

EFFECT OF REAGENT TYPE ON THE FROTH FLOATATION OF SOKOTO PHOSPHATE ORE

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Abstract

Effect of reagent type on the froth floatation of Sokoto phosphate ore for its beneficiation has been established. The samples of the Sokoto phosphate mineral ore used for the research work were sourced from mining locations in Dange-Shuni, Bodinga, Yabo, Wurno, and Rabbah Local Government Areas of Sokoto State. Size-Assay analysis conducted on scrubbed Sokoto Phosphates nodules revealed that nodules had a size distribution with 80% passing 29.3 mm. Flotation Tests using AERO704 (fatty Acid), Alkyl Hydroxamates, Melamine as collectors (alone or mixed with diesel), MIBC as frother, Calcium Hydroxide and Sulphuric Acid as pH regulators and Dextrin, Sodium Silicate and Aluminium Chloride as depressants produced poor P₂O₅ separation in the flotation products due to very poor liberation associated with very fine mineral grains. Based on the results obtained, AERO704 Collector gave the best result with a P₂O₅ recovery of 87.3% at 950g/t of reagent, 65% solid conditioning pulp density and pH of 10.

Keywords: Sokoto phosphate rock, beneficiation, deposit, metallurgical grade, AERO 704 (fatty Acid), Flotation, Alkyl Hydroxamates, Melamine.

Introduction

Phosphate mineral is used as a main component in the manufacture of fertilizer that provides phosphorus nutrients required by plants. Phosphorus replenishment of soil is currently efficiently achieved by direct application of phosphorus fertilizer to the soil. Phosphate products are also utilized in manufacture of animal feeds, detergent, and in various industrial processes such as in the control of rust and prevention of corrosion of

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ferrous materials applied with electrochemical conversion coatings. Phosphate mineral is also one of the basic raw materials in the manufacture of phosphoric acid and phosphorus based detergents [1].

The Nigerian demand for phosphate rock is estimated to be about 200,000 tonnes per annum with local production put at 20,000 tonnes per annum [2]. The increasing local demand for phosphate by the user industry especially for fertilizer production due to increased need to boost farm produce coupled with the global economic instability has made it mandatory for Nigeria to strive to develop her phosphate resources.

It is worthy to note that phosphate minerals are not found everywhere in the right concentration needed for optimum plant growth; thus the need to artificially add this mineral to the soil in the form of fertilizer. In view of the strategic importance of fertilizer to food security since no tangible farm produce can be produced without fertilizer and the fact that no known fertilizer grade is currently produced in the country, this makes the research apt. Hence, there is need to beneficiate the Sokoto phosphate rock deposit to a metallurgical grade, so that it can be used as feed for the production of fertilizer and other phosphorus based products.

Materials and methods

Samples

50kg of blended Sokoto Phosphate nodules obtained from Fifty (50) Sokoto Phosphate ore samples randomly collected (ten each) from artisanal miners in Dange-Shuni, Bodinga, Yabo, Wurno, and Rabbah Local Government Areas of Sokoto State were used for the research work.

Equipment/Apparatus

Denver flotation cell, XRF, small size laboratory jaw crusher, cone crusher, pulverizing machine, standard sieve shakers (sieve size fractions: -250+180 μ m, -180+106 μ m, -106+75 μ m, -75+38 μ m), Wifley shaking table.

Reagents

Reagents used during the research work comprised AERO704 (Fatty Acid), sodium carbonate, Reagent S-9849, (an alcoholic solution of alkyl hydroxamic acid from Cytec), Aerofroth 70 (methylisobutylcarbinol - MIBC), Dextrin, Phosphoric Acid and Melamine.

Methodology

Composite sample of Sokoto phosphate rock was subjected to the laboratory separation tests.

Laboratory Separation test

Bench Scale Flotation Tests

Bench Scale flotation tests were carried out on -250+180 μ m, -180+106 μ m, -106+75 μ m, -75+45 μ m and -45+38 μ m particle size fractions after screening in order to determine the optimum flotation feed size distribution using 1 liter Denver flotation cell.

The procedures for carrying out the test are as follows:

Predetermined volume of water was added into the one Liter flotation cell, depending on the conditioning pulp density (55%, 60% and 65% adopted based on findings by Zhang [3]) to be used. 350g of ground phosphate rock was then added and

conditioned for two minutes at desired percent solids (55%, 60% and 65%). Make up water was added and mixed for thirty seconds. Air was then introduced with floated material collected until froth contains clean air bubbles. Collected float materials were dried in an oven and analyzed for P_2O_5 using XRF Pan Analytical Mini PAL4 analyzer.

