

# Effect of Process Variables on the Oxidative Dephosphorization of Agbaja Iron Ore

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**Abstract:** Effect of process variables on the oxidative dephosphorization of high -phosphorus Agbaja iron ore were studied in the process of oxidative-reduction, chloridizing and leaching. Various parameters, including increasing concentrations (CaCl<sub>2</sub> and HCl), temperature and contact time, and reducing particle size were investigated. The material was collected, pulverized and sieved to produce different particle sizes. The ore was roasted at different temperatures. The elemental composition has been carried out using XRD technique. The Chemical composition of the unscrubbed, scrubbed, roasted and leached iron ore was determined. It was observed that increasing temperature, contact time, concentration and decreasing particle size gave a good dephosphorization. The results of oxidative- reduction, chloridizing and leaching are significantly improved by addition of calcium chloride and HCl, in comparison with those in the absence of additives and acid combined. During the oxidative –reduction and chloridizing, phosphorus oxide of 0.6% was obtained from Agbaja iron ore containing 1.5% phosphorus content at temperature of 900°C, particle size of 63µm and 19g additive after 45mins when roasted in the presence of calcium chloride. For the leaching process, temperature of 100°C, particle size of 63µm and acid concentration of 1.2M at leaching time of 120mins gave a maximum dephosphorization at 0.20% of the oxide composition of phosphorous. Thus effect of process variables on Agabaja iron ore can successfully be studied under roasting and leaching process.

**Keywords:** Agbaja-Kogi iron ore, Effect, Dephosphorization, Oxidative, Process variables.

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## I. INTRODUCTION

As oil is fast depleting, there is increasing need for the country to diversify to other natural resources for its economic growth. Iron ore which is one of the mineral resources is an alternative to oil for economic development. Generally, iron is one of the biggest eight element in the earth's crust, being the fourth most abundant element at about 5% by weight. Iron is the world's most commonly used metal - steel, of which iron ore is the key ingredient, representing almost 95% of all metal used per year. In recent times, an increase in world steel production has increased demand for iron ore with consequent increase in the price for this commodity, making hydrometallurgical phosphate removal viable (Kokal et al, 2003). Nigeria is one of the richest countries of the world in terms of mineral deposits; among these deposits is iron ore located at Agbaja in Kogi State of Nigeria (Obiorah et al., 2011). This iron ore is in largest quantity in Agbaj kogi of Nigeria. The Nigeria's Agbaja iron reserve, which according to Uwadiale (1991) is over 1.2 billion tons, is part of a much larger formation called the 'Lokoja ironstone; covering a surface are of 400km<sup>2</sup> and contains at minimum 2,300 million tons (Astier et al, 1989). The Agbaja iron ore reserve with an estimated 47% F<sub>c</sub> content is, however, also associated with high phosphorous (P) content and has been categorized as non beneficial (Amadi et al, 1982; Uwadiale and Nwoke, 1983). The two problems of the high phosphorus content in Agbaja iron ore and its beneficiations difficulties, which were subjects of sustained investigation by many researchers in the early 1980s were not addressed and it led to the abandonments of the reserve (Obot et al., 2012). Heat treatment and subsequent leaching is a way for upgrading high-P iron ores (Feld et al., 1968; Gooden et al., 1974; Muhammed and Zang, 1989; Kokal 1990; Araujo et al., 1994; Cheng et

al., 1999; Kokal et al., 2003. The phosphorus occurring as apatite phase could be removed by alkali-leaching, but those occurring in the iron phase could not (Ameh et al., 2013), therefore this present study will be basically on the effect of process variables on Agbaja iron ore during oxidative dephosphorization in the presence of alkaline earth metal halides calcium chloride ( $\text{CaCl}_2$ ) using hydrochloric acid.

## II. MATERIALS AND METHODS

### Sample collection and characterization:

The working iron ore sample was randomly collected by heap coned to pack the desired quantity from Agbaja in Kogi State of Nigeria. The iron ore sample was crushed using crushing machine in order to expose the surface area for scrubbing process. The pulverized unscrubbed ore was subjected to chemical analysis using XRF in order to define the chemical composition of the crushed raw Agbaja iron ore. After which it was scrubbed using sodium silicate and oleic acid, then dried and subjected again to chemical composition analysis using XRF.

