CHARACTERIZATION OF SOKOTO PHOSPHATE ROCK AND DESIGN OF PROCESS FLOWSHEET FOR ITS BENEFICIATION

BY

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DECLARATION

I declare that this thesis titled 'Characterization of Sokoto Phosphate Rock and Design of Process Flowsheet for its Beneficiation' was carried out by me in the Department of Metallurgical and Materials Engineering under the supervision of Prof .S.A. Yaro and Dr. M. Abdulwahab. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this thesis was previously presented for another degree or diploma at any university.

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(Signature) **Date**

CERTIFICATION

The thesis titled 'CHARACTERIZATION OF SOKOTO PHOSPHATE ROCK AND DESIGN OF PROCESS FLOWSHEET FOR ITS BENEFICIATION' by Umar Albarka Hassan meet the regulations governing the award of Master of Science Degree of the Ahmadu Bello University, Zaria and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This project is dedicated to my Late Father, Mal. Hassan Albarka, for cherishing me and giving me the right upbringing. May his gentle soul Rest in Perfect Peace, amen.

ACKNOWLEDGEMENT

All praise be to Allah for making it possible for me to complete this work successfully.

My profound and deepest gratitude goes to my supervisor, Prof S.A. Yaro for guiding me and always being there for me despite his other heavy schedules. I will also like to express my gratitude to my minor Supervisor, Dr. M. Abdulwahab for his guidance and support. May the Almighty Allah continue to guide, protect and grant you good health to enable you succeed in all your endeavours.

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Abstract

The characterization of Sokoto phosphate rock and design of process flowsheet for its beneficiation has been investigated and 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.

Chemical analysis of the Run-of-Mine sample gave 44.3%SiO_2, 12.1%P_2O_5, 19.1%CaO, 3.9%Fe_2O_3, and 10.5% Al_2O_3 while the sorted nodules without scalping assayed 14.2%SiO_2, 21.7% P_2O_5, 44.31%CaO, 6.19%Fe_2O_3, and 3.60% Al_2O_3.

Mineralogical analysis of Sokoto Phosphate Rock using XRD showed that the material contained calcite, quartz, hydroxylapatite, flourapatite and carbonate-flouapatiteas the main associating minerals with calcite dominating. Photomicrographs of a thin section of the phosphate nodules showed that associating mineral assemblage exist in very fine association with no visible mineral grains even under the microscope. This resulted in poor separation of associating mineral through flotation due to poor liberation.

Grindability test carried out on the Sokoto phosphate rock using Denver ball milling machine gave a work index of 56.7kwh/ton which is higher than the work indexes of 10.13-13.03kwh/ton for other phosphate mineral ores sighted in the literature. Size-Assay analysis conducted on scrubbed Sokoto Phosphates nodules revealed that nodules had a size distribution with 80% passing 29300µm. A density test revealed that Sokoto Phosphate nodules have a specific gravity of 2.9.

Flotation Tests using AERO 704 (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 P_2O_5 separation in the flotation products due to very poor liberation associated with very fine mineral grains.

AERO 704 Collector gave the best result with a P_2O_5 recovery of 87.3% at 950g/t of reagent, 65% solid conditioning pulp density and pH of 10.

Based on the results obtained from all the tests and analysis carried out on the ore, a process flowsheet was developed comprising a Scrubber fitted with a Screen Mesh of 12.7mm apertures to upgrade the ROM from 12.1% P_2O_5 to 24.5% P_2O_5 with a P_2O_5 recovery of 72% that will be milled to 80% passing 75µm thus producing a product with high enough P_2O_5 content that can be used for fertilizer production.

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ABBREVIATIONS

%	-	Percentage
µm	-	Micron (10^{-3})
Al_2O_3	-	Aluminium Oxide
BaO	-	Barium Oxide
Cao	-	Calcium Oxide
EDS	-	Energy Dispersion Spectrometer
Fe_2O_3	-	Iron Oxide
g/t	-	Gramme per tonne
Kg	-	Kilogram
Km	-	Kilometer
M	-	Mass
MBIC	-	Methylisobutylcarbinol
m^3	-	Cubic Meter
mm	-	millimeter
Mn	-	Manganese
N.D	-	Not Detected
P_2O_5	-	PhosophorousPentoxide
SEM	-	Scanning Electron Microscope
SiO_2	-	Silicon Oxide
TiO_2	-	Titanium Oxide
V	-	Volume
XRD	-	X-ray Diffraction
XRF	-	X-ray Fluorescence Spectrometer

Chapter One

1.0 Introduction

Mineral Phosphates are complex mineral formations which are the basic forms of the primary source of phosphorus, an element necessary to all forms of plants, animals and human life. Phosphorus finds many applications in life. With the urgent need for intensive farming due to increasing population, the fertility of the soil needs to be maintained by its external additions.

Nigeria has been importing fertilizers and other phosphorus related products for farming as well as to sustain her agricultural programme. This has led to the importation of millions of metric tons of fertilizer amounting to millions of US-dollars from the United States of America and other countries within the African continent like Tunisia, Senegal, Morocco and Togo despite the large deposits of phosphate mineral ores available in the country.

In the United States, Florida is the source of about 73% of the phosphate rock produced, North Carolina accounts for 15% while Western phosphate and Tennessee accounts for 12% of the total production of phosphate mineral ores. The matrix of the ores mineralogy are predominantly flouapatite orfrancolite (sometimes called collophane), in which an excess of fluorine and carbon replaces phosphate in the apatite lattice. They are marine sedimentary deposits and rocks and sometime, oolitic in nature. Phosphate contents of the deposits are classified into two: low grade ranging between 14.6 to 29% P_2O_5 and high grades from 30% P_2O_5 and above. The lime-to-phosphate (CaO/P_2O_5) ratio of the deposits

is within 1.59 to 2.1 in some cases, with low iron and alumina. Other minerals in association are silica, magnesium, chloride, lithium, feldspar and fluorite (Weiss, 1985).

Sokoto phosphate rock contains phosphatic nodules with an average grade of 22.5% P_2O_5 disseminated in clay matrix (Nigerian Mining Corporation, 1991). This P_2O_5 content thus makes the phosphate rock low grade ore requiring beneficiation before use for the purposes of fertilizer production by wet process which requires a phosphate rock with about 36% P_2O_5.

Hence, the need to beneficiate the Sokotophosphate rock deposit to a metallurgical grade that can be used as feed for the production of fertilizer and other phosphorus based products. This desire prompted the research on "the design of process flow-sheet for the beneficiation of Sokoto phosphate rock deposit", for possible utilization in the production of fertilizer and other phosphorus based products for metallurgical usage.

1.1 Location of Sokoto Phosphate Deposit

Sokoto phosphate rock is found in several locations comprising Bonganange, Dan Kilo, (Dange-Shuni LGA), Danchadi (Bodinga LGA), GidanDaji (Yabo LGA), Wurno (Wurno LGA), Rabbah (Rabbah LGA) amongst others. The deposit is located in the Sokoto basin which is bounded within latitudes 12^o 45' and 13^o45'N and longitudes 5^o00' and 6^o00E. The deposit has a phosphate content in excess of 300million tonnes (Carr, 1994).

1.2 Uses of Phosphate Rock

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 ferrous materials applied with electrochemical conversion coatings.

The national demand for phosphate rock is estimated to be about 200,000 tonnes per annum with local production put at 20,000 tonnes per annum (Raw Material Research and Development Council, 2009). 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.

1.3 Statement of Research Problem

Mineral deposits are known to have distinct characteristics regarding mineral composition and texture thereby necessitating the development of process route that will adequately handle their peculiarities.

Sokoto phosphate rock contains phosphatic nodules with an average grade of 22.5% P_2O_5 disseminated in clay matrix (NMC, 1991). This P_2O_5 content thus makes the phosphate

rock low grade ore requiring beneficiation before use for the purposes of fertilizer production by wet process which requires a phosphate rock with about 36%P_2O_5.

1.4 **Justification**

Phosphate mineral is an essential micronutrient for plants supplied naturally by weathering of rocks and artificially from fertilizers produced using mined phosphate rock. It is also one of the basic raw materials in the manufacture of phosphoric acid and phosphorus based detergents.

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 makes this proposal apt.

The successful completion of this research will:

a) Pave way for the production of fertilizer grade phosphate concentrate from Sokoto phosphate deposit.
b) Save foreign exchange that will otherwise be used in importing the fertilizer grade concentrate needed bylocal fertilizer producers.

c) Information and data obtained on the characteristics and the beneficiation of the Sokoto phosphate will be used to design the process flowsheet for upgrading of the ore to fertilizer grade

d) Appropriate technology for the beneficiation of the Sokoto phosphate will be developed and through this local engineers and technologists will acquire the necessary skills for the processing of the Sokoto phosphate mineral ore.

1.5 **Aims and Objectives of the Study**

The purpose of the study includes:

a. Determining the chemical and mineralogical composition of the phosphate rock sample;
b. Determining the work index and grinding characteristics of the Sokoto phosphate rock; and
c. Proposing a process flowsheet for beneficiating Sokoto Phosphate ore to fertilizer grade

1.6 **Scope of work**

The study would involve field work that will facilitate the collection of representative sample needed for the study as well as bench scale tests and analysis on the sample obtained from the field.

The breakdown of the scope of work is as follows:

a) Collection of representative sample of the phosphate rock by blending samples collected from phosphate rock stockpiles of phosphate processing plants located in and around Sokoto through coning and quartering of samples collected by random sampling.

b) Carrying out mineralogical characterization of the phosphate rock sample collected from the phosphate stockpiles produced by artisanal miners from Sokoto phosphate deposit using X-ray Diffraction Analysis (XRD), chemical analysis using X-ray Fluorescence analysis (XRF) with the quality of products produced during test work monitored by XRF to determine the $\%P_2O_5$ content of the various products produced during the course of the study.

c) Carrying out the size – assay analysis of the blended Sokoto phosphate rock.

d) Determining the work index of the phosphate rock using Berry and Bruce method and grinding characteristics using size- assay analysis on ground sample.

e) Carrying out flotation studies for the purpose of determining suitable reagents needed to float the phosphate rock, dosage requirement, point of addition(s), number of flotation stages as well as economic grind size.

f) Collation of results, production of necessary tables (metallurgical balance, etc) / graphs (grade- recovery curves, etc), analysis of results and final design.

1.7 Contribution to knowledge

The successful completion of this research work resulted in a process study of Sokoto phosphate mineral ore. The proposed study, carried out:

a) Availed the nation and the world over with the chemical and mineralogical composition of the Sokoto Phosphate mineral ore;
b) Revealed the comminution characteristics of the phosphate mineral ore;
c) Revealed the potential of floating phosphate bearing minerals using fatty acid without any addition of fuel oil; and
d) Produced a suitable process design flow-sheet for the beneficiation of Sokoto phosphate deposit to produce fertilizer grade concentrate.

Chapter Two

2.0 Literature Review

2.1 *Introduction*

Minerals are homogeneous, solid substances that are generally of inorganic origin having well-defined crystalline structure and a chemical composition that lies between well-defined limits (Jones (1987). However, due to the fact that not all materials that are classified as minerals are of inorganic origin or having fixed chemical composition amongst others, the term mineral is generally referred to as substances of economic value extracted from the earth (Wills, B. A. and Napier – Munn, T. J. (2006)). In nature, minerals of interest exist physically and chemically combined with the host rock.

Minerals are generally classified as:

i. Mineral oils and gases which refer to the hydrocarbons that occur as crude oil and natural gas;
ii. Metallic minerals from which metals are extracted, e.g. iron, copper, nickel, etc;
iii. Mineral fuels that refer to coal, lignite, etc.
iv. Non-metallic or industrial minerals made up of minerals that are used in bulk for their physical, chemical or other properties. E.g. Limestone, Clay, Gypsum, barites, feldspar, etc.
v. Precious minerals and

vi. Radioactive minerals.

2.2 *Source of minerals*

Almost all minerals are obtained directly from the earth's crust with a small proportion obtained from secondary sources such as scraps. The average crustal composition shows that the elements oxygen, silicon, aluminum and iron account for nearly 90% of the total, while the economically important elements like copper, zinc, tin, lead and gold occur only in minute proportions (Jones, 1987). The rocks of the earth are however not of uniform composition. Where the minerals are potentially of economic value, these natural concentrations are called *mineral deposits* and it is from these quite rare crustal features that minerals of commerce are obtained. Any mineral deposit from which it is possible to produce metallic products is called an *ore*.

2.3 *Ore processing*

Mineral deposits have continued to drop in mineral content as well as being of more complex mixture of worthless and valuable minerals with the mixture having no immediate or readily apparent value. This complex mixture of very low mineral values therefore needs to be upgraded to make it salable. The process through which this complex mixture undergoes in order to make it salable is termed mineral processing.

Mineral Processing in its broadest sense includes both the physical and chemical separation (hydrometallurgy and Pyrometallurgy) of valuable constituents from the

crude ore (or other raw material). It is the removal of unwanted minerals (gangue) from an association of valuable and non-valuable minerals resulting in the increase in concentration of the valuable mineral in an economically viable manner. This upgrading is usually done using differences in the properties of the associating minerals.

Mineral processing is usually carried out at the mine site, the plant being referred to as a mill.

The purposes of mineral processing are amongst others the following:

i. To separate valuable from gangue minerals thereby reducing the bulk of the ore which must be transported to and smelted by the smelter, by using cheap, low – energy physical methods to separate the valuable minerals from the waste minerals (gangue). This enrichment process greatly increases the contained value of the ore to allow economic transportation and smelting.

ii. To reduce smelter metal losses due to the production of less metal bearing slag.

iii. To allow for the production of high grade metal products free from deleterious impurities which are removed into the discard (tailings) thereby minimizing penalty charges usually imposed by smelters according to the level of deleterious impurities contained in the concentrate. E.g. removing

arsenopyrite from tin concentrate ensures that high grade tin metal is produced as it is difficult to remove the contained arsenic in tin smelting.

iv. To produce usable Products by separating valuable minerals from each other thereby ensuring that products of required specifications are produced.

v. To effect the removal of environmental harmful products from discards to acceptable levels before discharge to streams and the atmosphere.

vi. To produce products that meet the demands of new technology.

