

Comparative Effectiveness of Flotation Technique at Varying Conditions for Beneficiation of Itakpe and Agbaja Iron Ores

Serah Akande, Oladunni O. Alabi, E. O. Ajaka, and Temitope A. Olatunji

Abstract—Itakpe and Agbaja iron ores are part of prominent iron deposits in Nigeria, yet studies on their beneficiation via froth flotation are relatively limited. Thus, this research investigated comparatively the flotation behaviour of both ores at varied pulp pH, particle size, and collector type. The ores were also examined using Energy Dispersive X-ray Fluorescence Spectroscopy, Petrological, and fractional sieve size analyses. Fifty (50) kg sample each of the ores was sourced for the research. Then, size fractions (63, 75, and 125 μm) of each ore were prepared and subjected to froth flotation using different collectors; Potassium Amyl Xanthate (PAX), Sodium Ethyl Xanthate (SEX), and Oleic Acid, at varying pulp pH ranging from 9-11. From the results obtained Itakpe iron ore assayed 36.18% Fe_2O_3 and contains predominantly haematite, sillimanite, and quartz while Agbaja iron ore contains chiefly, quartz and haematite, and assayed 40.6% Fe_2O_3 alongside 1.505% P_2O_5 . The liberation sizes of both ores lie favourably in the range -125+75 μm . Beneficiation studies carried out revealed that significant enrichment of both ores was actualized. Thus, it was established that Itakpe iron ore is best processed using PAX at pH 11 and particle size of 125 μm yielding concentrates assaying 67.66% Fe_2O_3 at a recovery of ~90% while for Agbaja iron ore, PAX at pH 9 and particle size of 63 μm is considered best to yield enriched concentrates assaying 65.5% Fe_2O_3 at 52.5% recovery.

Index Terms—Agbaja and Itakpe Iron Ores, Comparative Effectiveness, Froth Flotation.

I. INTRODUCTION

Nigeria's iron ore exploration dated as far back as the 1970s when viable quantities were discovered at Itakpe, Kogi State with an estimated reserve of 200 million tonnes [1]. Other deposits explored include Agbaja, Ajabanoko, and Chokochocko; Agbaja's reserve, estimated to be about 2 billion tonnes has been the largest so far [1][2]. These discoveries birthed processing plants such as the Ajaokuta Steel Company charged with processing iron ore sourced from Itakpe deposit into steel and other products [3]. The modalities of this industry entail several plants, among

which is the froth flotation plant, yet to be developed to full capacity [4]. The chief beneficiation technique adopted to produce high-grade iron ore concentrates as feed for the agglomeration plant is froth flotation [5].

A. Froth Flotation: A Method of Mineral Separation

This is a physicochemical process that exploits the disparity in the surface properties of minerals; hydrophobicity and hydrophilicity, and the use of some chemical reagents to effect liberation [6]. Froth flotation can either be direct or reverse depending on the intended mineral of interest [7]. Froth flotation facilitates the beneficiation of intermediate and low-grade iron ores as alternatives to the depleting reserves of high-grade iron ore [8]. The majority of known iron ore deposits, including that of Itakpe and Agbaja, contain high siliceous minerals which are not fully liberated by the conventional gravity and magnetic techniques [9]. These minerals are found interlocked with the iron minerals, and liberation can only be effected at relatively fine sizes below 125 microns [10]. Since, the steel industry requires concentrates of bearable contents of silica, alumina, and other impurities, the need to employ sustainable, economical and effective beneficiation techniques, such as froth flotation, is imperative [11].

Experimental quantitative and qualitative data on the flotation behaviour of minerals are pertinent as they provide adequate information for the design and implementation of the processing plant. Flotation efficiency is influenced by parameters such as pulp pH, collector type, pulp density, and particle size [7][12]. These parameters are varied with the sole aim of arriving at sets-of-conditions that enhance separation efficiency to yield high grade concentrates at optimal recovery. Therefore, this research analyses comparatively the flotation behaviour of Itakpe and Agbaja iron ores at varied process parameters. Consequently, technical data provided therein can be exploited in the development of efficient froth flotation routes that can be utilized at the Ajaokuta Steel Company.

