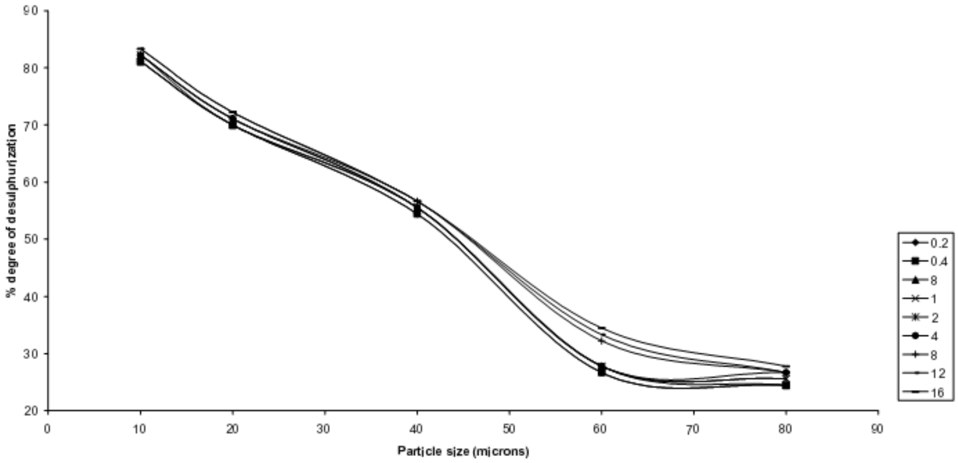

Figure 19: Effect of particle size on the % degree of reduction of sulphur for 20 mins using HCl.

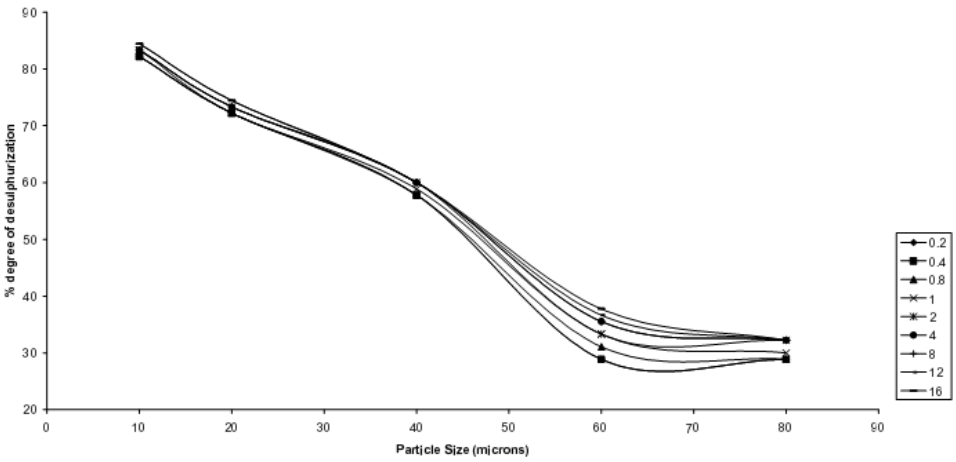

Figure 20: Effect of particle size on the % degree of reduction of sulphur for 30 mins using HCl.

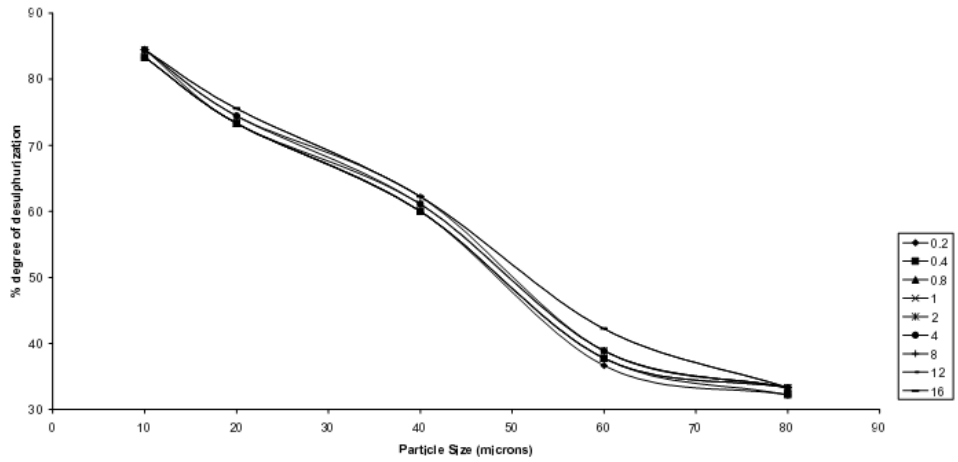

Figure 21: Effect of particle size on the % degree of reduction of sulphur for 60 mins using HCl.