### Roasting of ore:

Different set of roasting with Particle sizes of 63, 90, 125 and 150 $\mu\text{m}$ ,  $\text{CaCl}_2$  additive to iron ore dosage ratio of 10:90, 13:87, 16:84, 19:81 at different temperature 500 $^\circ\text{C}$ , 600, 700 and 900 $^\circ\text{C}$ . After cooling, the roasted mass was sent to XRF, the result obtained shows the effect of roasting with additive on the iron ore as to the extent of dephosphorization.

### Leaching of the ore:

The roasted ore of different sizes were transferred to a leaching vessel and converted to soluble form by adding dilute HCl acid to leach. 1.7-10% HCl acid was prepared from 37% bottle with the molarity of 0.2M – 1.2M. Each mole of the dilute HCl was poured into the beaker containing the cooled roasted mixtures at particle sizes 53.7958, 63, 90 and 99.2042(100)  $\mu\text{m}$  and stirred vigorously to obtain homogeneity then heated on a hot plate at different particle size and a temperature of 100 $^\circ\text{C}$ . At the end of each leaching time of 60min 80mins, 90mins and 120mins, the particular mixture was filtered using a filter paper and filtration funnel and the residue washed with distilled water to free it from soluble salts. The washed residue was air dried and sent to XRF for chemical analysis to determine the extent of dephosphorization.

## III. RESULTS AND DISCUSSION

The chemical analysis of the raw iron ore, scrubbed iron and roasted ore was carried out by XRF.

**Table 1: XRF results of Agbaja iron ore before and after scrubbing, roasted ore.**

S/N	Components	Oxides composition %		
		Raw ore	Scrubbed ore	Roasted ore
1	$\text{Fe}_2\text{O}_3$	85.95	92.15	86.34
2	2Fe	60.11	64.45	60.40
3	$\text{SiO}_2$	4.10	3.13	5.80
4	$\text{P}_2\text{O}_5$	1.40	1.50	1.30
5	$\text{Al}_2\text{O}_3$	4.60	1.96	5.20
6	CaO	0.20	0.14	0.09
7	Cl	-	-	3.83

Table 1 of the XRF technique shows the percentage oxide compositions of iron, phosphorus and other minor elements. After scrubbing the pulverized iron ore, it was observed that the compositions of the elements increased but began reducing during roasting with additive. It can be seen that after roasting with calcium chloride, chlorine was introduced into the iron ore.

**Effects of process variables on Agbaja iron ore during roasting:**

The effect of process variables on the phosphorus present in the iron ore during roasting. It can be seen from figures below that increase in oxidative additive gave a better decrease in phosphorus from the iron ore while decrease in particle size contributed to dephosphorization. The effects are shown in the plot below.

- **The effect of particle size on % composition of phosphorus:**

Fig 1 showed how particle size influences phosphorus reduction during roasting. It can be seen from the plot that as particle size decreases, there is increase in phosphorus removal.

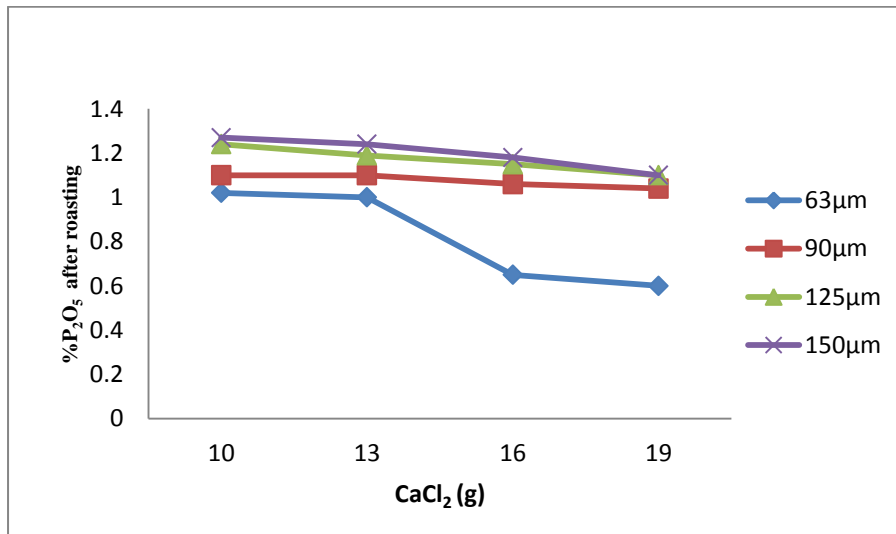


Fig 1: effect of particle size on phosphorus during iron ore roasting

- **The effect of temperature on % composition of phosphorus:**

Fig 2 below showed that temperature has affected the phosphorus during roasting of Agbaja iron ore as increase in temperature increased the rate of phosphorus removal.