II. MATERIALS AND METHODS

The procedural chart employed to achieve the objectives of this study is as shown in Fig. 1.

A. Materials Sourcing

Samples of Itakpe and Agbaja iron ores were sourced from the deposit areas located in Kogi State, Nigeria as shown in Fig. 2. Itakpe deposit is 7 km from Okene town having the geographical coordinates of latitude $7^{\circ}36'52''$ N and longitude $6^{\circ}19'7''$ E while Agbaja deposit is 15 km from

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Agbaja Central having geographical coordinates of latitude 7.9833° N and longitude 6.6500° E [2][9]. Fifty (50) kg representative sample was sourced from each deposit.

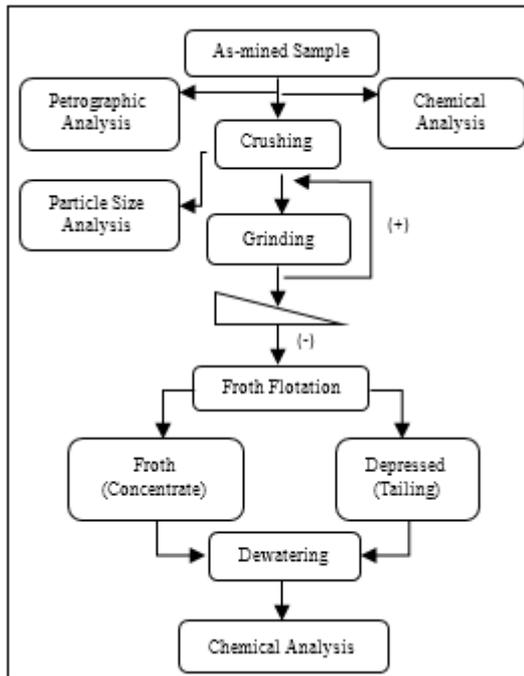


Fig. 1. Procedural flowchart employed

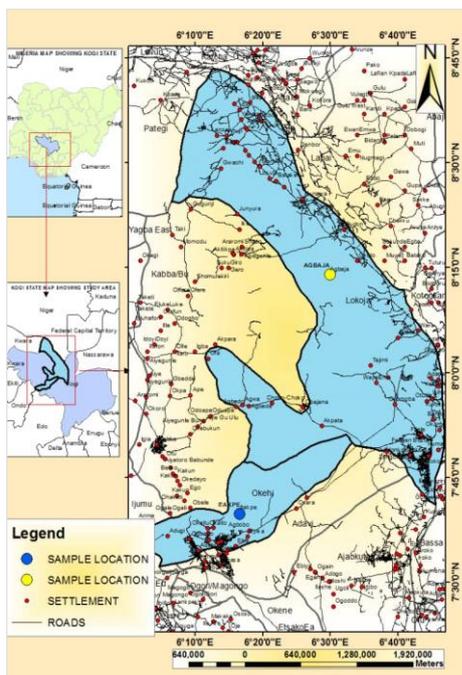


Fig. 2. Geographical map of the deposits

B. Petrological Analysis

Core Samples of the two ores were cut to standard sizes and their surfaces prepared by grinding using 500 μm and 1000 μm emery papers and then observed using the Leica Petrological Microscope (Model: EGB 100 DMX). The microstructural features of the two ores were analyzed under plane-polarized light at satisfactory magnifications to identify the mineral phases present therein.

C. Chemical Composition Analysis

The chemical compositions of the two ores were determined using Energy Dispersive X-ray Fluorescence Spectrometer (Model: PANalytical Minipal 7). Then, 100 g of each ore was pulverized to about 200 mesh, 20 g sample each was weighed out, mixed with cellulose binder, and pelletized at a pressure of 10-15 tons/in². The formed pellets were desiccated and then analyzed using the pre-warmed ED-XRF machine which identifies minor and major elements therein in percent concentration.

