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Reduction of Sulphur Content of AGBAJA Iron Ore Using Sulphuric Acid (H_2SO_4)

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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 also 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 87.77% the optimum % removal of sulphur is 87.73%. 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 Sulphuric Acid (H_2SO_4)

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 iron 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/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 and Whewell [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 [4].

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].

Comminution 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]. 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 20^{th} 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, comminution 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 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 Sulphuric Acid (H_2SO_4), 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.

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 agent H_2SO_4 for, reduction. Mineral processing involves collection of ore samples, the prepared ore samples were carried out by comminution 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.

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 agent includes H_2SO_4.

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 were performed (Figure 1).

Reduction of sulphur content of AGBAJA iron ore using sulphuric acid

The scrubbed iron ore was further pulverized and sieved with a view to obtaining particle sizes which range from 10 microns, 20 microns, 30 microns, 40 microns, 60 microns and 80 microns. Sulphuric acid (H_2SO_4), 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 prepared. 100g of particle size of 10 microns of the scrubbed iron ore was weighed and subsequently poured into a conical flask. 100 mL of 0.2 M of sulphuric acid was poured into the conical flask which contained the ore.

The mixture was vigorously stirred with a shaker to ensure proper homogeneity. The contents were allowed to leach for 5 min, 10 min, 20 min, 30 min, 60 min and 120 min. At the end of each period, the solutions were cooled and thereafter the solutions were filtered. The residues were collected and washed to ensure that the solutions were neutralized with distilled water; the residues were air dried and oven dried. The experiment were repeated for 0.4, 0.8, 1.0, 2.0 and 4.0 moles and particle sizes of 20 microns, 30 microns, 40 microns, 60 microns and 80 microns. Photographic Display of the Leaching Procedure is shown in 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

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

Figure 2: Weighing balance.

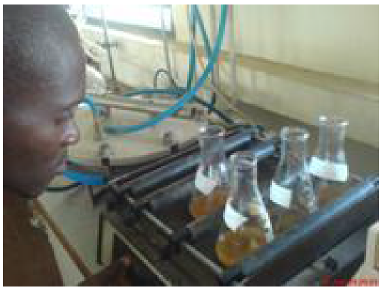

Figure 3: Mechanical Shaker.

Figure 4: Blast Furnace at Ajaokuta Steel.

Figure 5: Direct Reduction Iron (DRI) Plant, Aladja Delta Steel.

- Streak brown
- 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/69.9 g

=4.26

The micrograph in plate 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.

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=297.58g/69.9g

=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.

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 Table 1 below which implies that

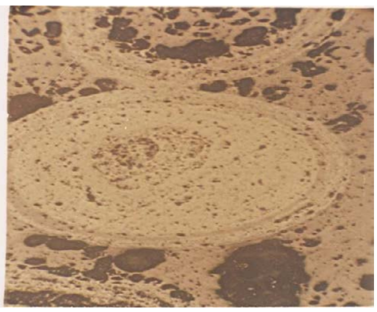

Figure 6: Micrograph of the ore as obtained after the thin section analysis.

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.

Experimental Results and Discussion

Reduction of sulphur of AGBAJA iron ore using sulphuric acid

Influence of sulphuric acid concentration on reduction of sulphur of AGBAJA iron ore: The influence of sulphuric acid concentrations on the reduction of sulphur content of AGBAJA iron ore are illustrated in Figures 7-11. In Figure 7, the percentage degree of reduction of sulphur content remained fairly constant between 0.2 M and 1 M. From 1 M, there was slight increase in percentage reduction of sulphur content. The highest percentage reduction was achieved at 46.66% for leaching time of 120 min and for particle size of 80 microns while the least value was obtained at 45.00% at 0.2 M concentration.

Between 0.2 M and 0.4 M, there was a slight increase in percentage reduction of sulphur content as shown in Figure 8. From 0.4 M, the percentage degree of reduction of sulphur content was slightly increases, attaining the highest value of 50.00%.

The percentage of reduction remained constant from 0.2 M to 1.0 M for leaching time of 120 min, 60 min and 30 min and slightly increased after1.0 M as depicted in Figure 9. For leaching time of 20 min, 10 min and 5 min, for all the concentrations, the increase in percentage reduction of sulphur content was gradually achieved. The highest value of percentage reduction was 67.77% for 4.0 M at particle size of 40 microns. The percentage degree of reduction of sulphur content was constantly achieved between 0.2 M and 0.8 M. There was a slight increase between 0.8 M and 1.0 M. As from 1.0 M, the percentage of reduction of sulphur content became fairly constant. 80.00% of sulphur was reduced as the highest value for concentration of 4.0M at leaching time of 120 min as shown in Figure 10. Figure 11 shows that the percentage of reduction was constantly achieved except for 120 min, where there was little increase from 2 M. The highest value of 87.77% was obtained for 10 microns at concentration of 4.0 M. It could be inferred that the percentage degree of reduction of sulphur content is directly proportional to H_2SO_4 concentration.