Results and discussion

Table I gives the percentage of chemical compounds present in the float (concentrate) and tailings products floated using various reagents, pH(s) at different sieve size fractions and flotation conditions. Figures 1 and 2 show % assay of the chemical compounds in the products recovered from the froth flotation processes using Reagent S-9849 Promoter (Alkyl Hydroxamates) and melamine reagents respectively.

From the result, an average of 50%CaO and 20% P_2O_5 , are floated into the float (concentrate), while 10% Fe_2O_3 and less than 10% SiO_2 are floated alongside with the CaO and P_2O_5 into the float. The separation of the chemical compound of the Sokoto phosphate mineral ore into float and tailings given in Table I show that the Sokoto phosphate ore could respond to froth flotation condition processes and hence could be beneficiated using froth flotation mineral processing technique as stated by Weiss [4]. Weiss [4] explained that froth flotation process is the widely adoptable method in the beneficiating of phosphate mineral ores in US.

The average value of 22.6 % P_2O_5 content obtained in the flotation of the Sokoto phosphate rock using a blended sample of scrubbed material is less than the 28.50 to 33.10 % P_2O_5 contents of that obtained in the flotation processes of the US phosphate mineral ores due to non-liberation of the various mineral components arising from its collophane nature. The iron contents obtained were also higher than that of the US. The % CaO contents obtained for the flotation of the Sokoto phosphate is higher when compared to the values obtained for the beneficiation of US phosphate mineral ores using the Crago methods of flotation processes. This could be attributed to the intimate association between the phosphate bearing minerals and the fact that iron and silica have been found to interfere with the chemistry of flotation process of apatite (phosphate) mineral ores [4].

Table 1. Percentage of Chemical Compounds present in the products floated using various reagents, pH(s) at different sieve size fractions and flotation processes.

Sample	Flota. Products	(%) Percent of Chemical Compound present in the products floated													
		HgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	CaO	MgO	TiO ₂	MnO	Fe ₂ O ₃	In ₂ O ₃	BaO	CeO ₂	Nd ₂ O ₃
Calcite Flota. +38µm/HA/pH6	Con.	0.65	2.50	7.18	21.50	0.70	54.28	2.01	0.41	0.84	9.10	0.46	0.10	0.16	0.04
	Tail.	0.64	2.00	7.93	24.4	0.51	54.33	0.06	0.27	0.81	8.18	0.57	0.10	0.14	0.04
Calcite Flota. +38µm/Amine/pH9	Con.	0.70	2.20	5.55	22.10	1.00	55.90	1.95	0.31	0.91	8.70	0.42	0.06	0.19	Nil
	Tail.	0.68	2.20	6.21	23.30	0.53	55.07	1.46	0.29	0.91	8.72	0.35	0.10	0.16	0.03
Calcite Flota. +45µm/HA/pH6	Con.	0.69	2.50	6.82	22.20	1.00	53.45	1.70	0.38	1.05	9.45	0.40	0.09	0.20	0.04
	Tail.	0.54	1.70	6.23	24.00	0.56	54.77	1.65	0.25	1.04	8.69	0.25	0.10	0.16	0.04
Calcite Flota. +45µm/Amine/pH9	Con.	0.74	2.80	7.59	23.30	0.62	53.88	0.08	0.38	0.88	8.71	0.73	0.10	0.15	0.04
	Tail.	0.56	1.90	7.37	24.00	0.49	55.01	1.51	0.23	0.82	7.60	0.25	0.17	Nil	0.08
Phosph. Flota.+38µm/Amine/pH6	Con.	0.67	2.00	5.47	22.80	0.77	54.29	2.39	0.28	1.13	9.40	0.37	0.18	0.18	0.05
	Tail.	0.76	1.90	5.44	22.90	0.61	53.67	2.30	0.31	1.22	10.20	0.30	0.16	0.18	0.04
Phosph. Flota. +45µm/Amine/pH6	Con.	0.58	2.80	7.78	22.50	0.61	54.20	0.03	0.40	0.86	9.11	0.81	0.10	0.16	0.06
	Tail.	0.71	1.90	7.76	23.50	0.65	53.97	1.67	0.29	0.81	8.20	0.30	0.17	Nil	0.06