D. Comminution and Particle Size Analysis

The as-mined samples were crushed to 50 mm using a sledgehammer, and then further reduced to 5 mm using a Denver D12 laboratory jaw crusher. The crushed samples were charged into a roll crusher to be further reduced to 1000 μm and then thoroughly homogenized. Fractional sieve analysis technique was adopted to assess the particle size distribution of the two ores and also ascertain their respective liberation sizes. A set-of-sieves arranged from 500–63 μm as per $\sqrt{2}$ series with a pan placed at the bottom end was utilized. Then, 500 g sample was charged onto the upper sieve (500 μm), and the set-of-sieves agitated for 30 minutes using an Automated Sieve Shaker (Model: Endecott AS400 control). The sieves were separated and the retained mineral particles on each measured to evaluate the cumulative percentage weight retained and passing for respective sieve sizes. Samples of the size fractions were also chemically characterized using ED-XRF.



Fig. 3. Denver D12 flotation cell

E. Concentration by Froth Flotation

The essence of this test is to establish the parametric data required for the optimal recovery of iron minerals from both Itakpe and Agbaja iron ores by direct froth flotation technique. A Denver D12 Flotation Cell (Fig. 3) of capacity 800 ml was employed. Five (5) kg crushed sample of each ore was pulverized to three size fractions; 63, 75, and 125 μm . These sizes were selected having carried out particle size analyses of the two ores. Significant liberation was achieved for both ores at these sizes and they also fall within 250–45 μm , which depicts the satisfactory size range for the flotation of iron ores (Wills and Napier-Munn, 2006). Then,

100 g of the 63 μ m sample was mixed with 500 ml of water and agitated for 2 minutes to form 16.7 wt. % solid pulp. The initial pH of the pulp was measured to be 8.2 using a pH meter. Thereafter, drops of sodium hydroxide (depressant) were added to adjust the pH to 9 followed by further agitation for 2 minutes. Then, two (2) drops of cornflour syrup (depressant) were added and the mixture was further agitated for 2 minutes, followed by the addition of two drops of potassium amyl xanthate, PAX (collector). The mixture was further stirred for 2 minutes after which 2 drops of methyl isobutyl carbinol (Frother) were added to ensure the stability of bubbles formed. The mixture was then stirred for another 2 minutes, thereby bringing the total

agitation time to 10 minutes per process. The machine's air valve was opened to introduce air into the pulp, thereby causing the formation of froth on the pulp's surface. The froth containing the iron mineral was skimmed off into a container leaving behind the unwanted minerals. This process was repeated at pH of 9 and 11 while the other collectors, sodium ethyl xanthate (SEX) and oleic acid were used instead of PAX. The above procedure was then repeated for the 75 μ m and 125 μ m samples of both Itakpe and Agbaja ores. The resulting products (froth and depressed) were filtered and oven-dried for 24 hours. Then, samples were taken for chemical analysis.

III. RESULTS

The results obtained are presented in Tables I-II and Fig. 4-9.

TABLE I: MINERALOGICAL MODAL ANALYSES OF PETROGRAPHIC SLIDES OF ITAKPE AND AGBAJA IRON ORES

Mineral	No. of Counts				Modal Count
	1 st View	2 nd View	3 rd View	Total	
<i>Itakpe Iron Ore</i>					
Quartz (Q)	43	41	48	132	38
Sillimanite	10	1	-	11	3
Haematite and other Opaque minerals (H, Op)	67	65	68	200	59
Total	120	107	116	343	100
<i>Agbaja Iron Ore</i>					
Quartz (Q)	45	53	49	147	58
Haematite and other Opaque Minerals (H, Op)	37	34	40	111	42
Total	82	88	89	258	100