Effect of leaching time on the percentage degree of reduction of sulphur content of AGBAJA iron ore

The leaching time effects are represented in Figures 12-16. In Figure 12, the percentage degree of reduction of sulphur increases between 5 min and 30 min significantly but from 30 min there was steady increase in percentage of reduction of sulphur content. The highest percentage reduction of sulphur was achieved at 46.66% for 4 M at leaching time of 120 min. The least value was obtained at 34.44% for 4 M at leaching time of 5 min.

There was a significant increase in the percentage degree of reduction of sulphur between 5 min and 60 min. But from 60 min there was slight increase in percentage reduction of sulphur as depicted in Figure 13. At exactly 30 min, for 4 M concentration 43.33% of sulphur content was reduced while the highest value of 50.00% was reduced at 120 min at the same conditions. This shows that time is of essence in sulphur reduction from the iron ore. Between 5 min and 60 min, the percentage degree of reduction of sulphur content was significant while from 60 min to 120 min there was an average slight increase in percentage reduction of 1.1% as shown in Figure 14. Figure 15 shows that between 5 min and 30 min, there was appreciable increase in percentage reduction of sulphur content. There was a slight percentage

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
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.

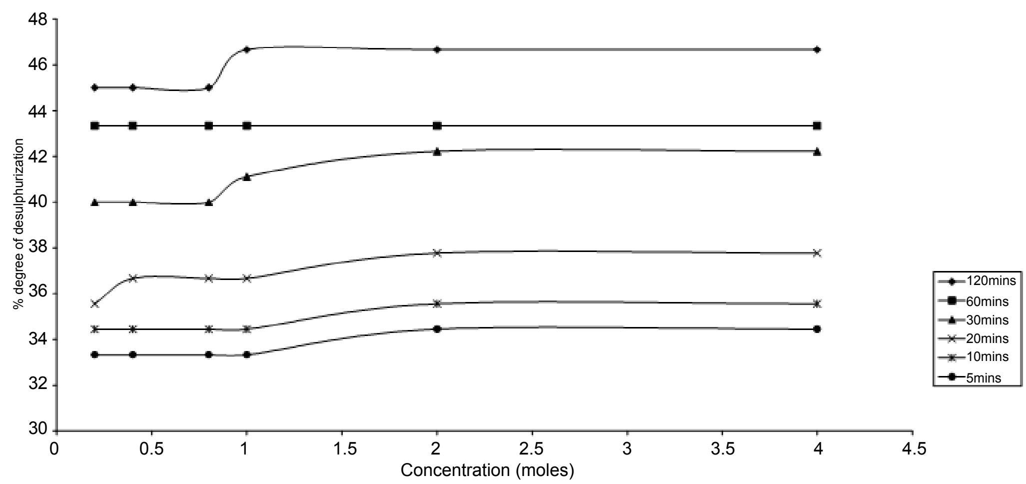

Figure 7: Influence of H_2SO_4 concentration on the %degree of reduction of sulphur for 80microns.

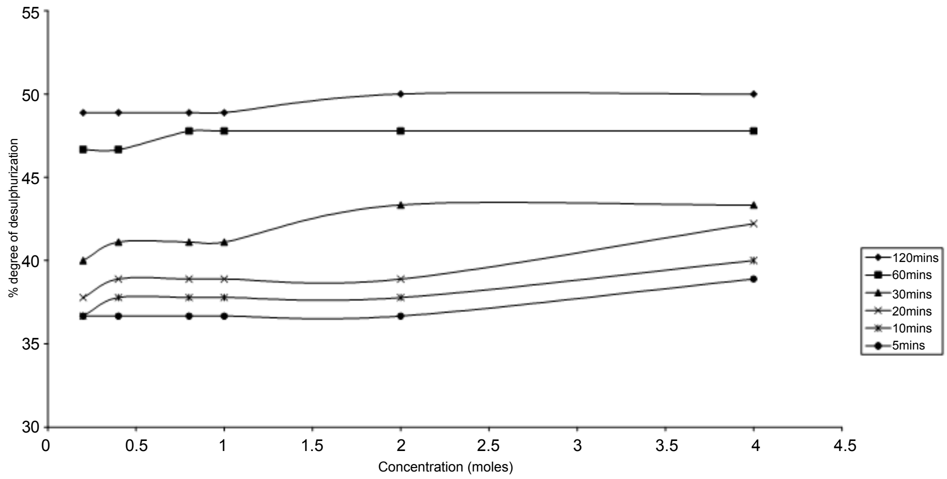

Figure 8: Influence of H_2SO_4 concentration on the %degree of reduction of sulphur for 60 microns.