The greatest challenge to a mineral process Engineer is to produce high grade concentrates consistently at maximum recovery from the ore body. To quantify recovery, a reasonable idea of the initial concentration of the mineral in the deposit being treated is required which can only be done using a representative sample. Proper sampling representing the ore body is therefore essential.

Losses to tailings are one of the most important factors in deciding whether a deposit is viable or not. Losses will depend on the ore mineralogy and dissemination as well as on the technology available to achieve efficient concentration.

Mineral processing operations are often a compromise between improvements in metallurgical efficiency and milling costs. This is particularly true for ores of low contained value where low milling costs are essential and cheap unit operations are necessary, most especially in early stages where the volume of material treated is relatively high. With such low-value ores, improvements in metallurgical efficiency by the use of

more expensive methods or reagents cannot always be justified (Wills, B. A. and Napier – Munn, T. J. (2006)).

Conversely high metallurgical efficiency is usually of most importance with ores of high contained value and expensive high-efficiency processes can often be justified on these ores. Other costs that need to be taken into account apart from processing costs and losses are ancillary services such as power supply, water, roads, tailings disposal that depend on the size and location of the deposit as well as taxes, royalty payments, investment requirements, research and development, medical and safety costs amongst others.

2.4 ***Mineral Processing Unit Operations***

Mineral processing unit operations that are mostly used in the course of beneficiating mineral deposits are as outlined below.

2.4.1 *Sampling and Analysis*

(a) Sampling

This refers to the collection of a fraction of given material in such a way that the fraction so collected is representative of the lot.

The essential requirements of a good accounting and control system are:

i. Efficient and representative sampling of process streams, upon which accurate analysis of the value components can be undertaken; and
ii. Reliable and accurate measurements of the mass flow rate of important flow streams.

The minimum weight of sample to be collected varies with size of the particles being sampled. This is generally estimated using the expression:

$y=0.2116x^2$ *(2.1)*

for low and uniform ores as devised by Targart (1927) and converted to metric system where x is the particle top size in mm and y is the sample weight in kg.

The other formula that could be used to estimate sample weight is that devised by Gy to calculate the size of sample necessary to give the required degree of accuracy and which is mostly used for streams transported on conveyors or pulp streams rather than heap deposits (Wills, B. A. and Napier – Munn, T. J. (2006)). It is expressed as:

$M = Cd^3/S^2$ *(2.2)*

where M is the minimum weight of sample required (g), C is the sampling constant of the material, d is the dimension of the largest piece in the material to be sampled (cm) and s is the measure of the statistical error.Common sampling points include ore bodies, trucks, conveyor belts, storage piles, flow streams, etc (Wills, B. A. and Napier – Munn, T. J. (2006)).

The various sampling techniques that can be used include random sampling, cone and quartering, riffling, etc.

(b) Analysis

Analysis refers to separating or breaking up of any whole into its parts, especially with an examination of these parts to find out their nature, proportion, function, interrelationship, etc. Chemically, it would refer to the separation of a substance into its constituent elements to determine either their nature (qualitative analysis) or their proportions (quantitative analysis) [www.yourdictionary.com, 2010].

Metallurgically, it would refer to the analysis of materials used in metallurgical processes which generally comprise the use of instrumental techniques or classical methods to determine the materials' chemical, mineralogical or phase compositions (Yaro, 1998). While the classical analytical methods rely on chemical reactions to perform an analysis, the instrumental methods generally depend on the measurement of physical property of an analyte such as light absorption, fluorescence or conductivity using an apparatus which is then compared with the values of a reference sample to obtain a figure for the physical property so measured. Examples of instrumental methods include the Atomic Absorption Spectrometry (AAS) and X-ray Diffraction (XRD) amongst others.

2.4.2 *Scrubbing*

Scrubbing refers to the disintegration of material by forces which are relatively light, judged by ordinary standards of comminution but are sufficient to breakdown the soft unconsolidated portion, mostly clay. Scrubbing is usually effected by rubbing the larger and harder grains together, as tumbling the mass of the crude backed by a rigid surface is usually sufficient. The problem that is normally encountered with this process is the

formation of clay balls which are highly resistant to disintegration. To solve this problem, the scrubber is usually run with a low pulp density. Preventing the ROM from coming in contact with moisture/water before treatment can also solve this problem.

2.4.3 *Sizing*

Sizing refers to the separation of material based on differences in particle size. It is made up of screening (sizing using screening surfaces) and classification (sizing on the basis of the velocity with which the mineral grains fall through a fluid medium, usually water or air).

Screening is industrial sizing that is used for size separations from 300mm down to about 40 microns. Screening can be carried out either wet or dry with dry screening limited to material above about 5mm in size while wet screening is generally carried out to around 250microns. Special type of screens, like circular or gyratory screens are however being used for fine screening applications, wet or dry, down to 40 microns.

The main purposes of sizing in mineral processing include:

a) Separation of particles by size to provide a downstream unit process with the particle size range suited to that unit operation;
b) To remove the coarsest size fractions in the feed material for crushing or removal from the process;
c) Production of a number of products within specified size ranges;

d) Media recovery especially in washing magnetic media from ore in dense media separation;
e) Draining of free moisture from wet sand slurry;
f) Removal of fine material from a wet or dry feed;
g) Removal of trash such as wood fiber from a fine slurry stream.

Examples of sizing equipment include vibrating screens, grizzleys, revolving screens (trommel), etc for screens and hydrosizer, settling cones, rake and spiral classifiers, hydocyclones etc for classifiers.

2.4.4 *Size reduction and liberation*

Size reduction is the gradual reduction in size of the run-of-mine (ROM) by comminution methods of crushing and grinding to the required size specification while Liberation refers to the production of clean particles of minerals and gangue using comminution methods. Comminution in its earliest stages is carried out in order to make the freshly excavated material easier to handle by scrapers, conveyors and trucks, and in the case of quarry products to produce material of controlled particle size. The aim of comminution is to maximize the liberation of the mineral from the host rock.

Blasting which remove ores from their natural beds in mining through the use of explosives is generally considered the first stage of comminution. Comminution in a mineral processing plant takes place as a sequence of crushing and grinding processes.

Crushing reduces the particle size of run-of mine ore to such a level that grinding can be carried out until the mineral and gangues are substantially produced as separate particles. It is accomplished by compression of the ore against rigid surfaces or by impact against surfaces in a rigidly constrained motion path. This is opposed to grinding which is accomplished by abrasion and impact of the ore by free motion of unconnected media such as rods, balls or pebbles.

Crushing is usually carried out dry using crushers such as Jaw, gyratory, cone and impact crushers amongst others and is performed in several stages with small reduction ratios (ratio of 80% passing feed size to 80% passing product size) ranging from three to six in each stage.

Grinding is mostly carried out wet to provide slurry feed to the concentration processes with dry grinding carried to produce finished products especially in industrial mineral processing like calcium carbonate, feldspar, kaolin, etc. Tumbling mills with either steel balls or rods, or sized ore as grinding media are generally used in the last stages of comminution. Other mills that are also used in mineral processing for grinding purposes include stirred mills, High Pressure Grinding Rolls (HPGR), etc.

The relationship between energy input and the particle size made from a given feed size are given by various theories of comminution with none being satisfactory (Wills et al, 2006). The reasons for this are:

i. Most of the energy input to a crushing or grinding machine is absorbed by the machine itself with only a small fraction of the total energy actually

used in breaking the material. In a ball mill for example, less 1% of total energy input is available for actual size reduction, the bulk of the energy being utilized in the production of heat; and

ii. A material that is plastic will consume energy in changing shape, a shape that it will retain without creating significant new surface. All the theories of comminution assume that the material is brittle, so that no energy is adsorbed in processes such as elongation or contraction which is not finally utilized in breakage.

The main comminution theories are as outlined below.

a) Von Rittinger's theory of comminution

This theory states that the energy consumed in size reduction is proportional to the area of new surface produced. The surface area of a known weight of particles of uniform diameter is inversely proportional to the diameter, thus equating Rittinger's law to

$$E = K\,(1/D_2 - 1/D_1) \qquad (2.3)$$

where E is the energy input, D_1 is the initial particle size, D_2 is the final particle size, and K is a constant.

b) Kick's theory of comminution

This theory states that the work required is proportional to the reduction in volume of the particles concerned. The theory is expressed as

$$R = f/p \qquad (2.4)$$

Where f is the diameter of the feed particles and p is the diameter of the product particles.

According to this law, E α log(R)/log (2) where E is the energy required for comminution.

c) Bond's theory of comminution

This theory states that work input is proportional to the new crack tip length produced in the particle breakage and which equals the work represented by the product minus that represented by the feed. Bond's theory is mathematically expressed as

$$W = 10W_i\,[1/\sqrt{P} - 1/\sqrt{F}] \qquad (2.5)$$

where W is the work input in kilowatt hours per short ton, W_i is the work index which is the resistance of material to crushing and grinding expressed as the kilowatt hours per short ton required to reduce the material from theoretically infinite size to 80% passing 100μm. The work index is the widely used measuring parameter for hardness and grindability of ores (Yaro, 1998). Work index of selected minerals are given in table 2.1 below.

Kick's law is reasonably accurate in the crushing range above about 10000μm in diameter; Bond's theory applies reasonably in the range of conventional rod-mill and ball-mill grinding (feed of 5 – 250mm and reduced to 40 -300 μm), and Rittinger's law applies fairly well in the fine grinding range of 10 - 10000μm (Wills et al, 2006).

Table 2.1: Average work indexes of some minerals

[Sources: Weiss (1985), Society for Mining, Met and Exploration, Inc. 2003; Yaro and Thomas (2009)]

Minerals	**Specific gravity**	**Work index(kwh/tonne)**
Manganese ore	3.74	12.46
Hematite specular	3.29	15.40
Hematite	3.76	12.68
Magnetite	3.88	10.21
Itapke iron ore	-	9.34
Magnesite burned	5.22	16.80
Coal	1.63	11.37
Copper ores	3.02	13.3 and 18.3-23.38
Gravel	2.70	25.17
Granite	2.68	15.14
Pyrite ore	3.48	8.90
Phosphate mineral ores :Fertilizer	2.65	13.03
Rock	2.66	10.13

2.4.5 *Mineral Separation / Concentration Processes*

Concentration refers to the separation of valuable minerals from the gangue producing in the process fractions that contain majority of particular minerals e.g. a fraction containing predominantly valuable minerals known as the concentrate or tailings comprising predominantly the non-valuable minerals.

An intimate knowledge of the mineralogical assembly of the ore is essential if efficient processing is to be carried out. Such knowledge should include nature of valuable and non-valuable minerals, size, dissemination, association and shape of the minerals within the ore. Separation of minerals into various fractions should always be considered in the context of the mineralogy of the ore in order to predict grinding and concentration requirements, feasible concentrate grades and potential difficulties of separation (Wills et al, 2006). Microscopic analysis of concentrate and tailings products can also yield very valuable information regarding the efficiency of the liberation and concentration processes (Wills et al., 2006). This practice is mostly useful in troubleshooting problems which arise from inadequate liberation where conventional optical microscopes are used to examine thin and polished sections of mineral samples or using mineral liberation analyser (MLA) and Quantitative Evaluation of Materials by Scanning (QEMSCAN).

Physical separation methods used in producing mineral concentrates are as outlined below.

2.4.5. (a) Sorting

This is a concentration method that is based on optical and other properties. This technique of mineral concentration used to be carried out by hand but it is now mostly accomplished by machine. Ore sorting involves the appraisal of individual ore particles and the rejection of those ore particles that do not require further treatment.

Hand treatment has declined in importance due to the need to treat large tonnages of low grade ore which require extremely fine grinding. Hand sorting that is generally practiced at some mines now includes removal of large pieces of timber, tramp iron, etc from the run-of-mine ore.

Machine sorting is generally carried out using electronic ore sorting equipment where it is applied to:

i. Pre-concentration in which barren waste is eliminated to reduce the tonnage reporting to the downstream concentration processes, such as in uranium or gold ore sorting, or
ii. Production of a final product such in limestone or diamond sorting.

The ore must be sufficiently liberated at a coarse size (greater than 5-10mm) to allow for the discard of barren waste without significant lost of value (Wills et al., 2006). Pre-concentration by sorting is seen as a method of improving the sustainability of mineral processing operations by reducing the consumption of energy and water in grinding and

concentration as well as achieving more acceptable tailings disposal (Cutmore and Ebehardt, 2002).

Rock properties that are normally used as basis of electronic sorting include reflectance and colour in visible light (magnesite, limestone, base metal and gold ores, phosphate, talc, coal, etc), ultraviolet (scheelite), natural gamma radiation (uranium ore), magnetism (iron ore), conductivity (sulphides) and x-ray luminescence (diamond), etc.

Electronic sorters inspect the particles to determine the value of some property e.g. light reflectance, and then eject those particles which meet some criterion such as light versus dark rocks. Either valuables or waste may be selected for rejection. It is essential that a distinct difference in the required physical property is apparent between the valuable mineral and the gangue.

The particle surfaces must be thoroughly washed before sorting so that any adhering slimes that could cause signal blurring are removed as well as ensuring that closed sized feed are treated at a time in a particular machine. The ore must be fed in a mono layer as display of individual particles to the sorting device must be effected. Examples of electronic sorters include the photometric sorters (gold ores, magnesite, feldspar, limestone, etc), x-ray sorters (diamonds), radiometric sorters that uses scintillation counters to detect gamma radiation, etc.

2.4.5. (b) Gravity separation

This is a separation method that is based on differences in density between minerals. Gravity concentration methods separate minerals of different specific gravity by their relative movement in response to gravity and resistance to motion offered by a viscous fluid, such as water or air.