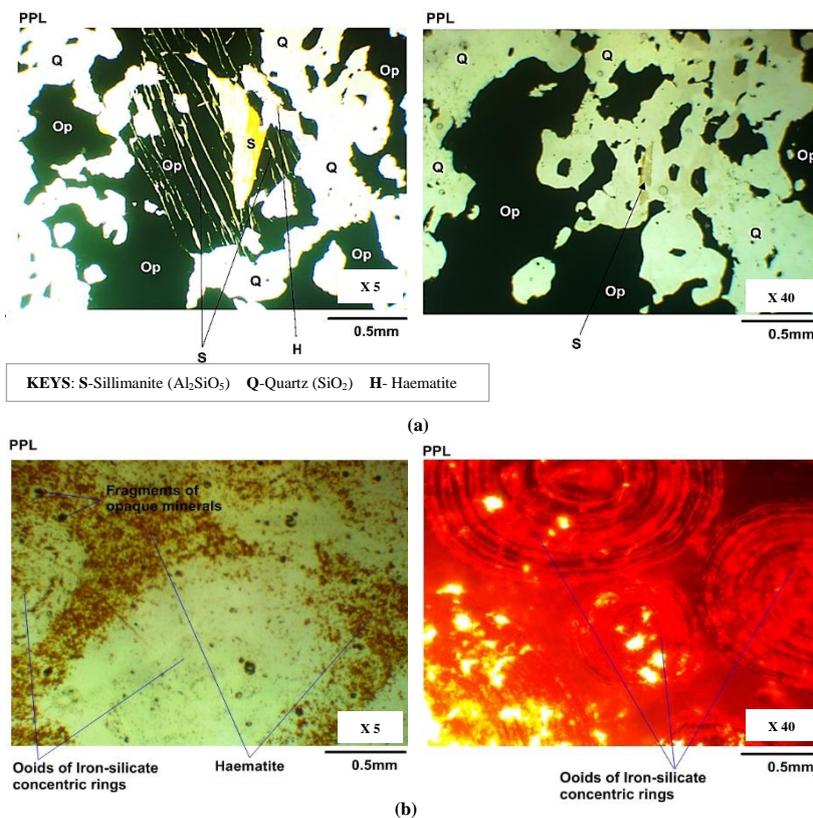


Fig. 4. Photomicrographs of the crude sample of (a) Itakpe, and (b) Agbaja iron ores at different magnifications

TABLE II: ED-XRF RESULT OF ITAKPE AND AGBAJA IRON ORES

Chemical composition of crude Itakpe iron									
Compounds	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	V ₂ O ₅	MnO	Fe ₂ O ₃	CuO	ZnO
% composition	4.20	53.05	0.24	0.559	0.008	0.068	36.18	0.034	0.006

Chemical Composition of Crude Agbaja Iron Ore									
Compounds	Al ₂ O ₃	SiO ₂	SO ₃	P ₂ O ₅	K ₂ O	CaO	Cr ₂ O ₃	Fe ₂ O ₃	CuO
% Composition	5.50	50.5	0.053	1.505	0.0089	0.1041	0.0193	40.6	0.025

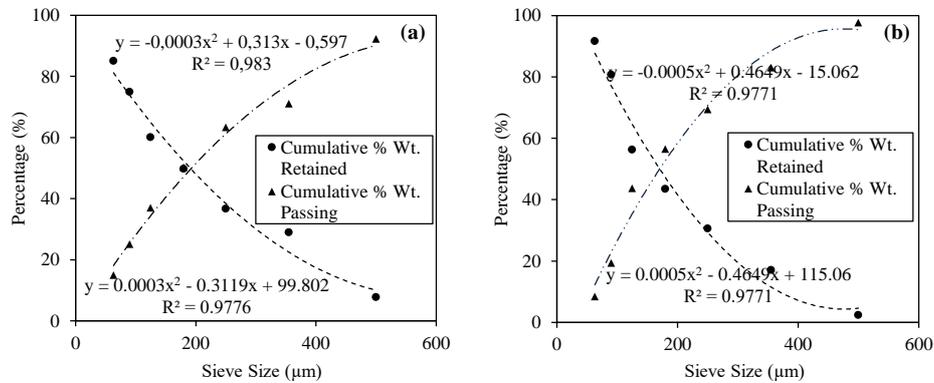


Fig. 5. The plot of cumulative % weight retained and passing of (a) Itakpe, and (b) Agbaja iron ores against sieve size (µm)