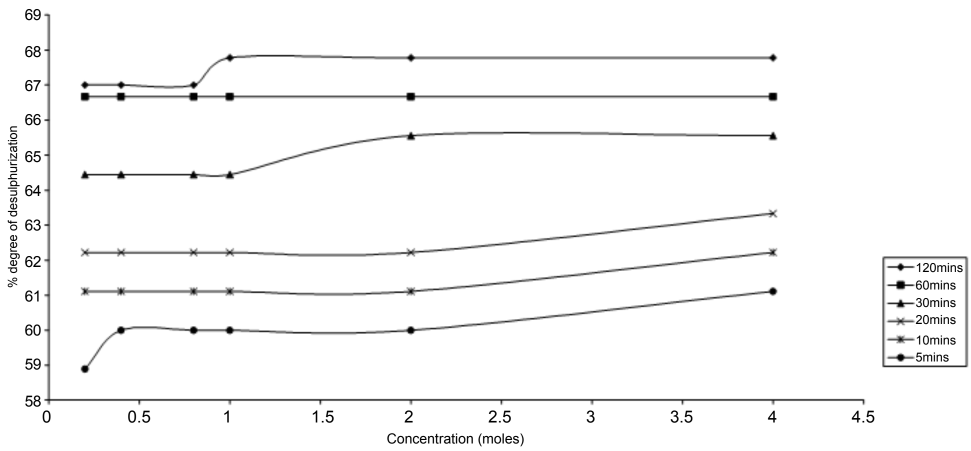

Figure 9: Influence of H_2SO_4 concentration on the %degree of reduction of sulphur for 40 microns.

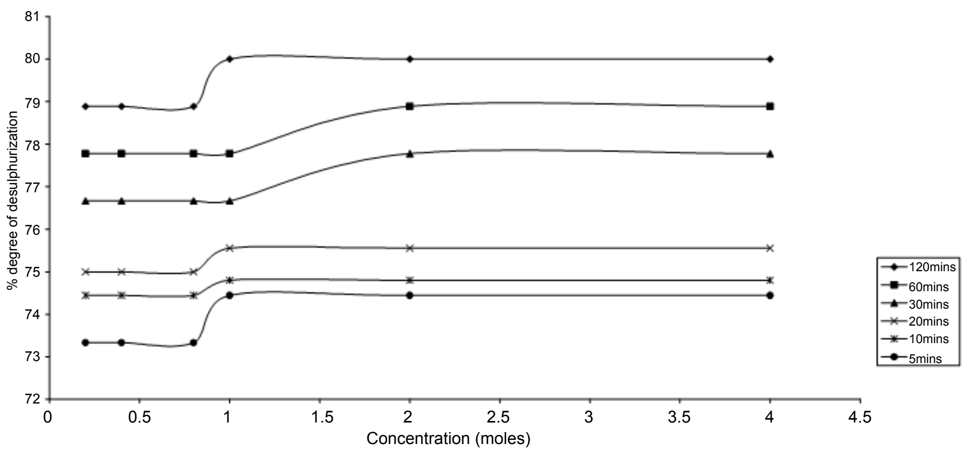

Figure 10: Influence of H_2SO_4 concentration on the %degree of reduction of sulphur for 20 microns.

of reduction of sulphur content from 30 min to 120 min for particle size of 20 microns. The highest value obtained was at 80.00% for 4.0 M concentration. The percentage degree of reduction of sulphur content significantly increased between 5 min and 60 min while from 60 min there was a slight increase in sulphur reduction as shown in Figure 16. As the leaching time increases the percentage degree of sulphur content equally increased.

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Effect of particle size on the percentage degree of reduction of sulphur content of AGBAJA iron ore: The effects of particle size on the percentage degree of reduction of sulphur content of AGBAJA iron ore are shown in Figures 17-22. In Figure 17, the percentage degree of reduction of sulphur content decreased sharply from 10 microns to 60 microns while from 60 microns to 80 microns there was a fairly significant percentage reduction of sulphur content. The highest value of 84.44% was obtained for the particle sizes of 10 microns at 4.0 M.