Table II and Figures 3 and 4 respectively represent the assays of $\%P_2O_5$ and $\%$ recoveries of P_2O_5 into the floats and tailings for the scoping studies on -106+38 μ m size fraction using 800g/t of various reagent types with sodium silicate and aluminum chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9. The results depict that diesel/AERO704 had a better recovery of 60.4% when compared with the recoveries of 55.4% and 48.8% respectively recorded for AERO704 alone and Alkyl Hydroxamates. These values are however lower than the recovery figures recorded when AERO704 (76.2%) and Alkyl Hydroxamates (61.5%) were used without any depressants. The grades of the various fractions collected showed mild differences as they all fell within the less than 25% P_2O_5 category indicating poor separation arising from non-liberation of mineral components due to the very fine grain sizes of the associating mineral assemblage coupled with the high content of iron and calcite which significantly affect phosphate flotation [4].

These results show that using AERO704 collector alone and without any depressant in floating Sokoto phosphate rock will give better recovery results than either Alkyl Hydroxamates or AERO704 combined with Diesel.

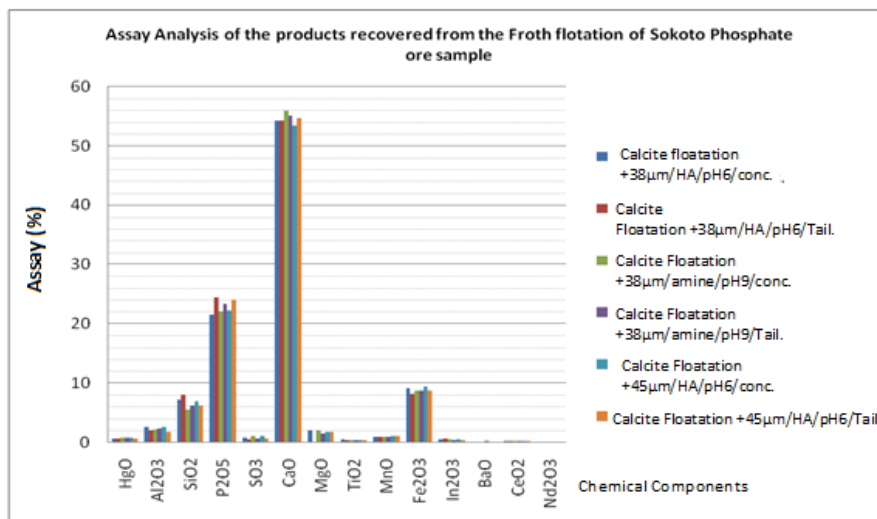


Fig. 1. Assay of the chemical compound in the products recovered from the froth flotation of Sokoto phosphate Rock sample using Alkyl Hydroxamates (HA) and Melamine reagents.

Table II: Results of Scoping Studies on -106+38µm size fraction using 800g/t of various Reagent Types with Sodium Silicate and Aluminum Chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9.

Fraction / AERO	704 S-9849Promoter			Diesel / AERO 704					
Parameter (Fatty Acid)	(Alkyl Hydroxamates)			(Fatty Acid)					
	Wt (g)	% P ₂ O ₅	% Rec.	Wt (g)	% P ₂ O ₅	% Rec.	Wt (g)	% P ₂ O ₅	% Rec.
Float	196.3	20.6	55.4	175.7	20.2	48.4	213.0	20.6	60.4
Tailings	153.7	21.2	44.6	174.3	21.7	51.6	137.0	21.0	39.6
Feed	350.0	20.9	100	350.0	20.9	100	350.0	20.8	100