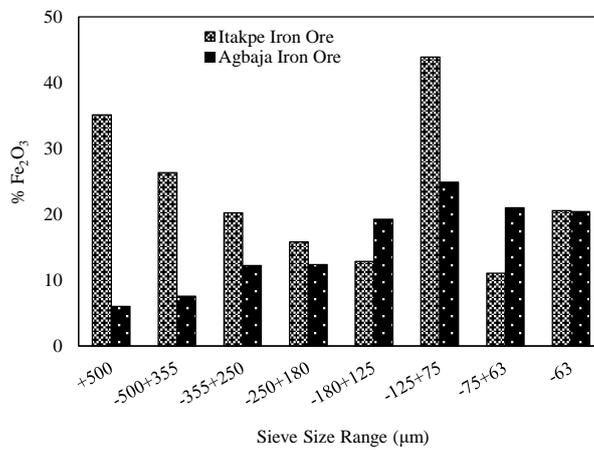


Fig. 6. A comparative appraisal of the Fe₂O₃ content in size fractions of Itakpe and Agbaja iron ores

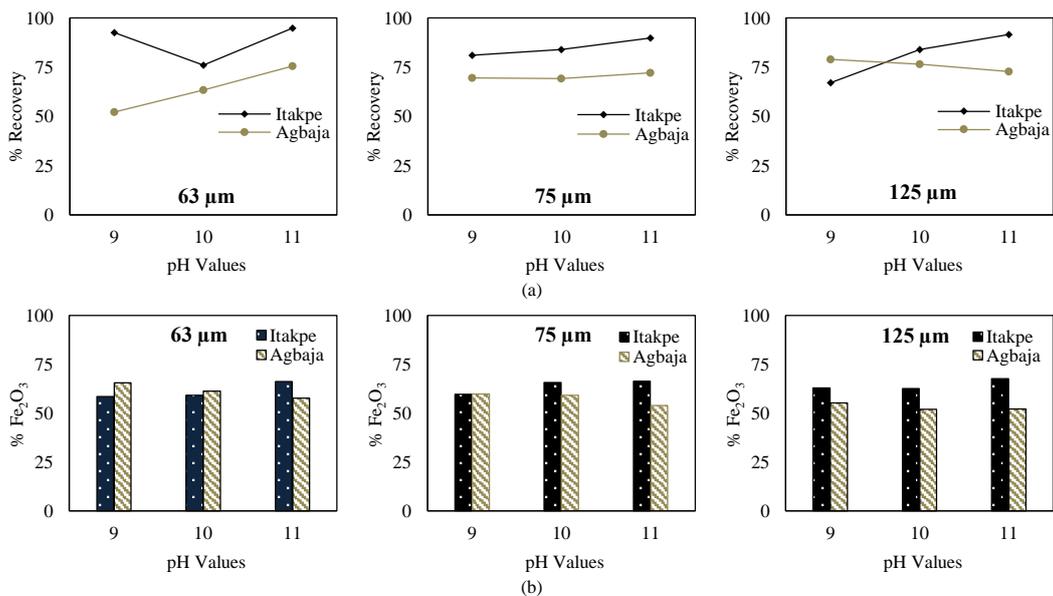


Fig. 7. Variation of (a) % recovery, and (b) assay (%Fe₂O₃) of PAX-processed Itakpe and Agbaja iron ores with pH values at different particle sizes