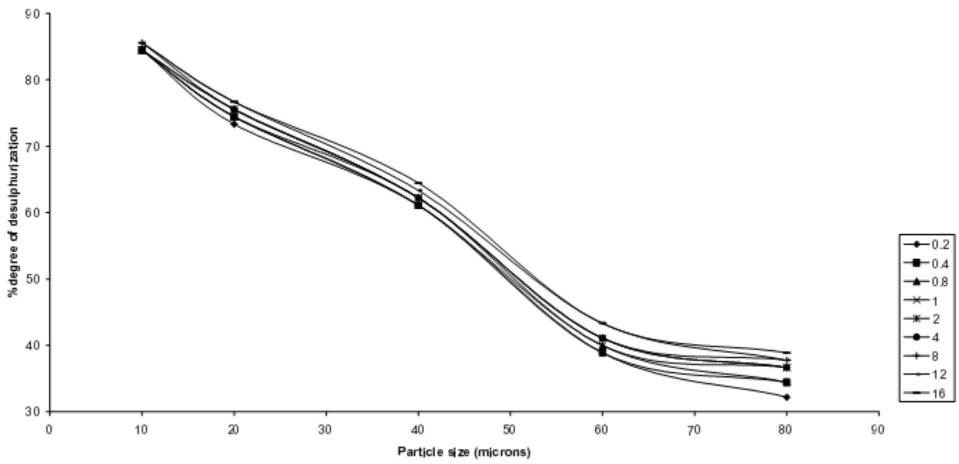

Figure 22: Effect of particle size on the % degree of reduction of sulphur for 120 mins using HCl.

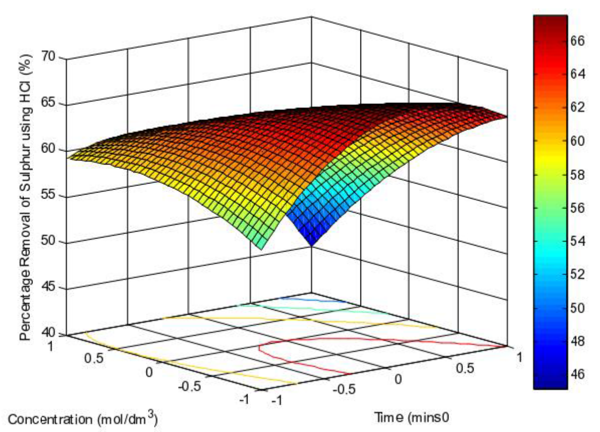

Figure 23: Percentage reduction of sulphur content using HCl time and concentration.

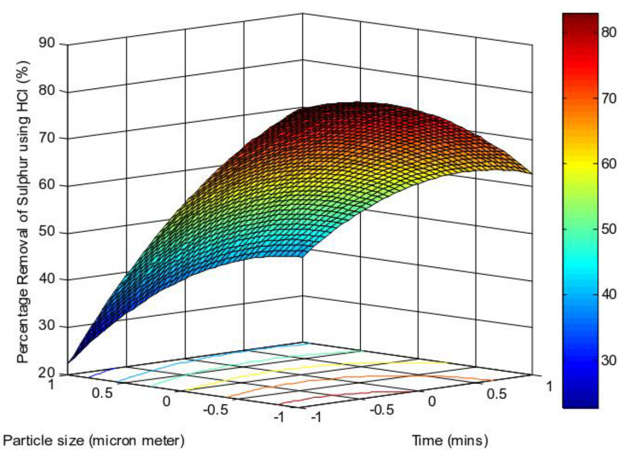

Figure 24: Percentage reduction of sulphur content using HCl (particle size and time as variables).