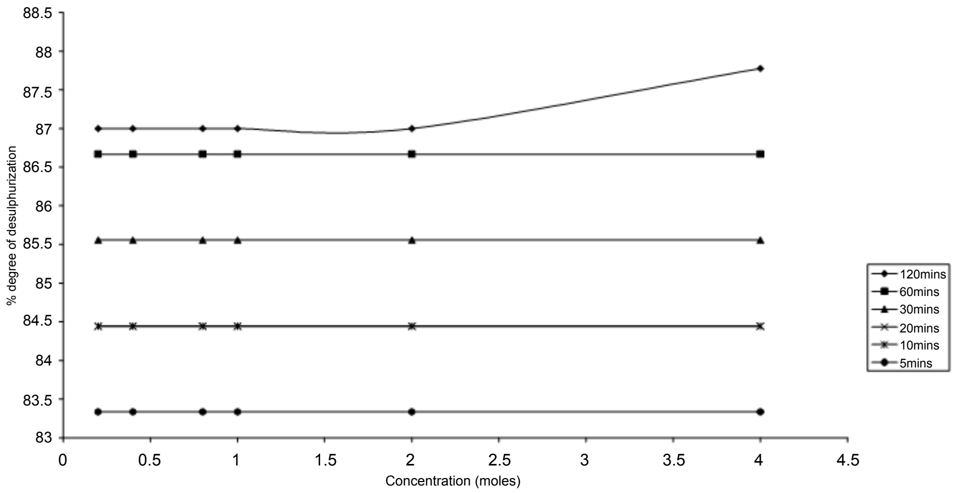

Figure 11: Influence of H_2SO_4 concentration on the %degree of reduction of sulphur for 10 microns.

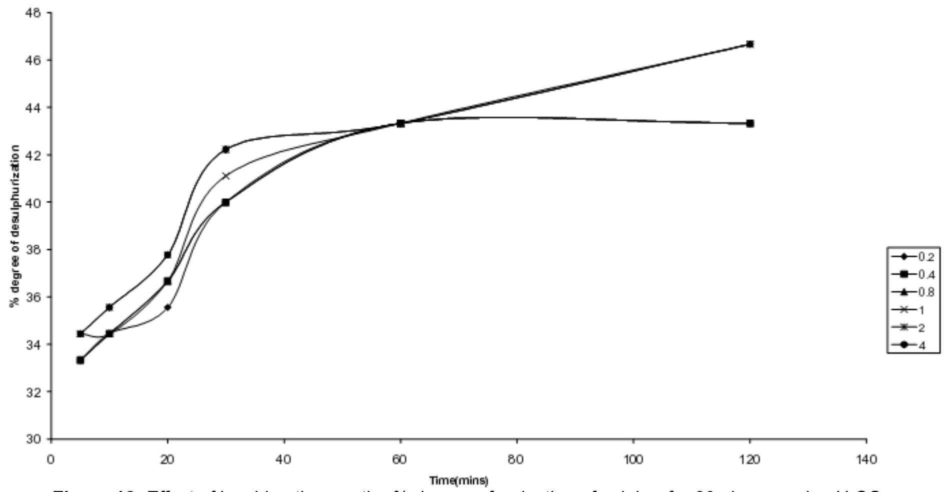

Figure 12: Effect of leaching time on the % degree of reduction of sulphur for 80microns using H_2SO_4.

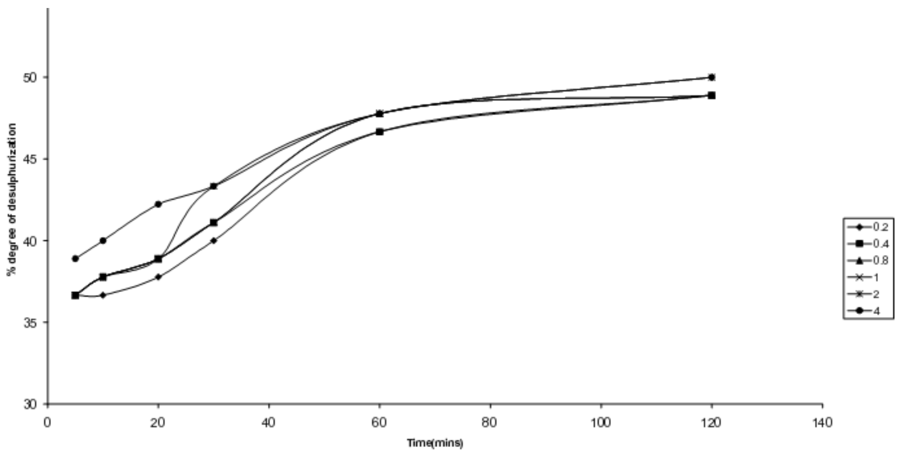

Figure 13: Effect of leaching time on the %degree of reduction of sulphur for 60microns using H_2SO_4.