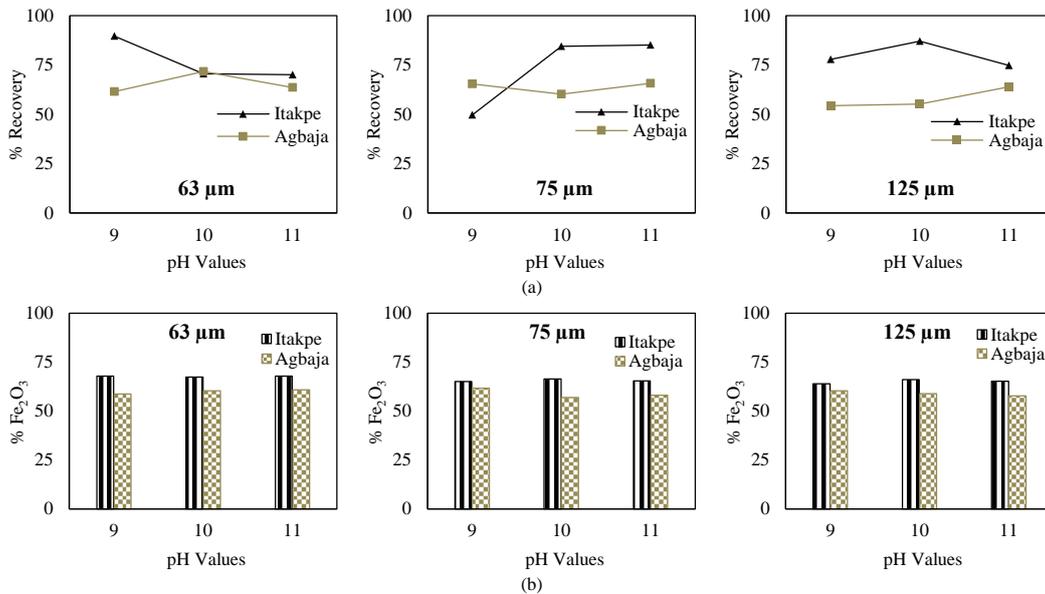


Fig. 8. Variation of (a) % recovery, and (b) assay (%Fe₂O₃) of SEX-processed Itakpe and Agbaja iron ores with pH values at different particle sizes

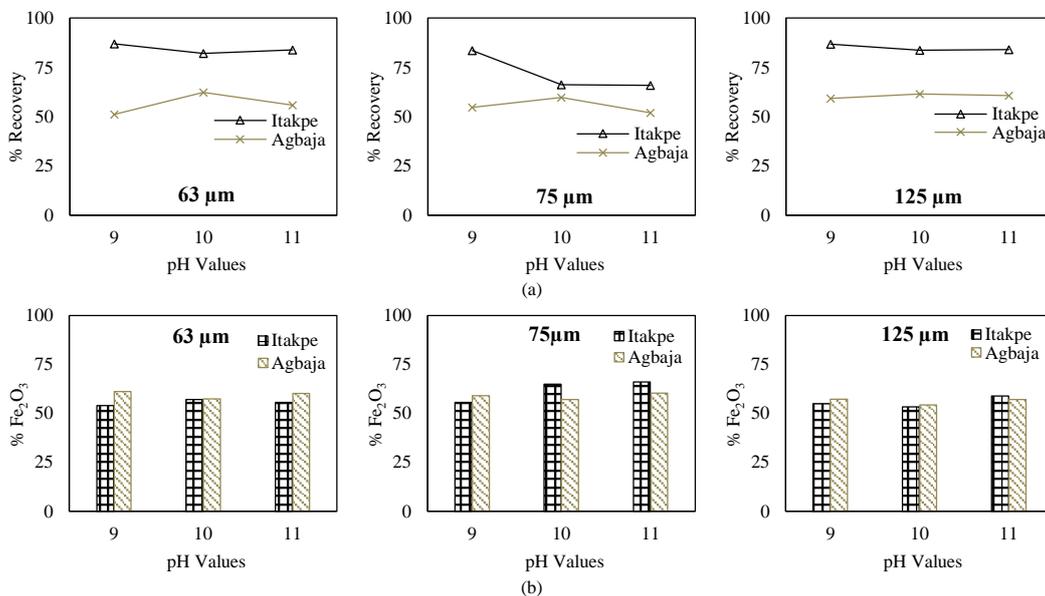


Fig. 9. Variation of (a) % recovery, and (b) assay (%Fe₂O₃) of Oleic Acid-processed Itakpe and Agbaja iron ores with pH values at different particle sizes

IV. DISCUSSION

A. Mineralogical Features of Itakpe and Agbaja Iron Ores

The results obtained for the petrological analysis of both ores are presented in Table I and Fig. 4. It can be deduced from Table I that Itakpe iron ore contains grains of quartz (SiO₂), sillimanite (Al₂SiO₅), haematite (Fe₂O₃), and other opaque minerals having modal counts of 34%, 4%, and 62% respectively. While the mineral grains contained in Agbaja iron ore are quartz (SiO₂) and haematite (Fe₂O₃) alongside other opaque minerals having a relative abundance of 58% and 42% respectively. This implies that the gangue mineral (SiO₂) is more predominant in Agbaja iron ore relative to the desired mineral (magnetite/haematite). Photomicrographs of Itakpe iron ore as shown in Fig. 4a reveal high concentrations of opaque mineral (possibly magnetite), haematite (occurring as reddish-brown stain within the rock), amphibole, and quartz. The reddish-brown specks within the rock depict a low degree of weathering which may not be observable with the ordinary eye. However,

the photomicrographs of Agbaja iron ore (Fig. 4b) reveal ooids of iron silicate of diameter ranging from few microns to 2 mm. The ooids shape vary from rounded to ellipsoid having different elongation degree. This observed surface morphology conforms to the findings in [12]. Also, the observable dark spots across the ore's matrix depict fragments of opaque minerals, mostly magnetite.