Gravity separation methods are used to treat a variety of minerals ranging from heavy metal sulphides such as galena (s.g. 7.5) to coal (s.g. 1.3), at particles sizes that can be as low as 50μm. They are extensively used for treating tin ores, coal and many industrial minerals.

At fine particle sizes, separation by density difference becomes less efficient. At this size range flotation is the dominant separation process, though the application of centrifugal acceleration has extended the useful separation size by gravity processes down to 5-10μm provided the concentration criterion is favourable.

Gravity concentration methods are most often adopted as the concentration method over others especially froth flotation due to:

i. Lower installed cost per ton of throughput than flotation;
ii. Lower installed power requirement per ton of throughput;
iii. The fact that gravity separation does not use expensive reagents; and
iv. Lower environmental impact of gravity plant effluent when compared to flotation due to the absence of organic chemicals.

Gravity concentration methods are mainly used to produce pre-concentrates resulting in energy and reagent costs savings when the more expensive methods are restricted to the processing of gravity concentrate. Gravity separation of minerals at coarser sizes as soon as liberation is achieved can also have significant advantages for later treatment stages due to decreased surface area, more efficient dewatering, and the absence of adhering chemicals which could interfere with further processing.

It is essential for effective separation that a marked density difference exists between the mineral and the gangue. The concentration criterion, expressed as

$$(D_h - D_f) / (D_l - D_f) \qquad (2.6)$$

where D_h is the specific gravity of the heavy mineral, D_l is the specific gravity of the light mineral, D_f is the specific gravity of the fluid medium gives some idea of the type of separation possible. In general, when the quotient is greater than 2.5, whether positive or negative, the separation tends to be relatively easy. The efficiency of separation tends to decrease with decrease in the value of the quotient.

Examples of gravity separation equipments include jigs, pinched sluices and cones, spiral concentrators, shaking tables, centrifugal concentrators (multi-gravity separators, Knelson concentrators, Falcon concentrators, etc), dense media separators (Wemco cone separator, Drum separator, Drewboy bath, cyclone dense medium separator – Dyna whirlpool separator, Tri-Flo separator, etc) amongst others.

2.4.5.(c) Froth Flotation

This is a both physical and chemical process of concentrating finely ground ores that depends on the selective adhesion to air of specific minerals while others remain submerged in the pulp. This process involves treatment with suitable reagents of an ore pulp to create condition favorable for the attachment of certain hydrophobic or aerophilic mineral particles to air bubbles and render the other solid hydrophilic or water loving submerged in the solution.

The aim bubbles carry the selected mineral to the surface of the pulp and form a stabilized froth, which is skimmed off while the other minerals remain submerged in the pulp (Willsand Napier – Munn, 2006).

The importance of flotation in modern mineral processing practice results from a number of factors. Which include some of the following reasons? It is necessary for mining and treating ores, in which the minerals are finely disseminated, and require finer grinding to liberate the valuable minerals. The resulting slimes from the fine grinding are difficult to handle by gravity methods but not a problem in flotation. In many complex ores it is desirable to separate concentrate to facilitate subsequent treatment. Galena (PbS), sphelerite (ZnS), pyrite (FeS) and other mineral separations are desired, but the spread of density between these minerals is so small that gravity method is unsatisfactory. Differential flotation has solved many of the milling problems concerted with complex ones.

Flotation concentration can be used successfully over a wide range of particle sizes and flotation feed does not ordinarily required elaborate preliminary treatment (screening, hydraulic classification etc). This together with the fact that flotation machines required less floor space and head room than gravity machines has made it possible to build smaller, simpler and more efficient mill (Willsand Napier – Munn, 2006)

Feeding range between 60mesh (250μm) and a few microns are practicable with flotation. Froth flotation is a proposal for production of super concentrate of Itakpe Iron ore to feed Aladja and Ajaokuta steel plants in Nigeria. The valuable mineral is transferred to the froth learning the gangue in the aqueous medium. This is known as direct flotation while in some cases however; the gangue is transferred to the froth fraction and is known as reserve flotation (Yaro, 1997).

The success of froth flotation operation lies in the correct selection of the reagents to be added to the aqueous medium. Some of these reagents affect processes in the aqueous phase while other affects them in the froth phase (Thomas and Adewuyi, 2005).

Furthermore, flotation, according to Gupta and Yan (2006), has found prominence because of the need to treat complex or low grade ores where the average particle size for liberation is too small for efficient gravity separation or where the gravity difference between minerals is too small. Flotation, being a selective process, can be used to obtain specific separations from complex ores such as lead-zinc, copper-zinc, lead-zinc-tin, etc. Areas of

application for flotation as a concentration method has been widened from the initial treatment of sulphide ores of copper, zinc and lead to platinum, nickel and gold-hosting sulphides as well as oxides (hematite, cassiterite, etc), oxidized minerals (malachite, cerussite, etc) and non-metallic ores (fluorite, phosphates and fine coal amongst others).

(i) *Flotation Mechanism*

The essential mechanism of flotation involves the attachment of mineral particles to air bubbles in such a way that the particles are carried to the surface of the pulp where they can be removed. The process of recovering a mineral by flotation from the pulp comprises the following steps:

a. Wet grinding of the ore to a size sufficiently fine enough to liberate the valuable minerals from one another and from the gangue minerals;
b. Creating conditions favourable for the adherence of the desired minerals to air bubbles;
c. Creating a rising current of air bubbles in the pulp;
d. Forming a stable mineral laden froth on the surface of the pulp; and
e. Removal of the mineral laden froth.

Although grinding of the ore is not a part of flotation, it does have an important bearing on the process. For optimum flotation results, the valuable minerals should be separated completely from the gangue and from one another in the grinding step (Abouzeid, 1990). This is often not economically feasible due to the problems caused by ultra fines produced

during grinding which tend to complicate subsequent processes. Grinding also creates new surfaces for better surface conditioning.

The mechanism of transferring a mineral particle from the pulp to the surface by flotation takes place according to Willsand Napier – Munn (2006) as follows:

a. Selective attachment to air bubbles (true flotation);
b. Entrainment in the water which passes through the froth; and
c. Physical entrapment between particles in the froth attached to air bubbles.

The attachment of desired mineral particles to air bubbles is the most important mechanism and represents the majority of particles that are recovered to the floated fraction. Although true flotation is the dominant mechanism for the recovery of desired mineral, the separation efficiency between the valuable mineral and the gangue also depends on the degree of entrainment and physical entrapment. Unlike true flotation, which is chemically selective to the mineral surface properties, both gangue and valuable minerals alike can be recovered by entrapment and entrainment. Drainage of these minerals occurs in the froth phase thus making the control of the stability of this phase very important to achieve an adequate separation. The problem of entrainment in industrial flotation plant practice is solved by using several stages of flotation (circuits).

Flotation is applied to relatively fine particles, because if they are too large the adhesion between the particle and the bubble will be less than the particle weight and the bubble will

therefore drop its load. It is usually carried out using an upper size limit of around 300μm and a lower size limit of about 5μm at which problems of oxidation and other surface effects occur (Willsand Napier – Munn, 2006).

Froth flotation is generally classified as direct flotation when the valuable mineral is transferred to the froth leaving the gangue in the pulp (tailing) or reverse flotation when the gangue is transferred to the froth leaving the valuable minerals in the pulp with the material (valuable mineral or gangue) less in volume normally floated.

The function of the froth phase is to enhance the overall selectivity of the flotation process. The froth achieves this by reducing the recovery of entrained material to the concentrate stream while preferentially retaining the attached material. This increases the concentration grade whilst limiting as far as possible the reduction in the recovery of the valuables. The relationship between recovery and grade is a trade-off that needs to be managed according to operational constraints and is incorporated in the management of optimum froth stability.

The creation of a rising current of air bubbles is created by a flotation machine which produces bubbles by the mechanical agitation of the ore pulp, the direct introduction of air under pressure or both.

The mineral particles can only attach to the air bubbles if they are to some extent water-repellent, or hydrophobic. When they reach the surface, the air bubbles can only continue

to support the mineral particles if they can form stable froth, otherwise they will burst and drop the mineral particles. To achieve these conditions numerous chemical compounds known as flotation reagents are used (Willsand Napier – Munn, 2006). The selection of the proper combination of reagents that will produce the required concentrate from a particular ore constitute the major challenge for a mineral process metallurgist.

(ii) *Flotation Reagents*

Flotation reagents are generally classified into collectors, frothers and regulators (activators, depressants or pH regulators).

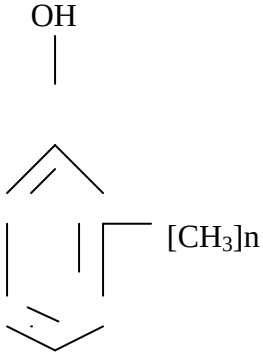
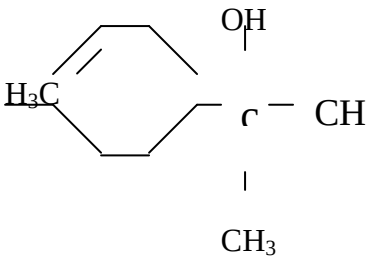
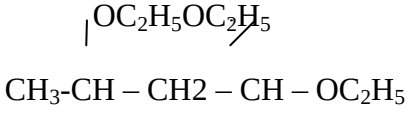
(a) *Collectors* are reagents that selectively adsorb on the surface of particulates to make them hydrophobic (i.e. floatable). These are added to the pulp and time allowed for adsorption during agitation known as conditioning time. Examples of collectors include xanthates for sulphide flotation, fatty acids (oleic and linoleic acid) and amines for non sulphide flotation. *For example, the primary amines(i.e. those containing only one hydrocarbon group with two hydrogen atoms) are strong collectors of apatite and can selectively float sedimentary phosphate from calcareous ores (*Willsand Napier – Munn, 2006*). The collector requirement is usually reduced by adding a non-polar reagent like kerosene which is co-adsorbed on the mineral surface.*

(b) *Frothers* are reagents that create stable froth capable of carrying mineral laden bubbles until they can be removed from the flotation cell. Examples include pine oil, cresylic acid, MIBC, etc.

(c) *Modifiers or regulators* are reagents that enhance (activators) or inhibit (depressants) the selective adsorption of collectors. Examples of depressants include starch, dextrin, sodium silicate, sodium cyanide, slimes, etc with copper sulphate used in the activation of sphalerite in lead-zinc flotation, sodium sulphide or sodium hydrosulphide used in the activation of oxidized minerals of lead, zinc and copper being some examples of activators.

Flotation where possible is carried out in an alkaline medium, as most collectors are stable under these conditions, and corrosion of cells, pipework, etc are minimized. Alkalinity is controlled by the addition of lime, sodium carbonate, (soda ash), and to a lesser extent sodium hydroxide and ammonia. Sulphuric acid is used where decrease in pH is required.

Table 2.2: Some Common Flotation FrotherReagents(Source: Abouzeid ,1990)

Chemical Type	**Structural Formula**	**Trade Name**
Methyl alcohol	C_5H_{11}- OH	Aero froth 70
Methyl Isobutylcarbinol	M.I.B.C.	M.I.B.C.
Cresylic Acid		Cresylic Acid
Pine Oil		Pine Oil Flotol
Polypropylene glycol ether	H_3C-$(O$-$C_3H_6)_n$- OH	Dowfroth 250
Triethoxybutance		T. E .B .Frother

(d) *Flotation Kinetics*

Flotation (rougher) kinetic tests are mostly carried out by collecting multiple concentrates taken over a measured time period. These tests provide valuable information in a single test that result in the establishment of grade and recovery relationship as well as flotation time. The evaluations are either carried out using the classical method of evaluating rougher flotation kinetic data as devised by Arbitter and Harris, 1962 or by the use of the incremental grade approach (Thompson, 2002).

The classical method generates a rate curve that fits a first order kinetic equation

$$ln\ (C_o/C) = kt \qquad (2.7)$$

where C_o is the concentration of the valuable mineral in the pulp at the beginning of the test (time zero minutes), C is the concentration of valuable mineral in the pulp at time t minutes, and k is the rate constant. Plotting ln $[C_o/C]$ against t produces a straight line with a slope equal to the first order rate constant.

The value of k, the slope, determines the collecting power of the reagent. The flotation kinetics increases with the rate constant. The steeper the slope, the greater the collecting power.

In the incremental grade approach, the grade of the individual timed concentrate samples and cumulative grade of the overall concentrate are plotted against time. The cumulative recovery is also plotted against time on a second axis.

Flotation is considered complete when the incremental concentrate grade curve crosses the head grade of the ore feed to the test, implying that no additional concentration from the ore is taking place. The time at which this intersection occurs determines the cumulative concentrate grade and recovery for the test.

(iii) *Flotation of Phosphate Rock*

Phosphate flotation as practiced by most phosphate producers (Grubber, 2003) is carried out using the crago double float process which entails dewatering sized flotation feed to a pulp density of 70% solids. This is conditioned with fatty acids / fuel oil at a pH of about 9 for three minutes. The phosphate is floated to produce a rougher concentrate and tailings.

The rougher concentrate is then dewatered, scrubbed using an acid scrubber and a wash box to remove the reagents from the phosphate surfaces. After rinsing, the de-oiled rougher concentrate is transferred into flotation cells where amine is added to facilitate the floating of silica at a neutral pH.

This process is highly inefficient arising from the fact that:

i. De-oiling consumes significant amount of sulphuric acid which calls for special safety caution and equipment maintenance;
ii. Inefficient de-oiling causes poor concentrate grade;
iii. De-oiling also causes loss of fine phosphate particles; and
iv. Amines are more expensive than fatty acids as well as being very sensitive to water quality particularly slime content in water.

The above disadvantages have led to various research works among which is the single collector all anionic phosphate recovery (Zhang, 2003). This involves the use of anionic collectors in the form of oleic acids, stearic acids amongst others. Other works include the use of alhcohlic solutions of hydroxamic acid in floating sedimentary phosphate rocks (Miller, Wang and Li, 2002; Zhang, 2003).