Figures 18 and 19 showed similar trend. More than 40.0% reduction of sulphur content was achieved between particle sizes of 10 microns and 60 microns. Between 60 microns and 80 microns about 2.0% reduction was achieved implying that large particle sizes do not favour the reduction of sulphur content.

The percentage degree of reduction of sulphur content increased significantly between 10 microns and 60 microns. But between 60 microns and 80 microns there was fairly significant percentage of reduction of sulphur content as depicted in Figure 20. The highest percentage of reduction of sulphur content was achieved at 85.55% for concentration of 4.0 M at particle size of 10 microns.

Between 10 microns and 60 microns the percentage of reduction of sulphur content was largely significant as shown in Figures 21 and 22. It slightly increased between 60 microns and 80 microns. The highest value obtained for Figure 21 was obtained at 86.66% while that of Figure 22 was obtained at 87.77% at particle size of 10 microns at the same concentrations. From the forgoing analysis, it could be inferred that percentage degree of reduction of sulphur content was inversely proportional to particle diameter. In other words, as particle size decreases, the percentages of reduction of sulphur content increases.

Surface Response Plots

Surface response as controlled by leaching variables. The surface responses were plotted using MATLAB. The surface response plot for percentage reduction of sulphur content using time and concentration as variables is represented in Figure 23. The time effect is more pronounced than concentration as shown in the figure. Optimum value is located on the surface at yellow-dark red colour.

In Figure 24, the surface response plot for percentage reduction of sulphur content using time and particle size as variable is depicted. The time responded better than particle size. Linear relationship is represented in this plot. Figure 25 represents the surface response plot for percentage reduction of sulphur using particle size and concentration. The influence of concentration was more than that of particle size. It also had a linear relationship.

Summary

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 sulphuric acid solutions is 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

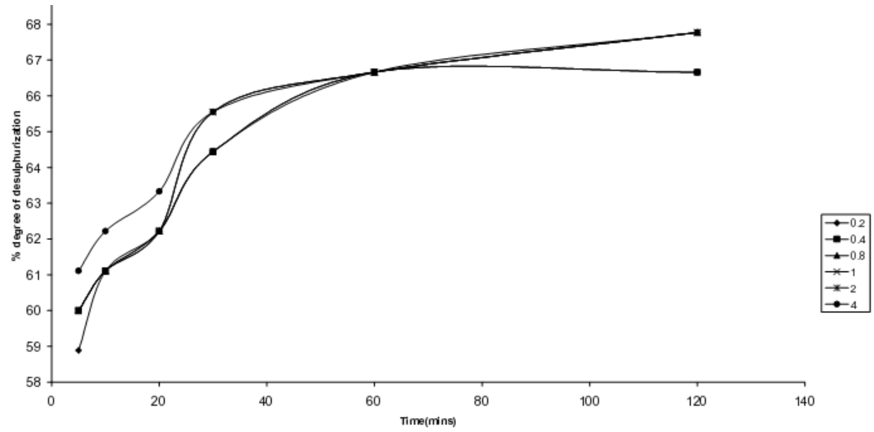

Figure 14: Effect of leaching time on the %degree of reduction of sulphur for 40microns using H_2SO_4.

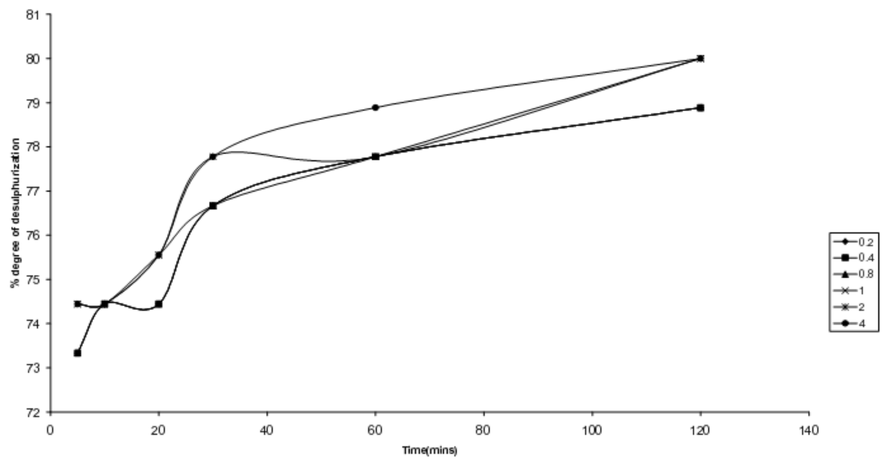

Figure 15: Effect of leaching time on the %degree of reduction of sulphur for 20microns using H_2SO_4.