B. Chemical Composition of Itakpe and Agbaja Iron Ores

It is apparent from Table II that both ores have appreciable iron content. Itakpe iron ore assayed 36.18% Fe₂O₃, 53.0% SiO₂, 4.20% Al₂O₃, and other trace compounds while Agbaja iron ore contains 40.6% Fe₂O₃, 50.5% SiO₂, 4.50% Al₂O₃, 1.505% P₂O₅ alongside other trace compounds. The major associated minerals in both ores are silica and alumina. The existence of phosphorus in Agbaja iron ore renders it non-economical despite its high iron content and rich reserve relative to Itakpe iron ore. These findings further affirm the petrological analysis result and also conform to that obtained in literature [2][9][11].

C. Liberation Studies of Itakpe and Agbaja Iron Ores

Prior to the concentration of ore, it is pertinent to establish the particle size distribution and liberation characteristics of the ore. This informs the efficiency of comminution to segregate the mineral grains in the ore into different size ranges as well as liberate the desired mineral(s) from the associated minerals. As such, the results obtained from the liberation studies of both ores are presented in Fig. 5 and 6. Fig. 5 illustrates the plots of cumulative percentage (%) weight retained and passing against sieve size for both ores while Fig. 6 shows comparatively, the iron content in size fractions of both ores. From Fig. 5a, it can be deduced that the trendlines of cumulative % weight retained and passing for Itakpe iron ore are mirror images having R-squared values of 0.983 and 0.9776 respectively, and they intercept at 180 μm . This size depicts the economic liberation size of Itakpe iron ore; that is, after comminution about 50% of the ore particles would have sizes below 180 μm . Fig. 5b illustrates the trendlines of cumulative % weight retained and passing obtained for Agbaja iron ore, both lines are also mirror-like having R-squared values of 0.9771 each, and they intercept at 125 μm , which depicts the economic liberation size of Agbaja iron ore. The R-squared values obtained for both ores satisfy the standard R-squared value of >75% which rates the significance of data for analysis [13]. Thus, the data obtained closely fit the regression lines/models with an accuracy of > 90%. Comparing both economic liberation sizes, it can be implied that more energy will be expended to comminute Agbaja iron ore, which renders the ore less economical relative to Itakpe iron ore. From Figure 4, it is evident that the Fe_2O_3 content in Itakpe iron ore decreases from +500 μm to -180+125 μm while that of Agbaja increases progressively. It was also established that the size range having the highest Fe_2O_3 content for both ores was -75+63 μm , having a nominal aperture size of 75 μm . This size depicts the actual liberation size of both ores where the optimal concentration of Fe_2O_3 was achieved. It also falls within the size range suitable for the flotation of iron ores as documented in [7][12].

D. Effects Varying Pulp pH and Particles Size on PAX-Processed Itakpe and Agbaja Iron Ores

The results obtained from the flotation of both ores using PAX at different pH values and particle sizes are presented in Fig. 7. The efficiency of the process was measured in terms of % recovery and assay. Indeed, appreciable increment in the assays of both ores was achieved. Itakpe iron ore assaying 36.18% Fe_2O_3 was enriched to a maximum of 67.66% Fe_2O_3 while that of Agbaja increases from 40.6% Fe_2O_3 to a peak of 65.50% Fe_2O_3 . At particle size of 63 μm , Itakpe iron ore exhibited an upward trend in % recovery while a progressive increase in % recovery was observed for Agbaja iron ore as the pulp pH was varied from 9 to 11. Also, Itakpe iron ore exhibited a progressive increment in assay with increasing pH and vice versa for Agbaja iron ore. It was established that the optimal % recovery for both ores was obtained at pH 11. Thus, it can be inferred that the froth flotation of both ores using PAX at a particle size of 63 μm is best carried out at more alkaline conditions. At particle