2.4.6. *Solid – Liquid Separation*

This refers to the removal of water from solids so that a relatively dry concentrate is produced for shipment. Dewatering techniques can be generally classified into three groups:

a. Sedimentation or thickening which refers to the settling of solid particles in a liquid producing in the process, a clarified liquid which can be decanted leaving a thickened slurry;
b. Filtration which refers to the process of separating solids from liquid by means of a porous medium which retains the solids but allows the liquid to pass; and
c. Thermal drying which refers to the heating of the cake to about $105^{o}C$ for the purpose of driving off water as water vapour.

Dewatering in mineral processing is usually carried out using a combination of the above techniques. The bulk of the water is first removed by sedimentation or thickening, which produces thickened slurry of about 55 – 65% solids by weight resulting in the removal of

about 80% of the water (Wills et al, 2006). Filtration of the thick slurry then produces a moist cake of about 80 – 90% solids which may require thermal drying to produce a final product of about 95% solids by weight.

2.4.7. *Tailings Disposal*

Tailings disposal refers to the doing away of solid waste material generated by the mineral processing plant during the course of producing the required mineral product. The disposal of mill tailings is a major environmental issue which is becoming more serious with increasing exploration for metals and the working of low grade deposits. Apart from the visual effect on the landscape, the major ecological effect is usually water pollution, arising from the discharge of water contaminated with solids, heavy metals, mill reagents, sulphur compounds, etc (Wills et al., 2006). Waste generated by the mill must therefore be disposed of in both environmentally acceptable and if possible, economically viable manner (Will et al., 2006).

Tailings are nowadays disposed of by positively using them such as reprocessing in order to recover additional values or to use them as a useful product in their own right like using the floats from a barite jigging operation as aggregate.

Tailings can also be used in filling mined out areas where coarse fractions of the tailings are used.

Tailings dams can also be constructed for the purpose of impounding milling tailings thereby preventing it from polluting the environment as well as reusing clarified water from the tailings pond as process water.

2.5 Mineralogy of Phosphate Rock Deposits

The mineralogy of phosphates is complex and obscure because of their tendency to occur as microcrystalline aggregates. Typical phosphate minerals are admixtures of the phosphate radical (PO_4) with calcium, water, and traces of fluoride and uranium.

Phosphates occur in rocks as matrix and as nodules, ooilths, pellet and phosphatized shells, bones and teeth. They also occur as a bulk replacement of limestones. Phosphates have been recorded from the modern sea bed, notably off the western coasts of America and Africa. Detailed studies show the wide range of occurrence of these deposits. Some phosphates have been recorded forming diagenetically within organic-rich diatomaceous oozes of the South West African shelf. On the South African shelf diagenetic replacement by phosphate occurs in Miocene limestones which are eroded to form phosphate gravel placers. Further north, off the West African shelf, phosphate placers formed are derived from Eocene Moroccan and Pliocene Sahara phosphorites. This trend explained possibly the occurrence of the of the Sokoto phosphate mineral deposit since it takes it routes from the Sahara phosphorites mineralization processes. The phenomenon illustrates three characteristic features of phosphate minerals in which their genesis which explained the replacement of carbonates; their ability to be eroded and reworked, and their occurrence on continental shelves (Weiss, 1985).

Table 2.3:Main modes of occurrence of economic phosphates mineral ore deposits

Source: (Weiss, 1985)..

S/No	**Type**	**Mineral**	**%Total world production**
1	Primary igneous	Apatite	24
2	Sedimentary {bedded} { placer}	Collophane} And Apatite}	74
3	Guano	Complex Phosphates and Nitrates	2
			100

Table 2.4: Mineralogical compositions of the economic phosphates mineral ores deposits

(Source: Weiss, 1985)

S/No	Mineral Name	Mineral Compound formula and % Composition
1	Apatite	$Ca_6(PO_4)_3(F,Cl,OH)$: CaO(55.38%), P_2O_5(42.06%), F(1.25%), Cl(2.33%)
2	Amblygonite	$LiAl(PO_4)F$: LiO(10.10%), Al_2O_3(34.46%), P_2O_5(48.00%), F(12.85%)
3	Lazulite	(Mg, Fe) $Al_2(PO_4)_3(OH)_2$: Mg:Fe(1.0), MgO(13.34%), Al_2O_3(33.73%),P_2O_5(46.97%)
4	Wavellite	$Al_3(OH)_3(PO_4)_3.5H_2O$: Al_2O_3(37.11%), P_2O_5(34.40%)

Table 2.5: Chemical analysis of some the world phosphate mineral ores deposits (Sources: Weiss, 1985; MIN, 1987)

S/ No	Mine field		% P_2O_5	% SiO_2	% MgO	% F	% Fe_2O_3	% Al_2O_3	% CaO	Insol %	% Phosphate Pebble	CaO/ P_2O_5 ratio	% Free $CaCO_3$
1	North Carolina (USA)		14.60	-	1.20	-	1.30	0.90	25.83	43.30	47.00	1.80	5.00
2	Idaho (USA)	F/Shale	24.70	26.0 0	0.54	2.38	1.57	2.48	35.75	-	-	1.50	-
		H/Grade	30.83	7.93	0.33	3.00	0.50	1.06	45.57	-	-	1.5	-
3	Togo		36.60	3.25	-	3.61	1.20	2.10	52.30	-	-	1.5	-

Other deposits with similar characteristics of the phosphate mineral mentioned above could exhibit the percentage of P_2O_5 as low as 5.8%, e.g. the Upper low grade phosphate ore deposit section found in North Carolina mine fields in United States. But this limitation of the deposit does not stop the utilization of the mineral deposit in producing low grade fertilizer and other phosphorus related products (Weiss, 1985).

Flotation has been identified as one of the mineral processing technique for the beneficiation of phosphate mineral deposits. But this flotation process sometime is complex due the interaction of the reagents used and the phosphate mineral ore to be beneficiated. Hence, the need to understand the chemistry of flotation process of phosphate mineral ores classified as one of the semi-soluble mineral ore salt (Weiss, 1985).

2.6 The chemistry of Semi-soluble Salt flotation

Semi-soluble salt minerals include carbonate, phosphate, sulfate, tungstate, and some halide minerals. These minerals are characterized principally by their ionic bonding and their moderate solubility in water. Many of these minerals have solubilities on the order of 10^{-4} mole/l. the surface charge of these mineral in water is a function of the concentration of the ions of which their lattices are composed. However, the PZC (point of zero charge) of many minerals is determined by the hydrogen ion concentration. Anionic collectors, in particular carboxylic acids, are used most frequently in the flotation of semi-soluble salt minerals. Soda ash rather than lime is used for pH control in many instances to avoid bulk phase precipitation of calcium salt of the collector and to prevent calcium activation of gangue silicates.

In most cases collector adsorption in these systems involves chemisorption. This phenomenon results from the stability of most multivalent cation-carboxylate salts and from the moderate solubility of the semi-soluble salt minerals.

(a) Calcite flotation

Due to the ubiquity of calcite in ore deposits, the surface properties, the adsorption characteristics of various collectors, and the flotation response of the calcite have received considerable study. Electro-kinetic studies have revealed that calcite exhibits an initial PZC at pH 10.8 water; the PZC is reduced in value until equilibrium is finally reached at pH8.2 which is the solution isoelectric point. This phenomenon occurs with long aging periods (days). For flotation purposes the PZC of calcite is pH 10.8

With regard to flotation, recovery has been established as a function of collector concentration and hydrocarbon chain length. Both fatty acids and sulfonates have been investigated, and the results obtained with fatty acids have been published. The summaries of the results showed that when the length of the hydrocarbon is increased, the concentration of carboxylate necessary for flotation is reduced. A kT plot of these data yields a slope which corresponds to a specific adsorption potential of -1.1kT, which value corresponds to the free energy decrease(-0.62 kcal/mole/CHz group) associated with the hydrocarbon chain removal from solution either by the formation of a calcium carboxylate salt or by hydrocarbon chain association(Weiss, 1985).

(b) Apatite-Collophane Flotation

Calcium phosphate occurs as either the mineral apatite or as collophane, a substituted cryptocrystalline calcium phosphate, chemically similar to apatite but quite dissimilar with regard to appearance, surface area, and porosity. Electro-kinetic experiments have shown that apatite has a point of –zero-charge at pH6.4 a complete flotation response is usually observed from the pH5 to 13, which reveals that oleate is adsorbing chemically on the surface. For a negatively charged ion (RCOO-) the PZC, a chemical reaction between carboxylate ion and surface calcium must be occurring. In fact, Rickard in Weiss, 1985 has established with infrared spectroscopy the formation of surface calcium oleate on apatite and collophane.

Processing of phosphate rock or minerals containing nodules of collophane involves desliming at -150mesh material, which frequently contains one-third of the P_2O_5 values, is discarded. Two unique features are involved in collophane flotation, namely the use of neutral hydrocarbon oils and conditioning at high pulp density. Both of these features probably reflect the porosity and resultant high surface area of collophane. Apatite, on the other hand, responds well to flotation without prior conditioning at high pulp density and in the absence of fuel oil. Apparently, the function of the neutral hydrocarbon oil, whether the collector is a fatty acid or a sulfonate, is to bridge adsorbed collector molecules and to fill the pores of the highly porous collophane. Such action would reduce collector consumption and aid in the surface dehydration process. Larger and more branched molecules would be required to accomplish this, perhaps explaining the effect of viscosity of the oil (Weiss, 1985).

(c) Modifiers and Depressants

Mechanisms involved in depression reactions have not been investigated thoroughly, and as a result, limited understanding of these systems has been achieved. Not only are the depressant adsorption reactions complex, but the depressants are frequently complex molecules themselves. The basic depressants used in semi-soluble salt flotation systems are sodium carbonate, sodium silicate, sodium meta-phosphate, lignin sulfonate, starch, quebracho, and tannin derivatives.

(d)Sodium carbonate:

surface carbonation is an important reaction that occurs in semi-soluble salt flotation systems, simply from the CO_2 in the atmosphere or from carbonate minerals present. In the case of fluorite, dissolved carbonate reacts with the surface to form a calcium carbonate compound. As a matter of fact, this phenomenon accounts for the PZC of fluorite being at pH 10. The practical implication of this phenomenon is that in the flotation of semi-soluble salts where soda ash is used for pH control, surface carbonation seems inevitable, and the fact that good selectivity is achieved seems remarkable.

(e) Sodium silicate:

Calcite is most commonly the principal gangue constituent in these systems. One depressant, which also acts as a dispersant and which has been used with a fair degree of success, is sodium silicate. It is used extensively in the separation of fluorite from calcite and in the separation of scheelite from calcite and apatite. Sodium silicate solutions are complex; the general equilibria are:

Log K

SiO_2(s) (amorphous) + $2H_2O$ = $Si(OH)_{4(aq)}$ -2.7

$Si(OH)_{4(aq)} = SiO(OH)_3^-$ + H^+ -9.43

$SiO(OH)_3^- = SiO_2(OH)_2^{-2}$ + H^+ -12.56

$4Si(OH)_4 = Si_4O_6(OH)_6^{-2}$ + $2H^+$ + $2H_2O$ -12.57

Source: Weiss, 1985

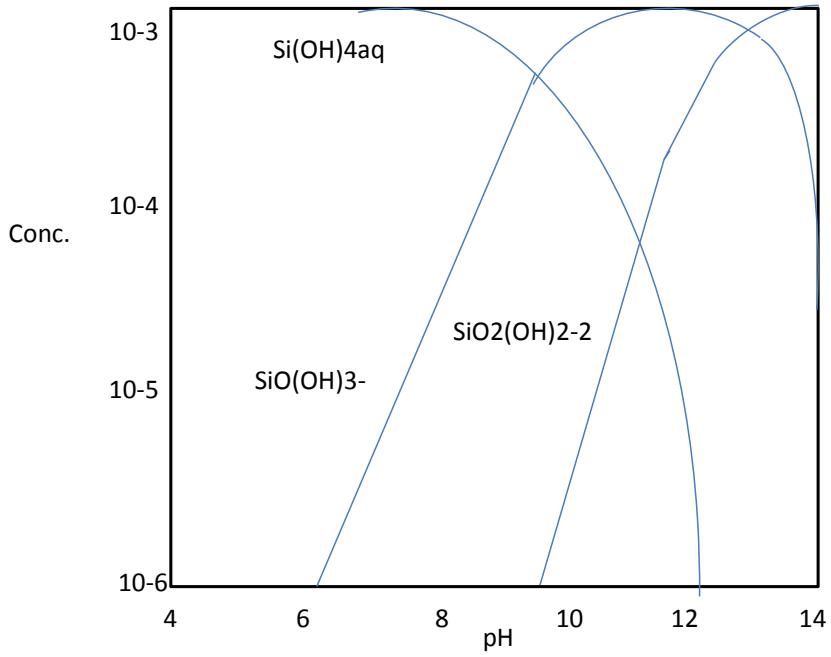


Fig. 2.1: The logarithmic concentration of dissolved silicate species in a system from $1x10^{-3}$M sodium silicate (Weiss, 1985).