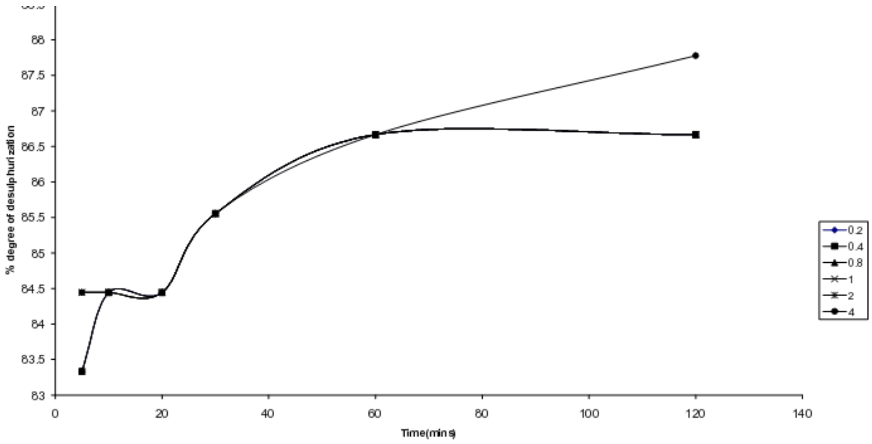

Figure 16: Effect of leaching time on the % degree of reduction of sulphur for 10microns using H_2SO_4.

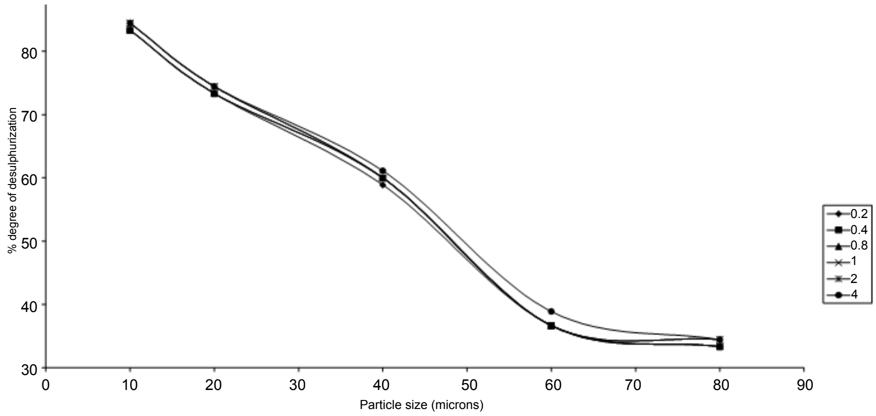

Figure 17: Effect of particle size on the %degree of reduction of sulphur for 5mins using H_2SO_4.

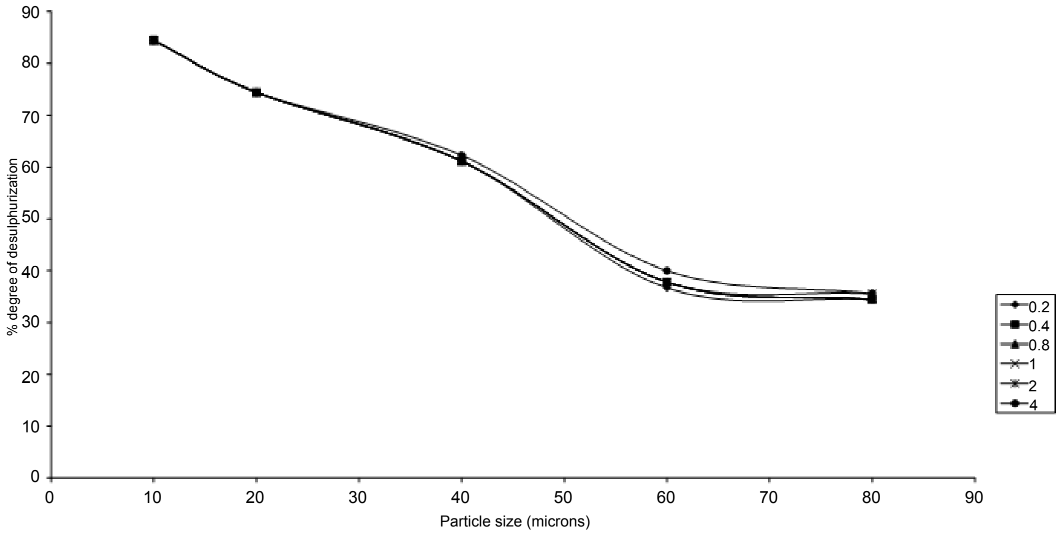

Figure 18: Effect of particle size on the %degree of reduction of sulphur for 10mins using H_2SO_4.