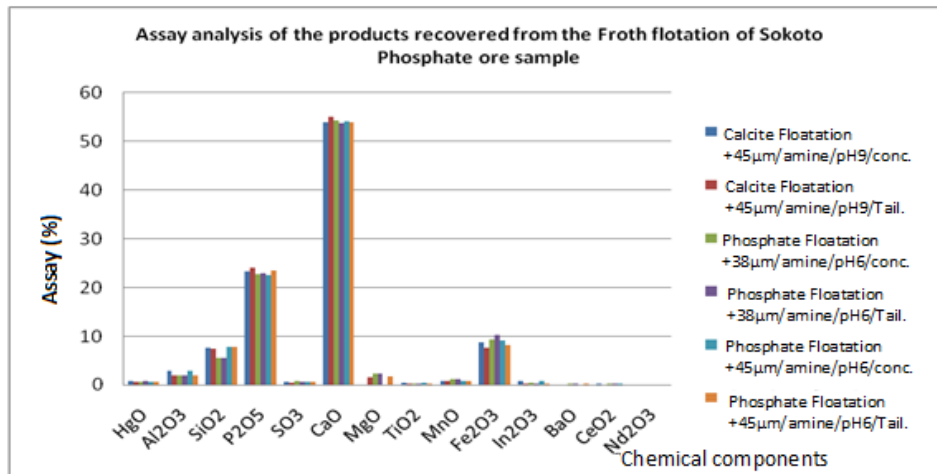


Fig. 2. % assay of the chemical compound in the products recovered from the froth flotation of Sokoto phosphate ore sample using Melamine reagent only.

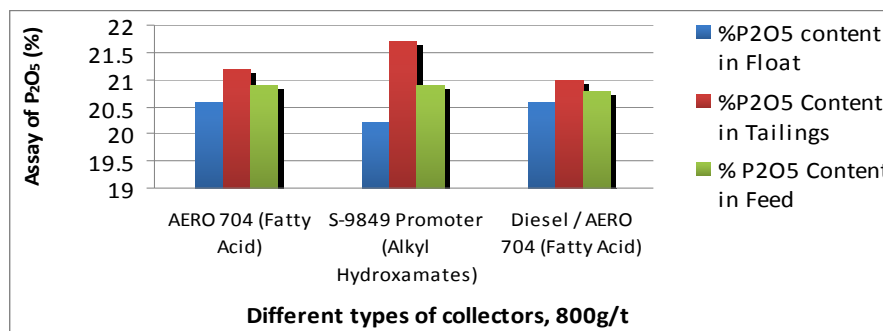


Fig. 3. Assays of %P₂O₅ in floats and tailings for Scoping Studies on -106+38µm size fraction using 800g/t of various reagent types with Sodium Silicate and Aluminum Chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9.

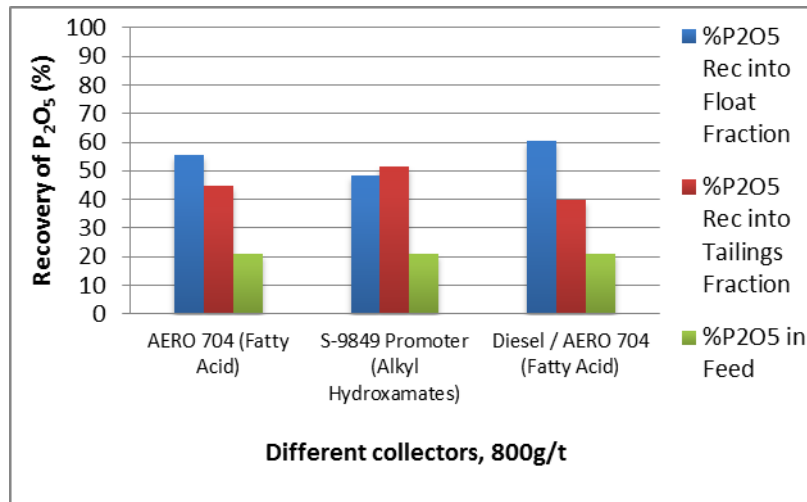


Fig. 4. % Recovery of P_2O_5 into flotation fractions of Scoping Studies on $-106+38\mu m$ size fraction using $800g/t$ of various reagent types with sodium silicate and aluminum chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9.

Table III and Figures 5 and 6 show % assay of P_2O_5 and recovery in the flotation products of $-106+38\mu m$ at 28.5% solids with AERO704 Promoter (Distilled Tall-oil Fatty Acids) and Methyl isobutyl carbinol (MBIC) frother reagents at pH8 and conditioning pulp densities of 55% solids and 65% solids and reagent dosages of 500g/t and 950g/t. From the results, the floats obtained by conditioning at 55% solids with a reagent dosage of 500g/t had an assay of 19.0% P_2O_5 and a recovery of 70.5% which is less than the assay values obtained by conditioning at 65% solids with 500g/t reagent dosage (20.3% P_2O_5 and a recovery of 72.5%). The differences in the values of the % assays of P_2O_5 contents and % recoveries of the floats are significantly very small and this could be attributed to the differences in the percent of solids floated. Where the higher the % of solids floated the higher the tendency for more % of the phosphate bearing minerals to report in the float. Similar reasoning could be advanced for the 55% and 65% solids of the 950g/t. The tailings of the 55% and 65% of the 500g/t and 950g/t have higher contents of % assays of the P_2O_5 and low % recoveries values of the phosphate bearing minerals. This could be attributed to the combined efficiency of the two promoters' reagent that enhances the transferred of much volume of the phosphate bearing minerals to the froth phase as floats (concentrates).