The concentration-pH domains for these species are shown in Fig. 1.0 alkaline conditions are seen to promote the formation of polyanions. Further, for all sodium silicate solutions, when the concentration of Si (OH) $_{4aq}$, is less than about $5x10^{-3}$ mole/l, colloidal silica is not stable. The critical nature of the use of this achieving selectivity in nonmetallic flotation systems has been presented by Joy and Robinson in Weiss, 1985 in their study on apatite, calcite and fluorite. They concluded that calcite is by far the most sensitive to sodium silicate additions. Where they stated that flotation recovery of calcite with $5x10^{-4}$ mole/l oleate in the presence of $5x10^{-4}$mole/l at a pH6 essentially complete is attained. This is the domain of Si(OH)4aq which presumably does not adsorb on positively-charged calcite. From the pH7 to pH10, however, complete depression can be noted. This is the domain of $SiO(OH)_3^-$. Four varieties of sodium silicate are available containing various ratios of SiO_2 to Na_2O. These ratios are 1.60:1; 2.75:1; 3.22:1 and 3.22:1, SiO_2:Na_2O. The effectiveness of these various types on calcite flotation showed that the higher the ratio of SiO_2 to Na_2O, the more effective the sodium silicate is in depressing calcite. When the oleate concentration is $1x10^{-4}$mole/l, even with the 1.6:1 sodium silicate, calcite is depressed at about pH10 and above. In case of fluorite, an addition of $1x10^{-4}$mole/l, sodium silicate has no effect on flotation response. With an addition of $1x10^{-3}$mole/l, however, flotation recovery is reduced above about pH10. While in the case of apatite, the system is depressed about one-haft unit in pH sooner in acid medium in the presence of $1x10^{-3}$mole/l sodium silicate.

(f)Starch:

Starch was recommended as a depressant as early as 1931. Industrial application has been extensive since that time, but basic systematic work dealing with mechanisms governing the depressant action has been limited. Structurally, starches and dextrins are polymers of dextrose monomeric units linked through 1-4 glycoside linkages (for straight chains) and 1-6 linkages (for branch points). In starches, the linear chain(amylase) and branched chain (amylopectin) components have molecular weights reaching millions. In dextrin formation these chains are fragmented and recombined to form low molecular weight but highly branched structures.

Starches are anionic species and their adsorption on positively charged surfaces can occur by coulombic attraction. More often, however, adsorption of starch occurs by hydrogen bonding with surface oxygen ions or by specific chemical interaction with metal ions of the surface. Corn starch contains carboxyl groups, while potato starch contains phosphate groups. It is these groups which react chemically with surface metal ions.

In a study of starch adsorption and its effect on semi-soluble flotation with oleic acid, Hanna in Weiss 1985, has shown that calcite is depressed at lower concentrations of starch than is barite which, in turn, is depressed at lower concentrations than fluorite.

2.7 Challenges of the Existing Phosphate Rocks Beneficiating Plants

The phosphaterock from the mine fields of the United States are processed to metallurgical grades for fertilizers production through sizing to the required liberation size, washed, deslimed and upgraded using froth flotation process applying the right volume of reagents. Slimy feeds increase the cost of flotation reagent and promote poor recovery of the phosphate minerals concentrates. Flotation process of phosphate mineral ores is inhibited if oversize in the flotation which is usually phosphate pebble present will simply not float. The cutoff points of between 1400microns to 841microns are preferable.

Many feeds are sized into fine and coarse fractions for beneficiation. Two factors influence this sizing:

(i) Better recoveries can often be obtained when the two size fractions are reagentized separately, because they usually need different reagent additions for best recovery.

(ii) The conditioning requirements often vary. Most plants use a large rotary conditioner for the coarse feed fractions and a paddle-type vertical mixing tank for the fine feeds.

The present Florida and other phosphate flotation plants in the United States generally use the method developed by Arthur Crago that is ‘‘double-float’’. This method enables the conditioning of the rougher floated concentrates with sulfuric acid. This cuts the fatty acid surface coating which is washed out with clean water. The washed rock is then subjected to flotation using cationic reagents to float silica. This method was a big improvement over the earlier phosphate oil float and is now used almost

exclusively. The phosphate oil float is relatively stable, but the silica float cannot tolerate lengthy conditioning or slow flotation practice. The Crago double-float provides more flexibility, although the straight phosphate oil float with cleaning is capable of making concentrate with only 6 to 7% insoluble. The double float can produce 2 to 4% insoluble products if the need arises. The reagents used in the Florida and others beneficiation plants in the US are alkali (NaOH, anhydrous NH_3 or NH_4OH), fatty acid (mostly tall oil), fuel oil, kerosene, amine and H_2SO_4. All the reagents are obtained in liquid form. The reagent cost is a very important item in a phosphate flotation plant. The quantities used vary from plant to plant. E.g. phosphate oil float: Alkali to pH 8.0 to 9.0 at conditioner, Fatty acid: 0.8 to 2.0lb per ton flotation feed, Fuel Oil: 1.0 to 4.0 lb per ton flotation feed;

De-oil: H_2SO_4 to pH 3.0 to 4.0; Silica float: Amine 0.10 to 0.20 lb per ton feed to cleaner circuit, kerosene: 0.05 to 0.25 lb per ton feed to cleaner circuit, Alkali if needed in silica circuit.

Table 2.6: Comparative Flotation Processes and the % Assays of a Beneficiated Florida Phosphate Mineral Ore

S/No.	Material	$\%P_2O_5$	%CaO	%MgO	$\%Fe_2O_3$	$\%Al_2O_3$	% Insoluble	CaO/P_2O_5 ratio
1	Single-flotation Concentrate	28.50	46.10	0.60	0.90	0.40	7.70	1.61
2	Single-flotation Concentrate calcined	29.50	47.20	0.50	0.70	0.40	8.60	1.60
3	Double-flotation Concentrate	30.70	49.10	0.50	0.70	0.40	2.10	1.60
4	Double flotation Concentrate calcined	33.10	53.20	0.50	0.80	0.40	2.10	1.61

Source: Weiss, 1985

In Idaho phosphate mine field, the ores occur as high grade beds, of +30%P_2O_5, and furnace-grade or milling-grade ores of 22 to 30%P_2O_5. There is a considerable tonnage of

lower grade shales that might be beneficiated before utilization. The phosphate ore is mined and blended carefully, with a minimum of free lime contamination, the different grades of the ores are blended to maintain a +24% P_2O_5 for furnace feed.

All ores under 30% P_2O_5 are beneficiated before they can be used efficiently in the wet-acid process for the production of phosphoric acid used for fertilizer manufacture. The beneficiation process includes, scrubbing, desliming, filtering, and drying. The higher the P_2O_5 content of the plant feed, the better the recovery with low operating cost. The reason for this is that the amount of P_2O_5 in the tailings does not change for sizes below -44 microns.see Table 2.7 and Figure 1.2 below.

Table 2.7: % assay of P_2O_5 in the various sieve size fractions in Idaho Phosphate (Source: Weiss, 1985)

Sieve Size(μm)	%Assay (P_2O_5)
-44 +15	12.70
-15 + 10	12.10
-10 + 5	11.30
-5 + 2	11.10
-2 + 0.25	12.00
-0.25	4.50

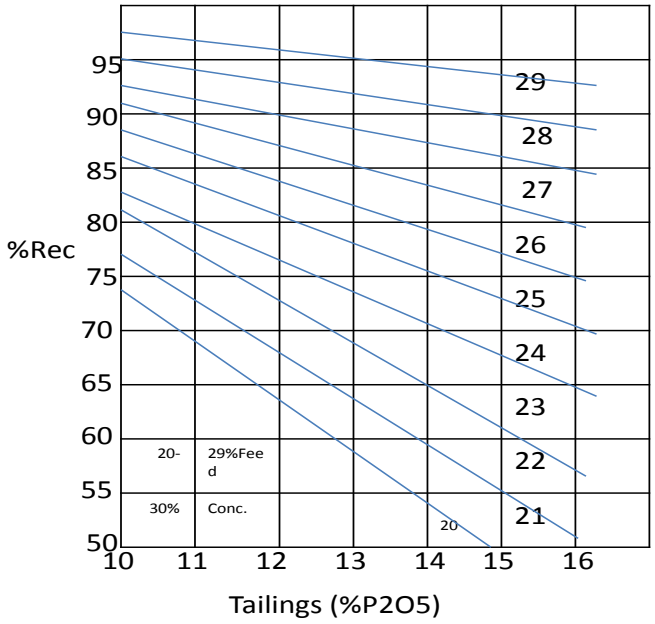


Figure 2.2: The relation between recovery and tailings for various western plant grades (Weiss, 1985).

In practice much is done to eliminate as much free lime (limestone) as possible. The elimination of free calcium carbonate in phosphoric acid feed ores is important from the economic standpoint of consumption of sulfuric acid in the production of phosphoric acid. In the beneficiation process of the phosphate ores, ores averages 20% P_2O_5 are firstly subjected to scrubbing and desliming which produces a grade of 24%P_2O_5, and flotation increases the grade to 31%P_2O_5. The Tennessee operations is another typical issued as beneficiation of the phosphate mineral ore is concerned. Here the ore mined runs about 21%P_2O_5, and the final washed product about 26 to 31%P_2O_5 which is high in iron and

alumina, making it unsuitable for making phosphoric acid by wet method. But it could be utilized in other metallurgical engineering applications (Weiss, 1985).

The North Carolina phosphate concentrate is calcined and is used to produce phosphoric acid and super-phosphoric acid of extreme purity, reaction with sulfuric acid and evaporation of water. Triple superphosphate is produced by reacting of ground uncalcined phosphate concentrate with phosphoric acid. Di-ammonium phosphate is produced by reacting ammonia with phosphoric acid, Phosphoric acid (54%P_2O_5), and superphosphoric acid (72% P_2O_5). Below is the percentage chemical composition of typical fertilizers used in agricultural applications produced from the North Carolina phosphate mineral ore: NPK: 0-46-0; NPK: 18-46-0;

2.8 Mineral Process Design

Mineral process design refers to the process of selecting a suitable combination of mineral process unit operations based on results obtained from process tests carried out on representative samples that will facilitate economic production of mineral concentrates in an environmentally friendly manner that meet desired product specifications in terms of grade, size range, moisture content, etc.

The development of mineral processing flow sheet for the beneficiation of a mineral deposit involves carrying out bench scale tests to determine the response of the mineral assemblage to process tests. The various tests that are usually carried out in the course of designing mineral process routes include:

a. Mineral ore characterization which involves:
    i. Chemical analysis to determine the chemical composition of the mineral ore,
    ii. Size/assay analysis to determine the size and chemical distribution of the ore,
    iii. Mineralogical analysis to determine the mineralogical composition and association of the mineral ore,
    iv. Grain size determination to evaluate the grain size of the various minerals contained in the mineral ore,
b. Liberation studies comprising work index and economic grind size determination,
c. Separation tests determined by the mineralogy of the mineral ore which may include sink float test, froth flotation test (batch / locked cycled tests), etc.

The results obtained from the above tests will determine the type of unit operations and thus the process flow sheet that would be needed to produce a marketable product(s).

2.8.1 *Flotation Flow-sheet Design*

Flotation flow-sheet design refers to the process of selecting and sequentially arranging flotation steps (circuits) required to produce mineral concentrate(s) of prescribed grade using results obtained from flotation tests carried out on representative mineral samples.

Steps involved in carrying out flotation flow-sheet design comprise the following:

i. Estimation of primary grind size based on either past experience or from mineralogical evaluations;

ii. Carrying out batch flotation tests using various reagents combination on samples ground to various degrees;

iii. Collection of incremental concentrates at measured period of time, weighing the concentrates collected and assaying same with the results evaluated by plotting recovery – time and recovery – grade curves etc to establish grade and recovery relationship as well as flotation time using the classical method of evaluating rougher flotation kinetic data as devised by Arbitter and Harris, 1962 or by the use of the incremental grade approach (Thompson, 2002).

Grind size is estimated from the knowledge of the size of grains in the ore which can be estimated using mineral liberation analysis.

The purpose of primary grind is to effect the attainment of economic recovery of the valuable ore minerals. Initially, a grind size is chosen which gives a reasonable rougher grade and recovery within acceptable flotation time. If grinding is too coarse, some of the valuable minerals locked in middlings grains will not be floated. However, excessive flotation time may allow some of these particles to be floated thereby reducing the concentrate grade.

Grinding, being the greatest single operating cost, should not be carried out finer than justified economically (Wills, B. A. and Napier – Munn, T. J., 2006) that is, it should not be carried out beyond the point where the net return for the increment grind becomes less than the operating cost.

After determining the primary grind size, further tests are carried out to determine optimum reagent additions, pH, pulp densities, etc.

Having optimized flotation recovery, further test work is carried out to produce the required concentrate grade and determine the flow-sheet which must be utilized to achieve this. This usually involves assessing the amount of secondary grinding required to reach the specified grade and the number of cleaning stages required.

Generally, most of the valuable minerals float within a few minutes whereas it takes much longer time for the residual small quantity to float (Wills, B. A. and Napier – Munn, T. J., 2006).

Flotation activity falls off very rapidly above the optimum particle size due to lesser liberation of the valuable minerals and to the decreasing ability of the bubble to lift the mineral particle.

The floated mineral is composed of fast floating fraction in the medium-size range and a more reluctant fraction comprising un-liberated material and fines.

Flotation circuits are generally made up of Roughing, Scavenging and Cleaning Stages with the fast floating minerals being floated in the roughing section while the more reluctant fraction is recovered by scavenging. Certain losses into tailings must however be accepted. The cleaning section involves floating valuables with less severe flotation conditions as obtained in the roughing stage or selectively floating impurity minerals to obtained specified concentrate grade.

The grade of the final concentrate is dependent on the grade of the rougher concentrate, and in order to obtain the optimum cleaning grade, it is necessary to keep the rougher grade at a predetermined value.

The decision as to where the rougher – scavenger split should be, can be made on the basis of batch tests where cumulative concentrate grade is plotted against time, the time limit for the rougher flotation being fixed as that required to give a rougher concentrate with a high enough grade to produce the specified final concentrate grade with the chosen number of cleaning stages. The remaining flotation time is for scavenging, this time being reduced by increasing the severity of flotation condition either by increasing the aeration rate or by the addition of more powerful collector after the removal of the rougher concentrate. The objective of scavenging is to promote maximum recovery by minimizing losses to tailings. Rougher – scavenger are split at the flotation time where the separation efficiency is maximized (Wills and Napier-Munn, 2006) which occurs when the grade of the concentrate produced is equal to the grade of the flotation feed. In practice, this is

established by using the incremental method of evaluating rougher flotation data as proposed by Thompson, (2002) obtained by plotting Incremental Grade versus Flotation Time; Cumulative Grade Versus Flotation Time and Cumulative Recovery on a second axis versus Flotation Time with the split time determined by the flotation time at which the incremental grade curve crosses the feed grade value. The cumulative recovery and cumulative grade of the rougher concentrate are then read off from the corresponding curves at the split time.