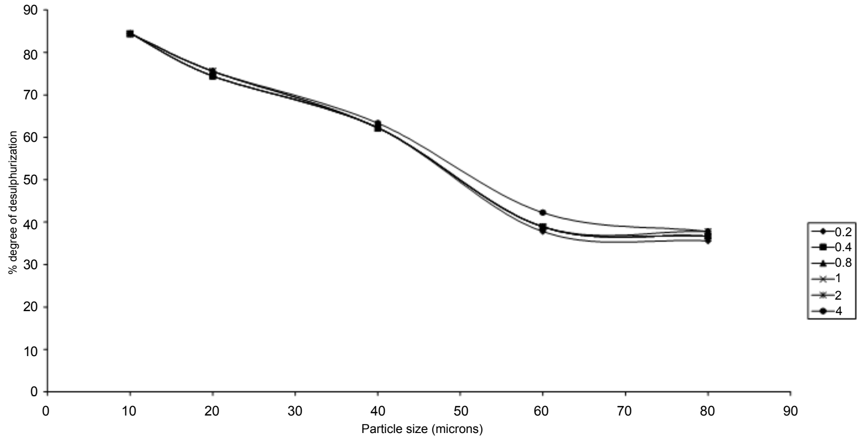

Figure 19: Effect of particle size on the %degree of reduction of sulphur for 20mins using H_2SO_4.

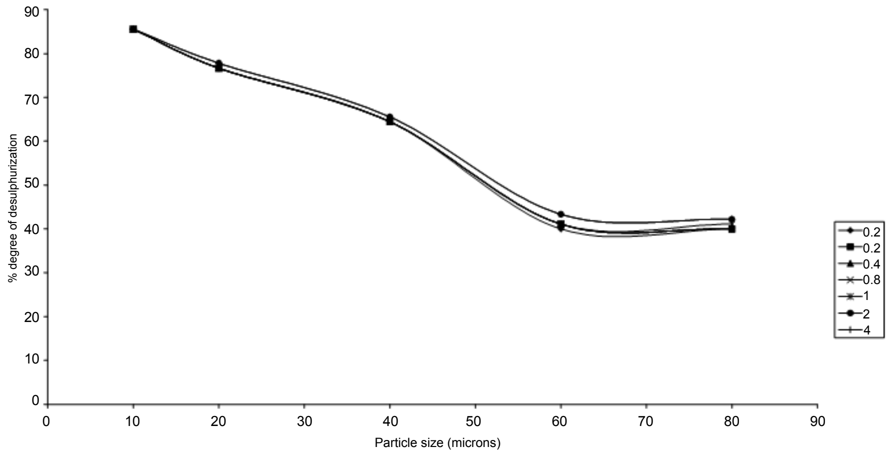

Figure 20: Effect of particle size on the %degree of reduction sulphur for 30mins using H_2SO_4.

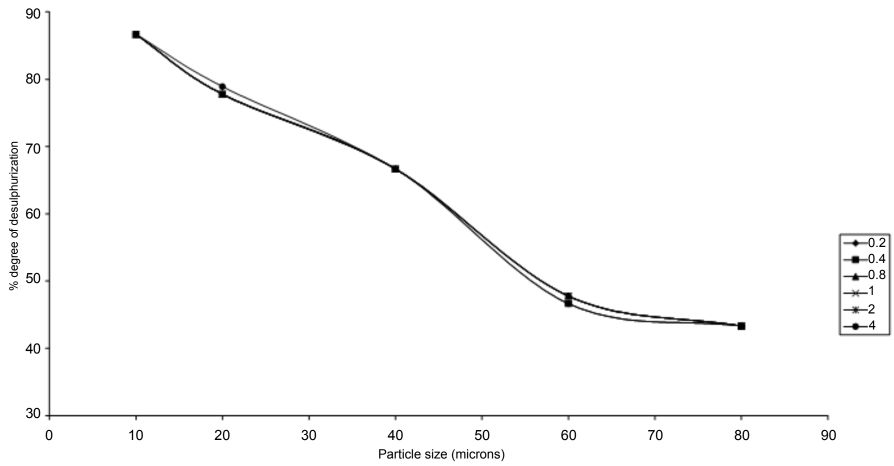

Figure 21: Effect of particle size on the %degree of reduction of sulphur for 60mins using H_2SO_4.