Table III: Flotation Test using -106+38 μm size fraction at 28.5% solids with AERO 704 Promoter (Distilled Tall-oil Fatty Acids) and Methyl isobutyl carbinol (MBIC) frother at pH8

Fractions/ Parameter	pH8											
	500g/t						950 g/t					
	55% solids			65% solids			55% solids			65% solids		
Wt (g)	%	Rec.	Wt (g)	%	Rec.	Wt (g)	%	Rec.	Wt (g)	%	Rec.	
	P ₂ O ₅		P ₂ O ₅			P ₂ O ₅			P ₂ O ₅			
Floats	250.2	19.0	70.5	256.3	20.3	72.5	234.2	20.0	66.0	257.7	20.4	72.7
Tailings	99.8	20.9	29.5	93.7	21.1	27.5	115.8	20.8	34.0	92.3	21.6	28.3
Feed	350.0	19.3	100	350.0	20.5	100	350.0	20.3	100	350.0	20.7	100

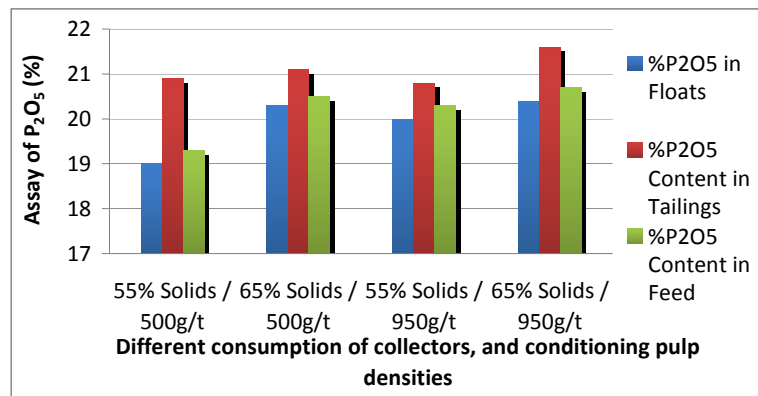


Fig. 5. % Assay of P₂O₅ of the flotation fractions of -106+38 μm at pH8/AERO704 Promoter/MBIC (frother).

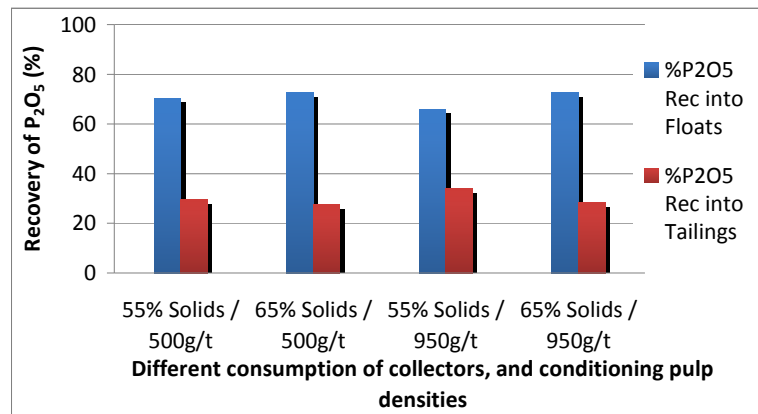


Fig. 6. % Recovery of P₂O₅ of the flotation fractions of -106+38 μm at pH8/AERO704 Promoter/MBIC (frother).

Table IV and Figures 7 and 8 depict % assay of P_2O_5 contents and recovery of the flotation products of -106+38 μ m at pH10/AERO704 Promoter (Distilled Tall-oil Fatty Acids) and Methyl isobutyl carbinol (MBIC) frother reagents. From the results, the floats of 55% solids have an assay of 20.4% P_2O_5 and a recovery of 75.2% more than the values obtained for the floats of 65% solids assaying 18.5% P_2O_5 but with a higher recovery of 78.9% for 500g/t. The differences in the values of the % assays of P_2O_5 contents and % recoveries of the floats are significantly small and this could be attributed to the differences in the percent of solids floated. The higher the % of solids floated the higher the tendency for more % of the phosphate bearing minerals to report in the float. Similar reasoning could be advanced for the 55% and 65% solids of the 950g/t. The tailings of the 55% and 65% of the 500g/t and 950g/t have higher contents of % assays of the P_2O_5 and low % recoveries values of the phosphate bearing minerals. This might be due to the combined efficiency of the two promoters' reagent that enhances the transfer of much volume of the phosphate bearing minerals to the froth phase as floats (concentrates).