Control of flotation circuits is usually carried by adding a thickener or agitator which serves as surge capacity for large changes in circulating load.

If cleaning does not give the required concentrate grade, regrinding of the rougher concentrate may be needed. Generally, the scavenger concentrate and the primary cleaner tailings are usually reground before recirculation to the roughing circuit.

(a) The Philosophy of Process design flow-sheet and Methods

The philosophy of the process design flow-sheet for the beneficiation of mineral ore deposit is discussed on the basis of the findings obtained during the research work. From the results of the chemical, mineralogical, petrological, grindability test and particle size analyses obtained for the mineral ore, the predominant minerals, minor, associates and their degree of association can be utilized in the design. The grindability test result usually reveals that ore texture whether it is of the soft type and with meaningful liberation of the valuable bearing minerals. Concentration test methods results obtained reveal the best

adoptable method for concentrating the mineral ore sample. The method used, raised the valuable mineral content to an acceptable metallurgical grade required for further utilization. Figure 2.3 is a typical example of a process design flow-sheet of one of a Nigerian iron mineral ore deposited in BirninGwari. The developed Flow-sheet illustrates the process route for the beneficiation of BirninGwari iron ore. The process design flow-sheet consists of two stages of operations and is aimed at producing sinters for blast furnace process reduction for iron making. The stages are as follows:

(i) Comminution(Crushing, grinding and screening)

(ii) Concentration

The philosophy of the process design route for the beneficiation of BirninGwari iron ore for use in iron and steel production is conducted in two phases. The first phase involved the determination of the chemical, mineralogical, petrological, grindability and particle size/assay analyses. From the analyses and the tests the grade, mineralogical, liberation characteristics and work index of the ore were determined. The second phase involved carrying out concentration tests using mineral processing techniques of magnetic, gravity and froth flotation. From the tests the effective adoptable method for the concentration of the BirninGwari iron ore was determined. Hence, it is on the basis of these findings that the proposed process design flowsheet for the beneficiation of BirninGwari iron ore for blast furnace process route was developed as shown below.

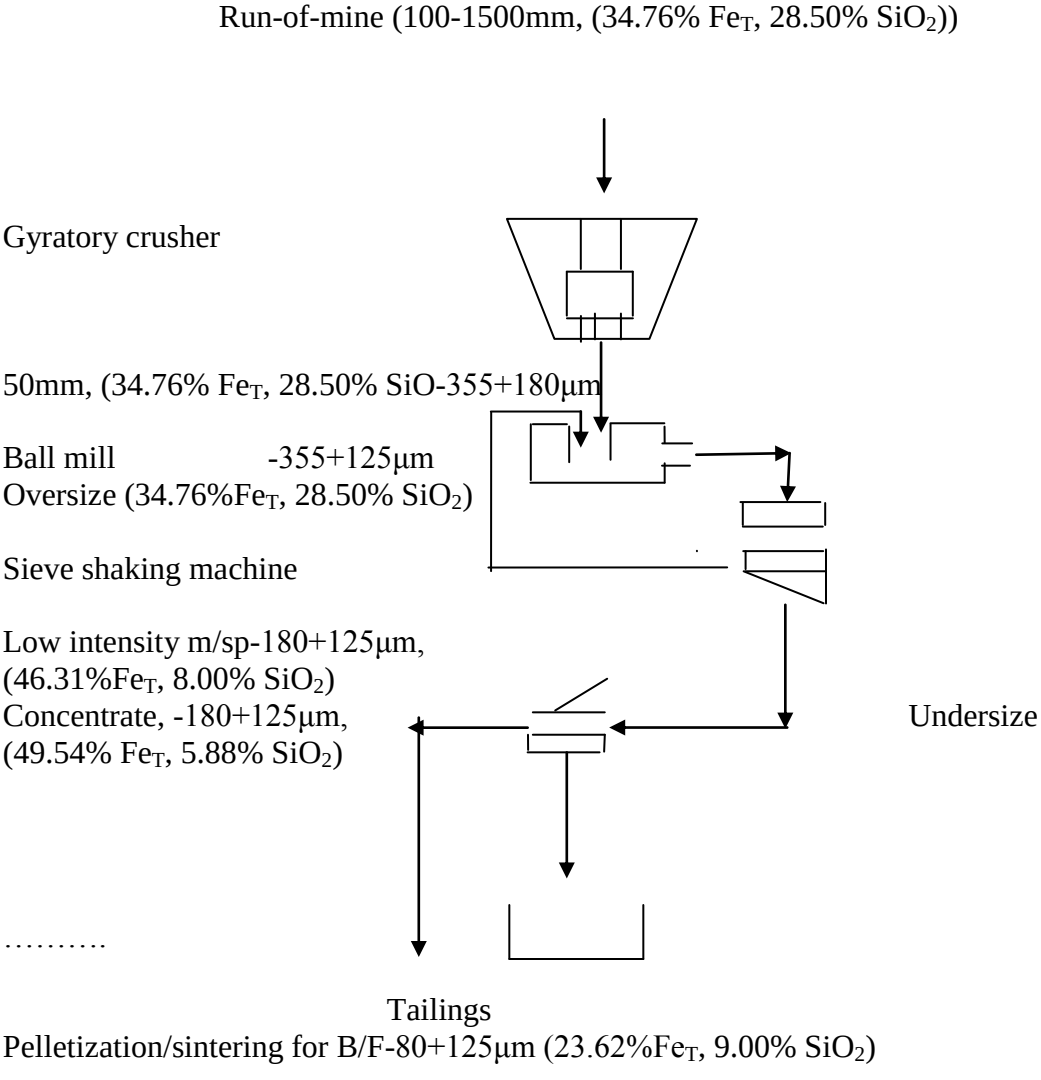


Figure 2.3: Process design flow-sheet diagram for the beneficiation of BirninGwari iron ore (Source: Thomas eta-al, 2013).

The philosophy, methods, figure and descriptions illustrated above will be adopted, modified and applied in the design of the process flow-sheet for the beneficiation of the Sokoto phosphate mineral ore deposit.

2.9 Analytical Methods Used For Metallurgical Analyses

Metallurgical analyses involve the qualitative as well as the quantitative analysis of various metallurgical raw materials like ores and products like metals, alloys and slags. The analytical methods employed in metallurgical analysis can be broadly classified into two: (i) Classical methods (ii) Instrumental methods.

In the instrumental methods of analysis, measurement is made upon the system as a whole. A particular physical or chemical property of the system is measured in order to get the desired analytical results. Comparisons of the result of the samples to be analyzes with standard samples of known composition is necessary in this method.

The most common instrumental analytical techniques used are Atomic Absorption Spectrophotometry (AAS), X-Ray Fluorescence Spectrometry (XRFS), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Electron Probe Micro Analysis (EPMA) (Funtua, 1999).

2.9.1 *X-Ray Fluorescence (XRF) Analysis*

This technique works on the principle that when a material is irradiated by x-rays, electrons are ejected from the atoms making up the material. The vacancies created by these ejected electrons are then filled by other electrons either from the higher shells within the atom or outside the atom. These electrons lose energy mainly in the form of radiation or x-rays. The x-rays will have a precisely defined energy and the wavelength (velocity of electron) are related by the Plank's energy equation

$$E1-E2=hc \qquad (2.8)$$

where E1= Former energy level of the electrons before filling the vacancy.

E2= New energy level of the electrons after filling the vacancy

h= Plank's constant

c= velocity of light

As the energies or wavelengths are characteristics of the atoms of the elements emitting the x-rays, they are used for qualitative analysis while the intensity of these characteristics x-rays is related to the concentration of the particular element in the material and is used for quantitative elemental analysis (Funtua, 1999).

2.9.2 *Scanning Electron Microscopy (SEM)*

This method is used to study the properties of materials at microscopic level. The most important applications are the investigation of the micro-structure and analysis of in-homogeneity such as defects, dislocation, submicroscopic precipitates, inclusions, domains

etc. It is also used for determining the composition of individual grains of particles and surface properties like topography.

In SEM, a small area of the specimen of a material is irradiated with direct electron beam and characteristics x-rays are produced which are used for the qualitative and quantitative analysis. The most important of the produced electrons (x-rays) called Backscattered or primary reflected electrons are detected and amplified. The output from this is a function of the composition of the area irradiated by electron beam.

The intensity of the Backscattered electron signals is dependent on the atomic number of the specimen and specimen surface of different chemical composition will produce different signal intensities and appear different on the SEM screen. The signal intensities are used for quantitative analysis.

2.9.3 *X-ray Powder Diffraction (XRD)*

X-ray diffraction analysis is a non-destructive method of analysis. It is use for both qualitative and quantitative analysis of materials. This method can be used to determine the actual compound(s) presents in a material and can also distinguish the different crystal forms of the compounds. However, this technique is only applicable to crystalline solids.

It is based on the diffraction of x-rays by polycrystalline solids and the reflections are called powder diffractometry. X-rays are deflected by crystals when the condition satisfying the Bragg's equation is fulfilled. The Bragg's equation is given by:

$$X=2dsinQ / n \qquad (2.9)$$

Where d= interplaner (lattice) space of Bragg angle

Q= angle between the family of planes and the x-ray in the reflecting position

X= x-rays wavelength

n= an integer called order of reflection.

When a sample of crystalline material is bombarded with x-rays and Bragg conditions are satisfied, some of the x-rays are deflected and they emerge with wavelength n from the crystals while all other wavelengths are either absorbed by the crystals or passes through it and are lost. These characteristics x-rays which are deflected by the crystals are detected by a detector and registered and displayed on paper chart recorder as series of peaks (x-rays patterns). Each compound has a unique x-ray powder pattern because the line position depends on the unit cell size and the type of atoms present and their intensities of powder lines are used for qualitative phase analysis while quantitative phase analysis depends on the accurate determination of the line intensity (Markowicz, 2002).

2.9.4 *Atomic Absorption Spectroscopy*

Atomic absorption spectroscopy has grown with amazing speed from its place in the early 1960's as a novel analytical technique to its present status as a method widely applied to all kinds of practical analytical problems. The principle of its operation is based on the fact that a ground state atom absorbs energy in the form of light of a specific wavelength and is elevated to an excited state, the amount of light energy absorbed at this wavelength will

increase as the number of atoms of the selected element in the light path increases (Barnasconi, 1996)

The basic instrumentation for atomic absorption requires a radiation source which emits the spectrum of the analyze element, an atomizer, such as a flame, in which the atoms of the sample to be analyzed are formed, a monochromator for the spectral dispersion of the radiation with an exit slit for selection of the resonance line, a detector permitting measurement of radiation intensity, followed by an amplifier and a readout device that presents a reading.

2.10 Nigerian phosphate mineral deposits

Occurrences of phosphate mineral are known in Sokoto, Ogun and Imo States. Some occurrences have also been reported in Edo and Delta States. Some chemical analysis results for some phosphate mineral samples are shown in Table 2.8 with source of information shown against each figure. As good as these figures look, none of the areas indicated has yielded any phosphate extraction activity due to inability to obtain phosphate materials of the qualities reported.

Nigeria is deficient in fertilizer minerals. The materials from Lake Chad area are tronaessentiallycontaining sodium and calcium carbonates, with practically no potassium salts. Other agricultural liming materials, that include ground limestone, lime, marl and oyster shells, all of which are soil conditioners. Their main function is to correct soil acidity, to granulate heavy clay soils and to provide plant food and promote the decay of

vegetable matter to form nitrates, and counteract some soil poisons are available in the country in many of the Nigerian states.

Table 2.8: Chemical analyses of some Nigerian phosphate mineral ore deposits.

Source: (Minerals and Industry in Nigeria, 1987)

S/No.	Location of Deposit	P_2O_5 content (wt. %)		Source of data
		Granular Phosphate	Nodular Phosphate	
1	Ogun State	31.99	31.38	Imperial Institute London
		31.40	31.50	Geol. Surv. Nigeria
2	Imo	31.50	32.00	Geol. Surv. Nigeria
3	Sokoto	-	35.00	Geol. Surv. Nigeria
		-	35.42	Integrated Fertilizer Development Centre
		-	36.25	Chem. Soc. Nigeria Bulletin, v7, p.144, 1982.
		-	35.10	Geology Dept. Univ. Manchester, England.

Phosphorus is also present in iron ores usually in the form of apatite orthophosphates of iron ($Fe_3(PO_4)_2$, $FePO_4$) and/or double oxides with iron as pent-oxide($xFe_2O_3.yP_2O_5$).

If x: y is less than 3 during the reorganization of ionic structure then it exists as $Fe_3(PO_4)_2$, otherwise $xFe_2O_3.yP_2O_5$ (Asuke, 2011). A typical source of this type of iron based phosphorus mineral ore is the Agbaja iron ore deposit, which has been reported to contain 2.08%P_2O_5. This phosphorus content is low compared to the other US phosphorus mined field deposits earlier mentioned. Secondly, it contained high percentage of iron compared to that of the US and hence making its utilization uneconomical in terms of acid consumption for the production of fertilizer (Weiss, 1985).

2.11 **Previous Work Done on the Sokoto Phosphate Mineral Ore Deposit**

Previous work carried out on Sokoto Phosphate Deposit comprised Exploration activities by the then Geological Survey Department of the Ministry of Mines, Power and Steel (GSD) and the Nigerian Mining Corporation (NMC) with processing studies conducted by the NMC.