size of 75 μm , the % recovery of both ores also increased progressively with increasing pulp pH. Likewise, the assays exhibited the same trend as recorded for 63 μm . However, at 125 μm , % recovery and the assay of Itakpe iron ore increased extensively while that of Agbaja iron ore decreases. This implies that using PAX as the collector for the flotation of Itakpe iron ore would yield considerable recovery at more alkaline conditions for all considered particle sizes. While PAX is more suited for the flotation of Agbaja iron ore at particle sizes of 63 and 75 μm also at more alkaline conditions (pH 11).

E. Effect of Varied Pulp pH and Particle Size on SEX-Processed Itakpe and Agbaja Iron Ores

Fig. 8 presents the plots of % recoveries and assays obtained for both ores against the pulp pH for the particle sizes considered. It is quite apparent that significant enrichment was also obtained for both ores. Itakpe iron ore was enriched from 36.18% Fe_2O_3 to 67.84% Fe_2O_3 while Agbaja's assay rose to a maxima value of 61.63% Fe_2O_3 . At 63 μm , it is evident that the % recovery of Itakpe iron ore decreases sharply as the pH value varies from 9 to 10, and then slightly decreases at pH 11. Conversely, the % recovery of Agbaja iron ore increases as the pH increases from 9 to 10 and then drops at pH 11. The assays of both ores increased appreciably as the pH rises from 9 to 11. The froth flotation of Itakpe iron ore at 75 μm exhibited a sharp increase in % recovery as the pH increases from 9 to 10, and then slightly rises at pH 11. However, Agbaja iron ore exhibited a slight reduction and increment in % recovery as the pH varies from 9 to 11. Also, a slight increment in the assay of Itakpe and Agbaja iron ores was observed at pH 10 and pH 9 respectively. Furthermore, at 125 μm Itakpe iron ore exhibited sloppy trend in % recovery while the % recovery of Agbaja iron ore rises progressively with increasing pH. It can be implied that the % recoveries obtained for the different sets-of-conditions followed no regular pattern and they are quite below that obtained when PAX was utilized. Yet, SEX proves efficient in the flotation of Itakpe iron ore at a particle size of 63 μm and pH of 9 owing to the optimal recovery (89.79%) observed at this set-of-conditions. Likewise, optimal recovery (71.72%) was obtained for Agbaja iron ore at 63 μm and pH of 10.

F. Effect of Varied Pulp pH and Particle Size on Oleic Acid-Processed Itakpe and Agbaja Iron Ores

Fig. 9 illustrates the results obtained from the flotation of both ores using oleic acid at varying pulp pH and particle sizes. Similarly, appreciable enrichment of both ores was obtained using oleic acid. Processed Itakpe and Agbaja iron ores assay a peak value of 66.01% and 61.07% Fe_2O_3 , respectively. Suffice to say that the % recoveries of both ores exhibit converse slopes for each particle size as the pulp pH varies from 9 to 11. In this regard, the effect of neutralization on the efficiency of the collector used (Oleic acid) and % recovery was also pronounced as the pulp becomes more alkaline for all set-of-conditions [14]. This accounts for the observed reduction in % recovery at higher pH values and the poor performance of oleic acid compared to other collectors used. Nonetheless, oleic acid also proved viable to the froth flotation of both ores. The optimal %

recovery of Itakpe iron ore was achieved at 63 μm and pH 9 while Agbaja's was obtained at the same size but pH 10.

V. CONCLUSION

The need to revamp the steelmaking industry is imperative to promote significant technological advancement and economic sustainability. The prospect of this industry calls for a continuous and adequate supply of the major raw material, iron ore. Thus, the need to exploit other alternatives to Itakpe iron ore (the chief raw material for Ajaokuta Steel Company) such as the Agbaja iron ore is pertinent to curb the rapid decline of this deposit, and also promote the blending of both ores as feed for the processing plant. Therefore, on the premise of the findings in this work, further study is proposed to investigate the blending possibilities of both ores to produce super-concentrates.

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