Table IV: Flotation Test using 350g of - 106+38 μ m size fraction at 28.5% solids with AERO@704 Promoter (Distilled Tall-oil Fatty Acids) and Methyl isobutyl carbinol (MBIC) frother at pH10

Fractions/ Parameter	pH10											
	500g/t						950 g/t					
	55% solids			65% solids			55% solids			65% solids		
	Wt (g)	% P_2O_5	Rec.	Wt (g)	% P_2O_5	Rec.	Wt (g)	% P_2O_5	Rec.	Wt (g)	% P_2O_5	Rec.
Floats	266.2	20.4	75.2	278.5	18.5	78.9	278.1	19.9	78.3	236.2	20.1	87.3
Tailings	83.8	21.4	24.8	71.5	19.3	21.1	71.9	21.3	21.7	32.5	21.2	12.7
Feed	350.0	20.6	100	350.0	18.6	100	350.0	20.2	100	350.0	15.5	100

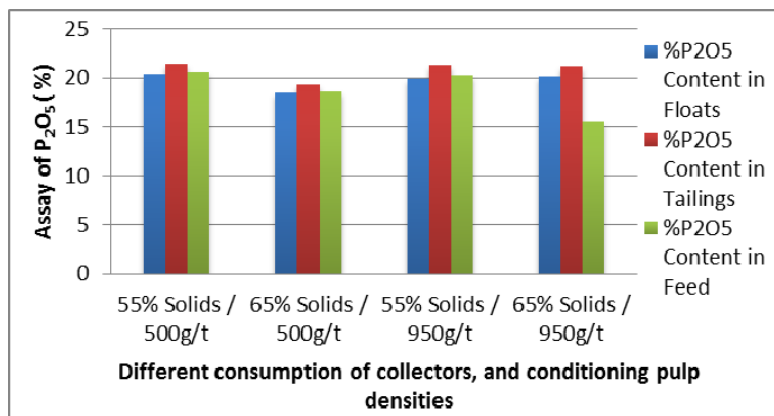


Fig. 7. % Assay of P_2O_5 in the flotation samples of -106+38 μ m at pH10/AERO704 Promoter/MBIC(frother).

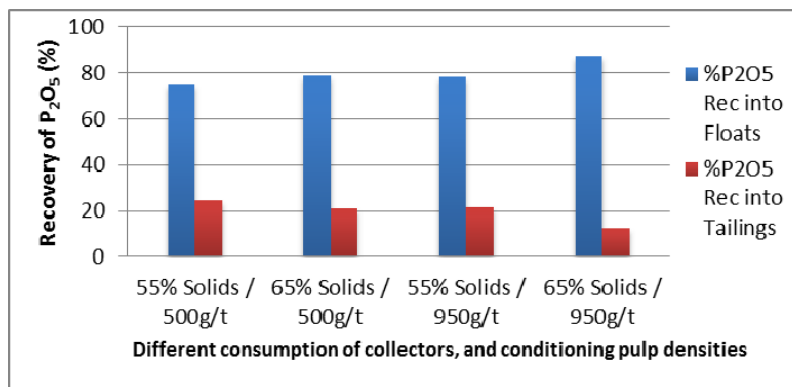


Fig. 8. % Recoveries of P_2O_5 into floats produced during floatation test using 350g of -106+38 μm size fraction at 28.5% solids with AERO704 Promoter (Distilled Tall-oil Fatty Acids) and Methyl isobutyl carbinol (MBIC) frother at pH10.

In comparison with the phosphate floated at pH8 using the same reagents, the average % assay of P_2O_5 obtained is higher compared to those floated at a pH10, but with low % recoveries values. This phenomenon could be explained by the fact that Sokoto phosphate bearing minerals are sensitive to low basicity pH condition and higher above that, their qualities (% assay) reduce while their recoveries increase.