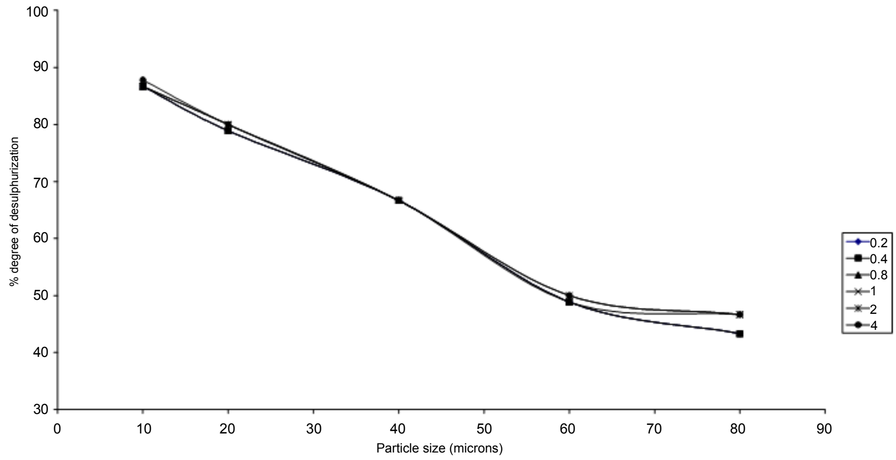

Figure 22: Effect of particle size on the %degree of reduction of sulphur for 120 mins using H_2SO_4.

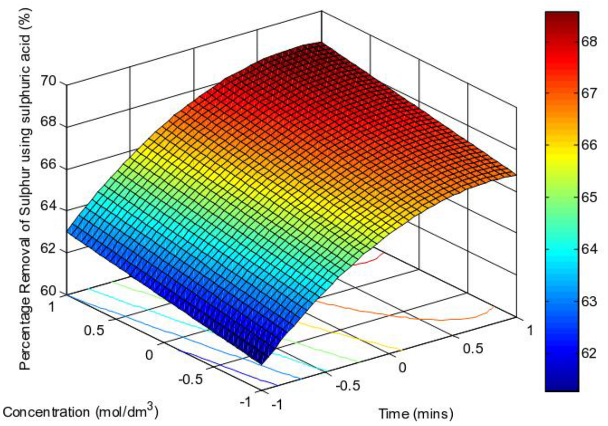

Figure 23: Percentage reduction of sulphur content using H_2SO_4 (time and concentration as variables).

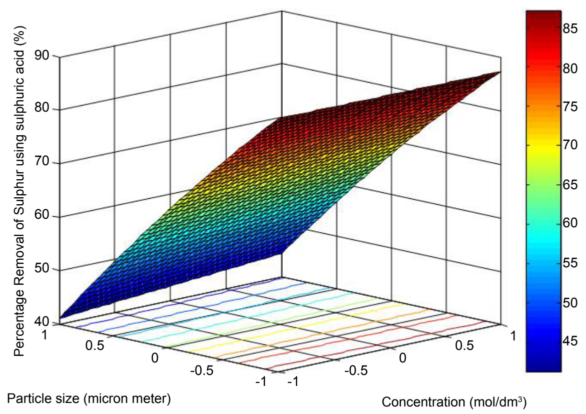

Figure 25: Percentage reduction of sulphur content using H_2SO_4 (particle size and concentrations as variables).

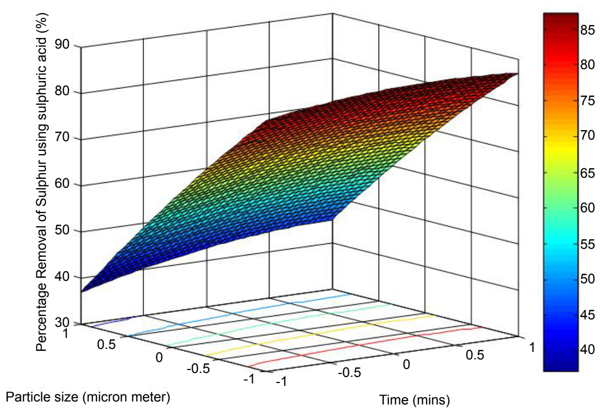

Figure 24: Percentage reduction of sulphur content using H_2SO_4 (time and particle as variable).

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 model was 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 are 87.77%. It is quite significant to note that the optimum values for percentage reduction of sulphur content obtained from the model for H_2SO_4 is 87.73%.

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. 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.

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