Surface Response Plots

Surface response as controlled by leaching variables. The surface response plots were plotted using MATLAB. In Figure 23 the surface response is represented for percentage reduction of sulphur content for time and concentrations of HCl as variables. Time responded more than concentration and optimum is located the dark red colour of the surface.

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The surface response plot for percentage reduction of sulphur content using particle size and time as variables is shown in Figure 24 times responded more than particle size as shown in the figure and the optimum is located on the dark red portion of the surface.

Figure 25 depicts the surface response plot for percentage reduction of sulphur content where particle = size and concentration are variable. The optimum is located as the dark red colour on the surface and concentration responded more than particle size.

Conclusion and Recommendation

AGBAJA ore is an acidic oolitic ore consisting of goethite, magnetite and major amount of aluminous and siliceous minerals. It cannot be used directly in a blast furnace or other reduction process without further treatment, sintering, pelletizing or briquetting. Acid leaching is an effective method of reducing sulphur content from iron ores.

The recycle of hydrochloric acid solutions are incorporated processes which obtained results for the reduction of sulphur content of the AGBAJA Iron ore as a by-product which makes the whole process for reduction of sulphur content more economical.

It is therefore, noted that the percentage degrees of reduction of sulphur content using the acid leaching gave remarkable results. The Central Composite Design (CCD) used relevant parameters and data to develop the models.

The developed models were tested and the results were quite adequate in the reduction of sulphur content of AGBAJA iron ore. The experimental results for percentage degree of the reduction of sulphur content were 85.56%. It is quite significant to note that the optimum values for percentage reduction of sulphur content obtained from the model for HCL was 89.66%.

The experimental results also showed that as the leaching time and concentration increases, the percentage degree of reduction of sulphur content also increases while the particle size decreased as the leaching agent's increases. It is significantly observed that the level of reduction of sulphur content indicates that all the necessary processes were put in the right perspectives as being carried out in this research work, the final aims and objectives of the research work has been achieved.

Conclusion

The research work has availed the researcher the opportunity to explore all the experimental processes to understand the techniques involved in the reduction of sulphur content of AGBAJA iron ore using the leaching process.

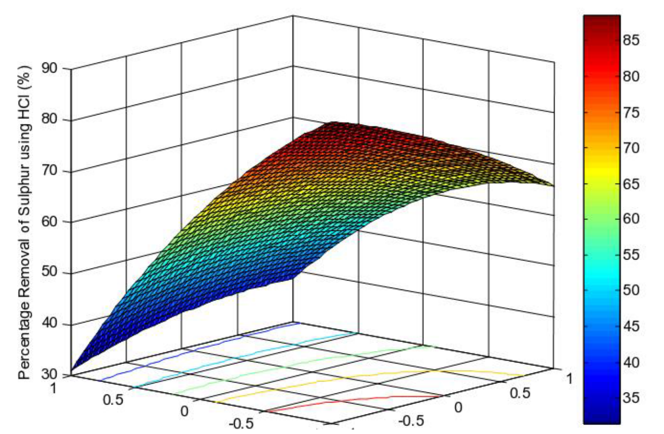

Figure 25: Percentage reduction of sulphur content using HCl (particle size and concentration as variables).

It should therefore be known that the large deposit of the iron ore which was initial adjudged as not suitable for the production of Direct Reduced Iron (DRI) and for the usage at the Blast Furnace at Ajaokuta Steel Company Limited due to the harmful nature of the ore because of high sulphur of 0.12% could be drastically be reduced.

This will further indicate that the fear of harmful impurities/ effects that were initially envisaged to cause some harmful effects will be reduced to a minimal level through the reduction of the sulphur content using acid leaching process as indicated in this research work.

Reduction of Sulphur Content of AGBAJA Iron Ore Using HCL

```
A =[1 1 1];
>> b=[3];
>> lb=[-1;-1;-1];
>> ub=[1;1;1];
>> x0=[0;0;0];
>> [x,fval,exitflag,output]=fmincon(@leaching1,x0,A,b,[],[],lb,ub)
x =
-0.3098
-1.0000
-1.0000
fval =
89.6659
exitflag =
1
output =
iterations: 3
funcCount: 19
stepsize: 1
algorithm: 'medium-scale: SQP, Quasi-Newton, line-search'
firstorderopt: 9.6488e-007
cgiterations: []
message: [1x144 char]
```

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