Conclusions

The beneficiation parameters of Sokoto phosphate mineral ore have been determined for the beneficiation of the Sokoto phosphate mineral ore to a metallurgical grade for the production of fertilizer and phosphate related metallurgical products. The conclusions of the study were:

- The separation of the chemical compound of the Sokoto phosphate mineral ore into float and tailings shows that the Sokoto phosphate ore could respond to froth flotation condition processes and hence could be beneficiated using froth flotation mineral processing technique.
- The higher the % of solids floated the higher the tendency for more % of the phosphate bearing minerals to be collected in the float.
- Sokoto Phosphate ore is not amenable to beneficiation by flotation concentration arising from the poor separation of the phosphate bearing minerals from their associating gangue minerals due to poor liberation.
- AERO704 collector alone and without any depressant in floating Sokoto phosphate rock gave the best collector response than either Alkyl Hydroxamates or AERO704 combined with Diesel.

Reference

- [1] Food and Agriculture Organization. Use of Phosphate Rock for Sustainable Agriculture, Pp 54. 2002.
- [2] Raw Materials and Development Council. Technical Brief on Mineral Raw Materials in Nigeria, Revised Edition, ISBN 978-8070-15-9. 2009.
- [3] Zhang P. An Investigation of Flotation Reagents, FIPR 97-02-125, www.fipr.state.fl.us/FIPRreport2003/recently-completed-contracts-beneficiation.htm(Accessed 6/28/2010)
- [4] Weiss, N.L., Mineral Processing Handbook, American Institute of Mining Metallurgical and Petroleum Engineering Incorporated in the United States of America, Kings Port Press, Pp 5/72 – 5/79; 21/1 – 21/17. 1985.
- [5] Carr D. D. Industrial Minerals and Rocks, Society of Mining, Metallurgy and Exploration Inc, Pp751 – 764. 1994.
- [6] Encyclopedia Britanica . www.britanica.com/EBchecked/topic/457399/phosphate-mineral, accessed 28/6/2010
- [7] Fuerstenau C. M. and Han N. K. Principles of Mineral Processing, Society for Mining, Metallurgy, and Exploration, U. S. A., PP245 – 306. 2003.
- [8] Gruber G., Pilot Plant Demonstration of Anionic Rougher – Cleaner Flotation on Florida Phosphate, Final Report 02-151-200, FIPR 01-02-151, www.fipr.state.fl.us/FIPRreport2003/recently-completed-contracts-beneficiation.htm (Accessed 28/6/2010). 2003.
- [9] GSD. Geological Survey Report on Sokoto Phosphate Deposit, Dange- Shuni LGA, unpublished. 1991.
- [10] Gupta and Yan. Mineral Processing Design and Operations, PP555 – 621. 2006.
- [11] Jones, M. P. Applied Mineralogy, A quantitative Approach, pp 13 – 69, 150 -176. 1987.
- [12] Miller J. D. A Selective Collector for Phosphate Flotation, Final Report 02-142-187, FIPR 00-02-142, 2003. www.fipr.state.fl.us/FIPRreport2003/recently-completed-contracts-beneficiation.htm , Accessed - 28/6/2010
- [13] Mular, L., & Bhappu, R. B., SME Mineral Processing Plant Design, 2nd Edition, Pp113 – 306, 447 – 465. 1980.
- [14] Nigerian Mining Corporation. Feasibility Study Report on Sokoto Phosphate Deposit, Dange-Shuni LGA, unpublished. 1991.
- [15] Thomas, D.G., and Adewuyi, E.A. Effects of Local Frothers on the Flotation of Toto MuroIron Ore By. Proceedings of the 35th Confer of the Nigerian Society of Chemical Engineers, Pp116-120. 2005.
- [16] Thompson P. The Selection of Flotation Reagents via Batch Flotation Test. In: SME Mineral Processing Plant Design, Practice, and Control: Proceedings, Vols. 1, Pp137 – 144. 2002.
- [17] Umar, A.H, Characterization and design process flowsheet for the Beneficiation of Sokoto phosphate, Ph.D Thesis, A.B.U., Zaria. 2014.
- [18] Wills, B. A. and Napier – Munn, T. J. Mineral Processing Technology, 7th edition Elsevier Science & Technology Books, PP108-117; 267-352; 378-408) 2006.
- [19] Yaro, S.A., Development of Process Route for the Beneficiation of MallamAyuba Manganese deposits to Ferromanganese feed grade, Ph.DThesis, A.B.U., Zaria. 1997.