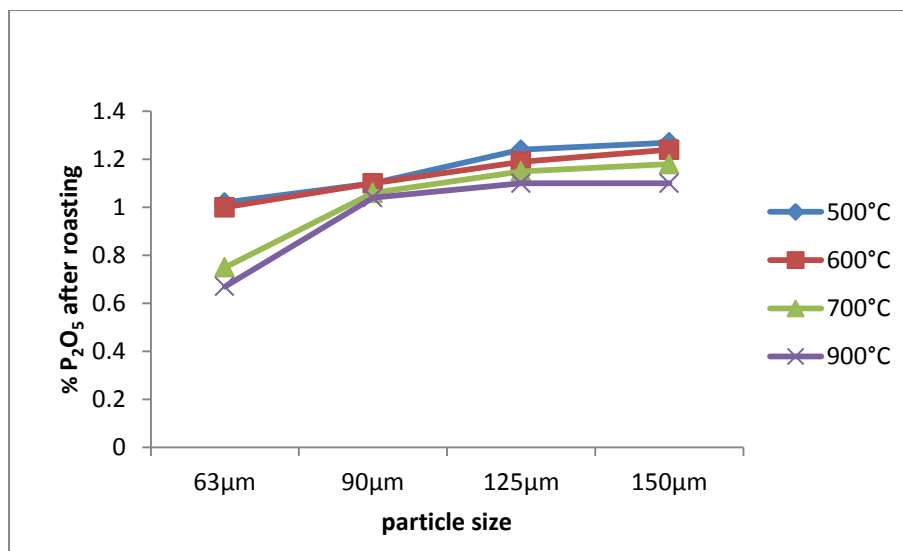


Fig 2: Effect of temperature on phosphorus during iron ore roasting

- **The effect of CaCl<sub>2</sub> additive on % composition of phosphorus:**

Calcium chloride, an alkaline earth metal was used to roast Agbaja iron ore; CaCl<sub>2</sub> had influenced the phosphorus in the ore as it helped in reduction of phosphorus as will be seen in figure 3 below.

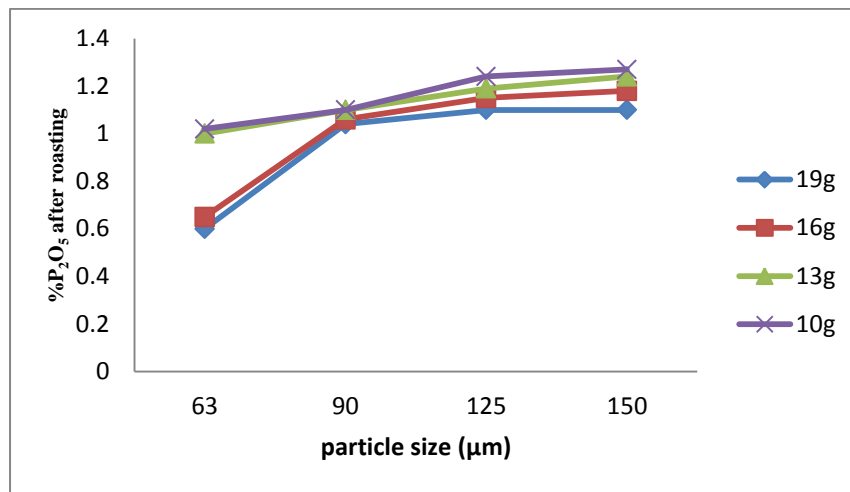


Fig 3: Effect of CaCl<sub>2</sub> (alkaline earth metal additive) on phosphorus during iron ore roasting.