No conclusive proven reserves were reported. However, the NMC reported the following results arising from their work on the Bonganange area of the deposit.

a. An average phosphate nodule grade of 22.5% P_2O_5;
b. A thickness of the phosphate nodules layer range of 0.2 – 2.10m;
c. An average weight percent nodules of 1.2%; and

d. A process flow-sheet made up of washing, screening and grinding with the capability of producing a concentrate of 80% passing 200microns, assaying 25.4% P_2O_5 with a recovery of 93.8%.

Furthermore, little have been done on the design of the process route for the beneficiation of the Sokoto phosphate mineral ore. The process used to produce a concentrate of 80% passing 200microns, assaying 25.4% P_2O_5 with a recovery of 93.8% does not make the concentrate produced fall into the high grade class required for high grade fertilizer production, but rather a concentrate of low grade as stated in Weiss, 1985. Hence the need for further exploitation of new techniques that will produce the required concentrate grade of highly less impurities. In doing this on the basis of the uniqueness of each mineral deposit characteristics which may vary from one depth to the other, specific and unique mineral processing techniques have to be adopted for the design of such mineral processing beneficiation route.

Chapter Three

3.0 Materials and Experimental Methods

3.1 *Materials*

(a) *Samples*

50kg of samples for test work were obtained by collecting random samples of Sokoto phosphate resource (rock and shale) mined by artisanal miners in Dange-Shuni, Bodinga, Yabo, Wurno, and Rabbah Local Government Area of Sokoto State. These were sorted to produce phosphate nodules that were blended and re-sampled to produce composite working quantities.

3.2 *Reagents*

Reagents used during the test work comprised AERO 704 (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.

3.3 *Experimental*

Composite sample of Sokoto phosphate rock was subjected to the following laboratory test work aimed at generating data needed to develop a process route for the beneficiation of Sokoto phosphate rock. The various tests carried out are as follows.

3.3.1 *Sokoto Phosphate Rock Characterization*

Sokoto phosphate rock was characterized by:

a) Determining the specific gravity of the Sokoto phosphate rock using the pycnometer (density bottle) method (Jones , 1987) as follows.

   i. Weighed an empty and dried 50ml density bottle (w);

   ii. Weighed empty bottle plus introduced Sokoto phosphate rock sample (w_1);

   iii. Weighed the bottle plus Sokoto phosphate rock filled with water to the top of the hole in the stopper (w_2);

   iv. Weighed density bottle filled with water only (w_3).

The specific gravity of the phosphate rock is given by expression:

Specific Gravity = Weight of Sokoto phosphate rock / Weight of water displaced by the Sokoto phosphate rock

Where:

Weight of Sokoto Phosphate rock = (w_1-w); and

Weight of water displaced by the phosphate rock = $(w_3-w)-(w_2-w_1)$

Implying that the specific gravity of

Sokoto Phosphate Rock = $[(w_1-w) / \{(w_3-w)-(w_2-w_1)\}]$ *(3.1)*

b) Determining the chemical and mineralogical composition of the phosphate rock along with the sizes of the various associating mineral grains XRD and XRF.

c) Determining the size – assay analysis of the Sokoto phosphate rock using a set of sieves mounted on a sieve shaker. The various size fractions retained on the various sieves were then weighed and assayed for $\%P_2O_5$ content using XRF analysis.

3.3.2 *Comminution Studies*

(a) Economic Grind Size

Representative samples of the blended phosphate rock were reduced in size using a combination of laboratory jaw crusher and rod mill with the ground products sized to determine their size distributions. These products were then subjected to separation tests to determine the economic grind size.

(b) Work Index

The work index of the phosphate rock was determined using the Berry – Bruce (1966) method which entails milling two mineral samples of the same weight, a reference ore of known work index (Magnetite, with a work index of 15.86 Kwh/t (Yaro, 1998) and the Sokoto phosphate rock using the same quantity of energy.

The work index of the phosphate rock was thereafter calculated using the Bond's energy equation, by equating the energy equations of the two milling processes.

3.4 *Separation Studies*

(a) Bench Scale Flotation Tests

Bench Scale Flotation tests were carried out on -106 +38µm, - 180 +106µm, +180 -250µm, - 106 +75µm, - 75 +45µm and - 45+38 µm size ranges obtained in 3.3.2.a after screening in order to determine the optimum flotation feed size distribution using one (1) litre Denver flotation cell.

Scoping flotation tests were carried out as follows.

a. 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 (2003) to be used.
b. Correct dosages of reagents were added with the pH adjusted to the desired reading using either sodium carbonate or calcium hydroxide.
c. 350g of ground phosphate rock was then added and conditioned for two minutes at desired percent solids (55%, 60% and 65%).
d. Make up water was added and mixed for thirty seconds.

e. Air was introduced with floated material collected until froth contains clean air bubbles.

f. Collected float materials were dried in an oven and analyzed for P_2O_5 using XRF Pan Analytical Mini PAL4 analyzer.

g. Incremental float samples were collected after every two (2) minutes

Chapter Four

4.0 Results

4.1: *Chemical and Mineralogical Analysis*

Table 4.1.1: The result of the chemical analysis carried out on Run-of-Mine material

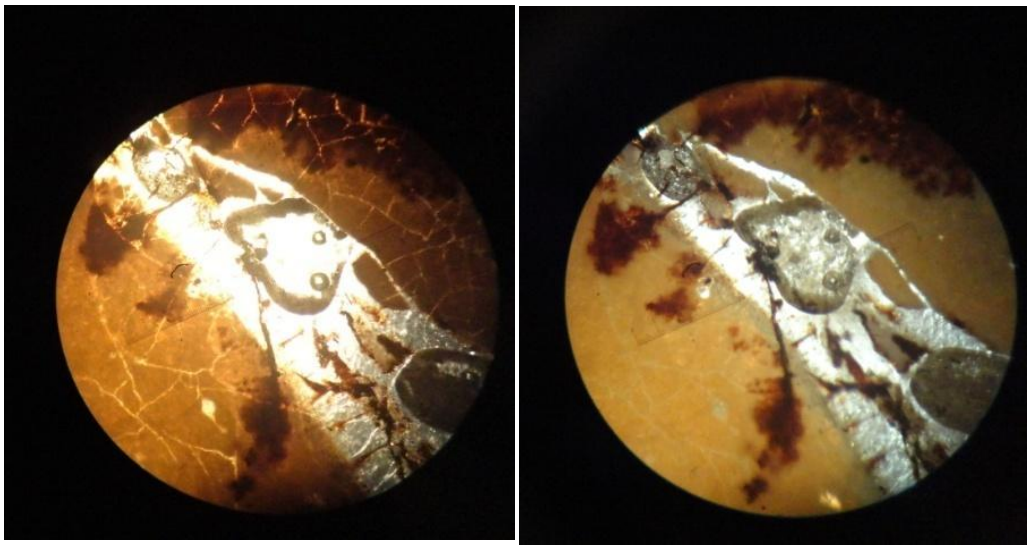
Oxide	Al_2O_3	SiO_2	P_2O_5	SO_3	CaO	MgO	TiO_2	V_2O_5	MnO	Fe_2O_3	Na_2O	K_2O	F-
%	10.50	44.30	12.10	0.14	19.10	0.73	2.44	0.11	0.34	3.91	2.42	0.34	2.24

Table 4.1.2: Chemical Analysis of Sorted Sokoto Phosphate Rock

Oxide	Al_2O_3	SiO_3	P_2O_5	SO_3	CaO	MgO	TiO_2	V_2O_5	MnO	Fe_2O_3	In_2O_3	BaO	Nd_2O_3	HgO	LOI
%	3.60	14.20	21.70	0.36	44.31	0.45	0.83	0.05	0.59	6.19	0.41	0.22	0.09	0.68	6.32

Table 4.1.3: Mineralogical composition of the phosphate rock as determined from XRD

	Mineral name	Compound name	Empirical formula	Chemical formula
1	Hydroxylapatite(a)	Calcium-Phosphate-Hydroxide	$Ca_5HO_{13}P_3$	$Ca_5(PO_4)_3(OH)$
2	Carbonate-flourapatite	Calcium-Phosphate-Flouride-Carbonate	$CCa_{10}F_2O_{23}P_6$	$Ca_{10}(PO)_6F_2(CO_3)$
3	Calcite	Calcium-Carbonate	$CCaO_3$	$Ca(CO_3)$
4	Flourapatite	Calcium-Iron-Phosphate(v) Flouride-Hydroxide	$Ca_{10}F_2FeHO_{24}P_6$	$(Ca_{10}Fe)(PO_4)_6(F_2(OH))$
5	Quartz	Silicon-Oxide	O_2Si	SiO_2
6	Hydroxylapatite(b)	Calcium-Oxide-Phosphate-Deuterioxide	$Ca_5DO_{13}P_3$	$Ca_5(PO_4)_3O(OD)$



A. **Plane Polarized LightB. Cross Polarized Light**

Plate 4.1.1: Photomicographs of Sokoto Phosphate Rock Thin Slide (X400)

Table 4.1.4: Size - Assay Analysis of Sokoto Phosphate Rock Head Sample

Size Range (µm)	**Wt (g)**	**Wt% Retained**	**Assay P_2O_5 (%)**	**Units**	**Dist (%P_2O_5)**	**Sieve Size (µm)**	**Cum. Wt% Passing**	**Cum. Dist Passing (%P_2O_5)**
+40000	243.7	12.05	13.20	32.17	7.28	40000	87.93	92.72
-40000 +25400	272.2	13.45	28.20	76.76	17.36	25400	74.48	75.36
–25400 +19050	129.6	6.41	25.90	33.57	7.60	19050	68.08	67.77
–19050 +12700	653.9	32.32	26.90	175.90	39.79	12700	35.76	27.98
–12700 +9050	328.8	16.25	20.20	66.42	15.02	9050	19.51	12.96
–9050 +6350	151.7	7.50	18.00	27.31	6.18	6350	12.01	6.78
–6350 +4750	33.0	1.63	28.60	9.44	2.13	4750	10.38	4.65
–4750 +2000	75.9	3.75	12.10	9.18	2.08	2000	6.63	2.57
–2000 +1400	5.1	0.25	9.91	0.51	0.11	1400	6.38	2.46
– 1400 +710	33.5	1.66	8.86	2.97	0.67	710	4.72	1.78
– 710+425	21.3	1.05	8.04	1.71	0.38	425	3.67	1.4
-425	74.3	3.67	8.30	6.17	1.40			
Total	**2023.0**	**99.99**	**-**	**442.11**	**100.00**			

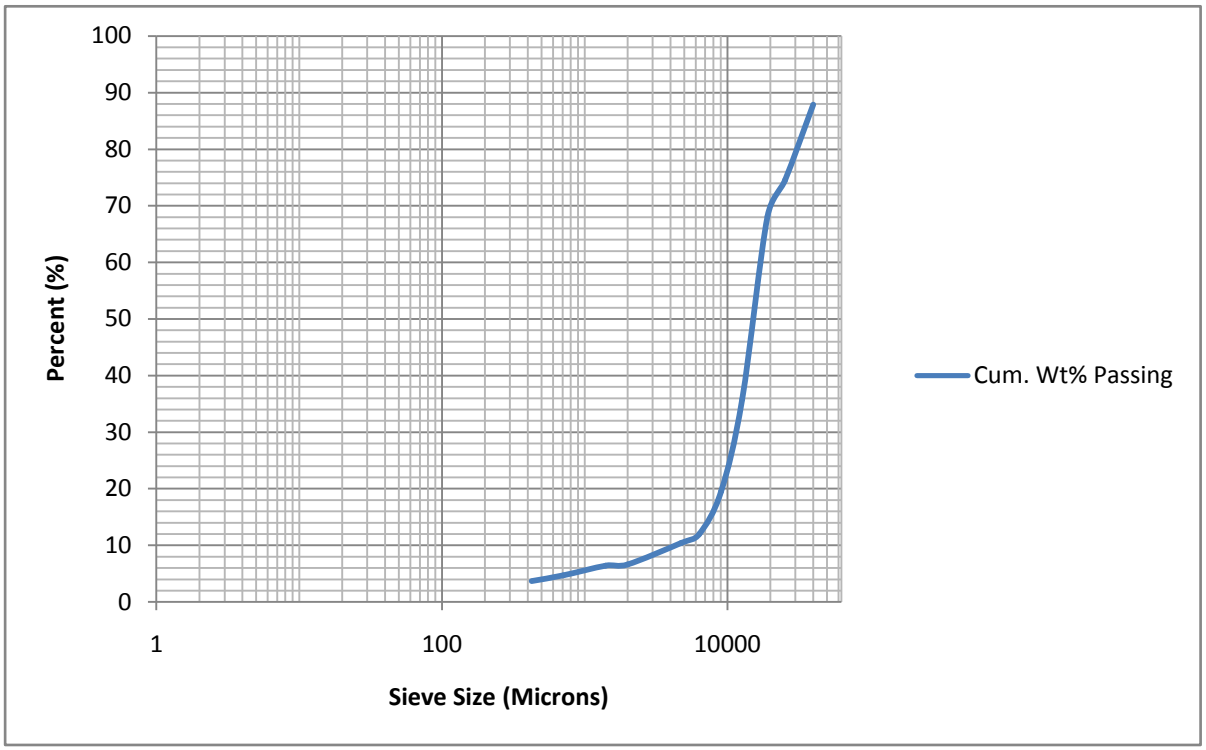


Figure 4.1.2: Cumulative Weight % Passing Semi-log Graph of Sokoto Phosphate Rock Sample

Using Gaudin-Schuhmann equation where:

80% Cum Passing Size = (80% / %passing size 1)^{2}x size 1 *(4.1)*

If, 25400µm = 74.48%

Then 25400µm (80/74.48)^2= 29,304.5µm

This result calculated corresponds with the value obtained in plotted Figure 4.1.2.