During roasting of iron ore with alkaline earth metal, Figs. 1, 2 and 3 showed the effects of process variables on phosphorus present in Agbaja iron ore during roasting. In fig 1, it can be observed that maximum reduction of phosphorus oxide is achieved at lowest particle size of 63μm as d smaller the particle size, the larger the surface area is created for the additive to attack the phosphorus in the ore for maximum removal. Fig 2 showed that roasting at a higher temperature has helped in reducing the phosphorus, and fig 3 equally showed that in the presence of calcium chloride, phosphorus was removed to a great extent.

**Effect of process variables on leaching:**

The effects of process variables such as particle size, acid concentration, temperature and contact time had been studied. It were observed that decreased in particle size creates more surface area and leads to increased in dephosphorization, and from the literature, particle size creates large surface area on the iron ore for effective leaching. Leaching time also plays important role in leaching process, as process duration increase; it gives the acid more time to attack phosphorous in the iron ore and also the higher the quantity of acid concentration for the process, the higher the rate of extraction of the impurity phosphorus.

• **Effect of particle size on oxidative dephosphorization:**

The effect of particle size was studied as a process parameter at a temperature of 100°C, 1.2M acid concentration and at varying contact time. It can be seen from fig 4 that decreased in particle size affected the rate dephosphorization. During leaching, as the particle size decreased from 99.2μm to 53.79, it increased removal of phosphorus, which was in accordance with the previous works as stated in the literature.

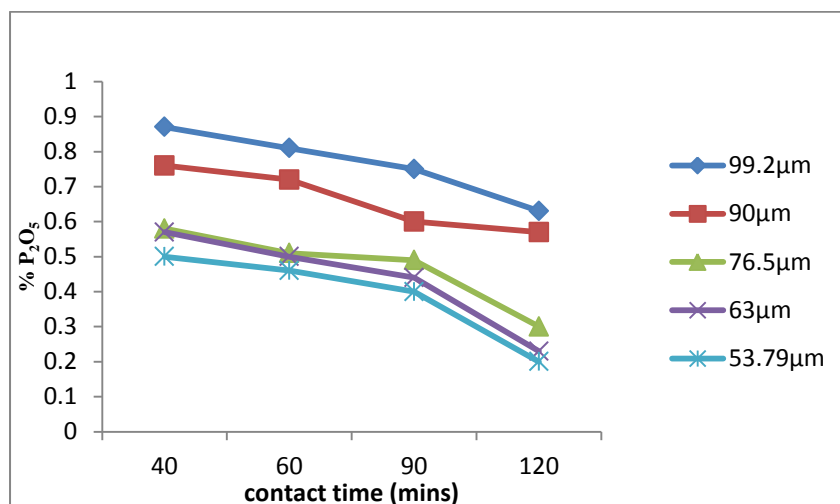


Fig 4: Effect of particle size on leaching

• **Effect of contact (leaching) time on oxidative dephosphorization:**

Fig 5 studied the effect of leaching time using particle size on oxidative dephosphorization, it showed that time was also a contributory factor to phosphorus removal. As the contact time was prolonged, it gave more room for the acid to attack the phosphorus in the iron ore thereby reducing it to a great extent. It agrees with the previous report on effect of contact time on iron ore leaching.

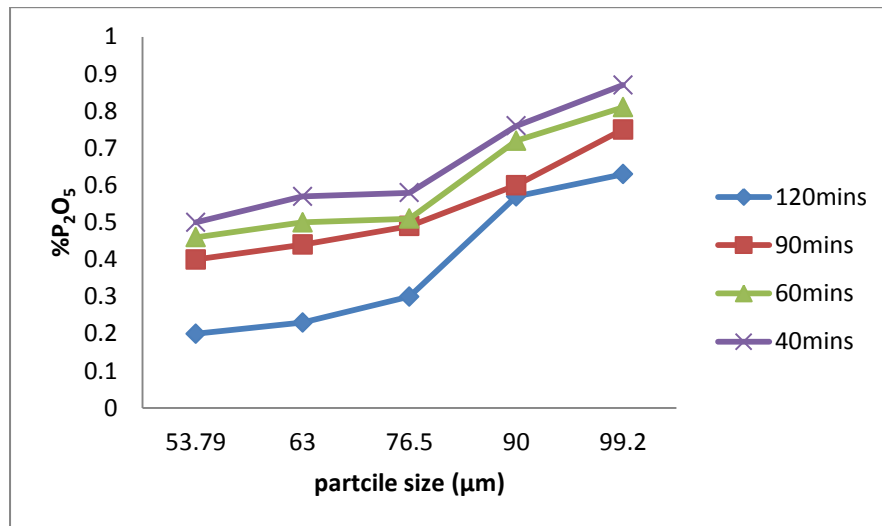


Fig 5: Effect of contact time on leaching

• **Effect of temperature on oxidative dephosphorization:**

Fig 6 showed that increased in temperature from 30 to 100°C gave rise to rate of extraction of phosphorus from the ore. When the temperature is raised high, good dephosphorization was achieved. This was in accordance with previous report on dephosphorization.

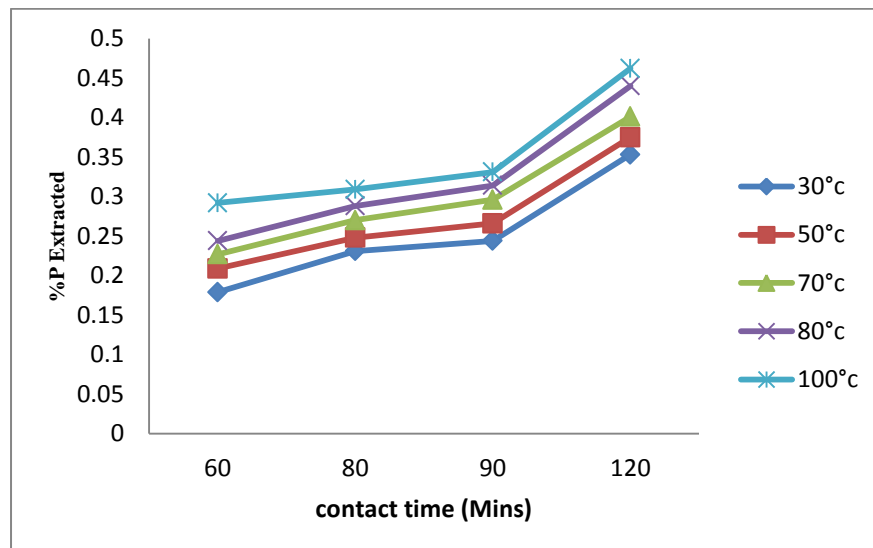
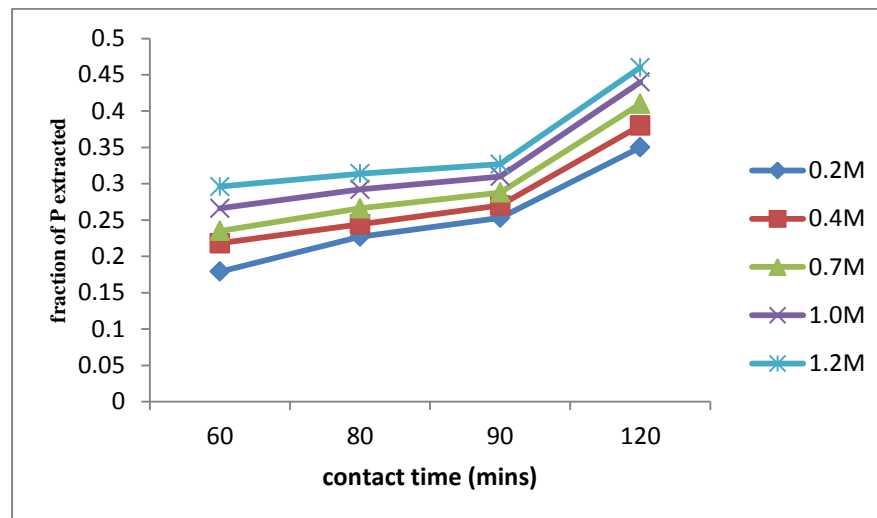


Fig 6: Effect of temperature on oxidative desphosphorization

• **Effect of acid concentration on oxidative dephosphorization:**

From the fig 7, it can be seen that increased HCl acid concentration from 0.2M to 1.2M gave rise to fraction of phosphorus that were extracted from the ore. This was in accordance the previous authors, that increasing the amount of acid concentration for the leaching process increased the fraction of phosphorus impurity removed.



**Fig 7: Effect of acid concentration on oxidative dephosphorization**

#### IV. CONCLUSION

In this current study, the possibility of making use of an alkaline earth metal ( $\text{CaCl}_2$ ) to roast Agbaja iron ore before commencement of HCl acid leaching were studied using the process variables. The roasting of the iron ore was found to be dependent on the roasting process variables such as oxidative additive, particle size and temperature, and the leaching was dependent on the independent variables too, such as temperature, (contact time) leaching time, acid concentrations and particle size of iron ore sample. And it had proven that dephosphorization of Agbaja iron ore is dependent on the aforementioned process variables.

#### REFERENCES

- [1] Araujo, D. Fonseca, and C. Souza, Hydrometallurgical routes for the reduction of phosphorus in iron ore. In : Wilkomirsky, I, Sanchez, M. and Hecker, C (Eds). A Sutulov memorial volume, Vol III, Chemical Metallurgy. Iv meeting of the southern Hemisphere on Mineral Technology, Universidad de Concepcion, Concepcion Chile, 83 – 92, 1994.
- [2] Cheng, V. Misra, J. Clough, and R. Mum, Dephosphorization of Western Australian iron ore by hydrometallurgical process. Mineral Engineering, 12, 1083 – 1092, 1999.
- [3] G.G.O.O Uwadiale ‘‘Electrolytic Coagulation and Selective Flocculation of Agbaja Iron Ore’’ J. Min. Geol. 1991.
- [4] G. G. O. O. Uwadiale ‘‘Upgrading Nigerian iron ores,’’ Minerals and Metallurgical Processing, vol. 6, no. 3, pp. 117–123,1989.
- [5] G. G. O. O. Uwadiale, M.A.U Nwoke ‘‘ Beneficiation of Agbaja Iron Ore by Reduction Roasting- Magnetic Separation: Semi Pilot Plant Scale-up and Establishment of Residence Point of Phosphorus’’ National Steel Council, Metallurgical Research and Tests Division, Jos, Nigeria. 1983.
- [6] H.R. Kokal, M.P. Singh, and V. Naydyonov ‘‘Removal of phosphorus from Lisa kovsky iron ore by roast – leach process’’. Hydrometallurgy – proceedings of the 5th international symposium, Vol, 2 Vancouver, B. C. Canada. The minerals, metals and materials, society (T M S ) Warrandale PA 15086, USA ,2003.
- [7] H.R Kokal ‘‘The origin of phosphorus in iron making raw materials and methods of removal’’ AIME Proceeding, 51st Annual Mining symposium Duluth – Minnesota, 225-258.January 17-18, 1990.
- [8] Ddd I. Feld, T. Franklin, and M. Lampkin, Process for removing phosphorus from iron ores. US patent No. 3, 402, 041, 1968.

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- [9] J.E Astier, M Donzeau, G.G.O.O Uwadiale “The Loklja Oolitic Ironstone Deposit: Possible Use in the Ajaokuta Iron and Steel Plant” J. Min. Geol., 25: 1-2, 1989.
- [10] J. Gooden, W. Waller, and R. Allen, ADEMPHOS- a chemical process for decomposition of iron ore. Proceedings of the National Chemical Engineering conference, process industries in Australia – impact and growth sinters paradise, Queensland, Australia , 38 – 49, 1974.
- [11] M. Muhammed, and Y. Zhang, “A hydrometallurgical process for the dephosphorization of iron ore. Hydrometallurgy” 277 – 292, 1989.
- [12] N.J Amadi, Odunaike AA, Mathur JP “Preliminary Bench Scale Beneficiation Studies with Three Lumps of Iron Ore Sample from Agbaja”. (Tech. Rep.). Central Metallurgical Research and Development Centre, Jos, Nigeria. 1982.
- [13] O. W. Obot and C. N. Anyakwo ”Removal of phosphorus from Nigeria’s Agbaja iron ore through the degradation ability of Micrococcus species” International Journal of Water Resources and Environmental Engineering Vol. 4(4), pp. 114-119, 2012.
- [14] S.M.O. Obiorah, M.C.Menkiti and E.E.Nnuka, “ Beneficiation Processing of Agbaja Iron Ore By Chemical Leaching Technique” New York Science Journal. 2011.