4.2: *Work Index Determination Result*

Table 4.2.1: Size Analysis of Phosphate Rock sample as Feed Material for work index test

Size Range (µm)	Wt (g)	Wt% Retained	Sieve Size (µm)	Cum. Wt% Retained	Cum. Wt% Passing
+12700	5.5	1.37	12700	1.37	98.63
– 12700+9050	13.4	3.33	9050	4.70	95.29
–9050 +6350	77.2	19.21	6350	23.92	76.08
– 6350 +4750	92.2	22.95	4750	46.87	53.13
– 4750 +2000	104.5	26.01	2000	72.87	27.12
– 2000 +1700	9.6	2.39	1700	75.26	24.73
– 1700 +1180	5.8	1.44	1180	76.71	23.29
– 1180 +850	32.7	8.14	850	84.84	15.15
– 850 +710	0.3	0.07	710	84.92	15.08
– 710 +425	19.1	4.75	425	89.67	10.32
– 425+212	18.6	4.63	212	94.30	5.70
– 212+150	8.8	2.19	150	96.49	3.51
– 150+104	8.5	2.12	104	98.61	1.39
–104	5.6	1.39		100.00	
Total	401.8	99.99			

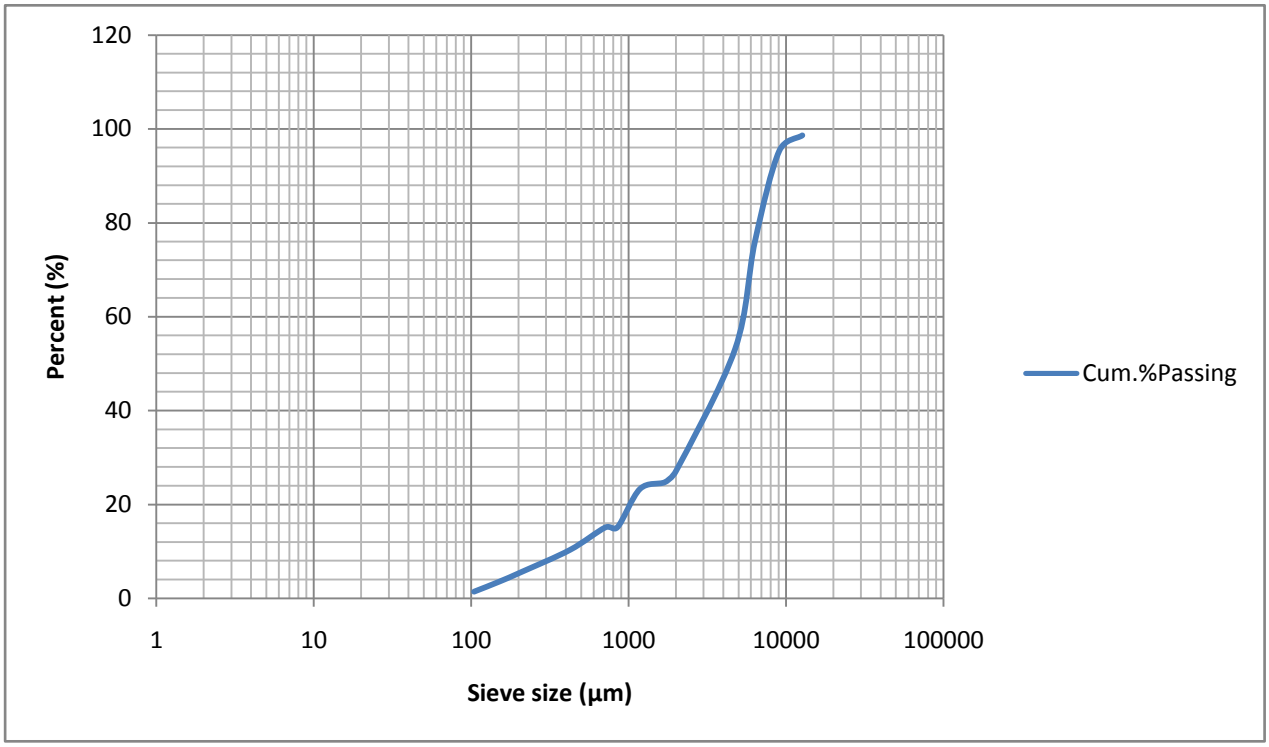


Figure 4.2.1: Cumulative Weight % Passing Semi-log Graph of Sokoto Phosphate Rock Feed for Work Index Determination Test.

Using Gaudin-Schuhmann equation (4.1),

If, 6350µm = 76.08%

Then 6350µm $(80/76.08)^2$= 7021.12µm

This result calculated corresponds with the value obtained in plotted Figure 4.2.1.

Table 4.2.2: Size Analysis of Phosphate sample product after 20 minutes grinding

Size Range (µm)	Wt(g)	Wt% Retained	Sieve Size (µm)	Cum. Wt% Retained	Cum. Wt% Passing
+9500	5.9	2.9	9500	2.9	96.9
– 9500 +6350	22.8	11.4	6350	14.3	85.5
– 6350 +4760	18.5	9.2	4760	23.5	76.3
– 4760 +2000	22.5	11.2	2000	34.7	65.1
–2000 +1400	0.4	0.2	1400	34.9	64.9
– 1400+850	5.2	2.6	850	37.5	62.3
– 850 +710	0	0.0	710	37.5	62.3
– 710 +425	3.4	1.7	425	39.2	60.6
– 425 +355	3.4	1.7	355	40.9	58.9
– 355+250	8.4	4.2	250	45.1	54.7
– 250+180	7.4	3.7	180	48.8	51.0
– 180+106	34.4	17.2	106	66.0	33.8
– 106+63	23.3	11.6	63	77.6	22.2
– 63+38	36.0	18.0	38	95.6	4.2
– 38	8.4	4.2	-	99.8	0
Total	200.0	99.8	-	-	-

If, 4760μm = 76.30%

Then 4760μm $(80/76.30)^2$= 5233.14μm

This result calculated corresponds with the value obtained in plotted Figure 4.2.3.

Table 4.2.3: Size Analysis of Phosphate Rock product after 10minutes Grinding

Size Range (µm)	Wt (g)	Wt% Retained	Sieve Size (µm)	Cum. Wt% Retained	Cum. Wt% Passing
+9500	2.5	1.25	9500	1.25	98.75
– 9500+6350	35.2	17.60	6350	18.85	81.15
– 6350+4760	27.4	13.70	4760	32.55	67.45
– 4760+2000	41.3	20.65	2000	53.20	46.8
– 2000 +1700	1.7	0.85	1700	54.05	45.95
– 1700 +1400	0.2	0.1	1400	54.15	45.85
– 1400 +850	0	0	850	54.15	45.85
– 850 +710	0	0	710	54.15	45.85
– 710 +425	22.3	11.15	425	65.3	34.7
– 425+355	6.3	3.15	355	68.45	31.55
– 355+250	8.7	4.35	250	72.8	27.2
– 250+180	5.0	2.50	180	75.3	24.7
– 180+106	16.8	8.40	106	83.7	16.3
– 106+63	8.9	4.45	63	88.15	11.85
– 63+38	16.6	8.30	38	96.45	3.55
– 38	7.1	3.55	-	100.00	-
Total	200	100	-	-	-

If, 4760µm = 67.45%

Then 4760μm $(80/67.45)^2$= 6696.34μm

Using interpolation technique, the 80% passing size (μm) is given as follows:

80% Passing size = LSS + (CSH-80) / (CSH-CSL)*(HSS-LSS)

Where:

LSS = Sieve Size corresponding to the immediate cumulative wt% passing reading below 80%

CSH = Immediate cumulative wt% passing figure higher than 80%

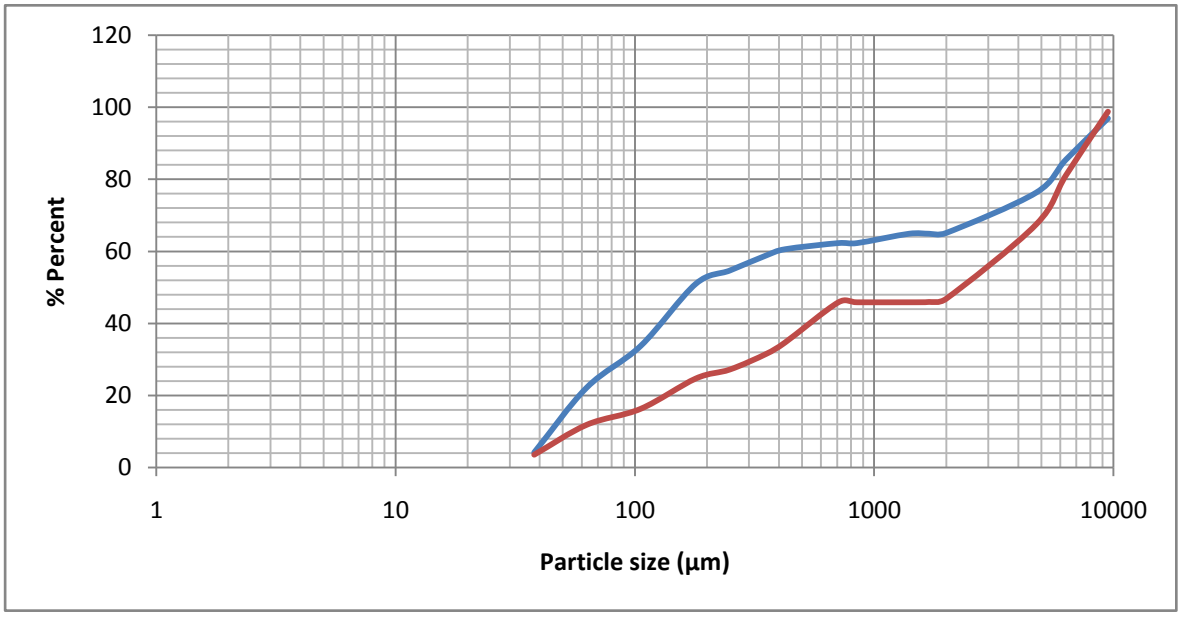
CSL = Immediate cumulative wt% passing figure lower than 80%

HSS = Sieve size corresponding to the immediate cumulative wt% passing reading higher than 80%

=4760+[(80-67.45)/(81.15-67.45)](6350-4760)

=6216.53

The result calculated using the interpolation technique corresponds with the value obtained in plotted Figure 4.2.2.



Figures 4.2.2: Gives the Cum. % Passing Particle Size Analysis of Phosphate Rock Product after 10 and 20minutes of grinding.

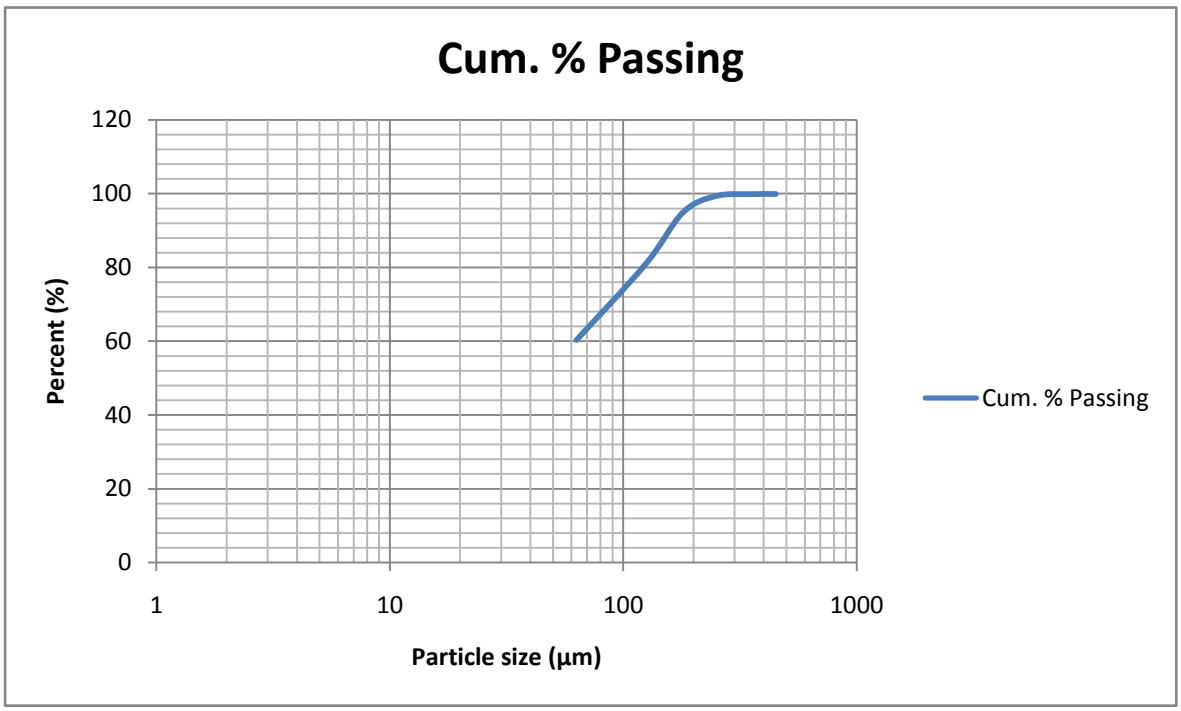
Table 4.2.4: Size Analysis of Magnetite (Reference) Feed Material for work index test

Size Range (μm)	Wt (g)	Wt% Retained	Sieve Size (μm)	Cum. Wt% Retained	Cum. Wt% Passing
+1700	0.0	0.00	1700	0.00	100.00
– 1700 +1180	1.0	0.58	1180	0.58	99.42
– 1180 +850	19.0	11.05	850	11.63	88.37
– 850 +710	1.0	0.58	710	12.21	87.79
– 710 +425	88.0	51.16	425	63.37	36.63
– 425 +212	57.0	33.14	212	96.51	3.49
– 212 +150	5.0	2.91	150	99.42	0.58
– 150 +63	1.0	0.58	63	100.00	0.00
– 63	0.0	0.00		100.00	
Total	172.0	100.00	-	-	-

If, 710μm = 87.79%

Then 710μm $(80/87.79)^2$= 589.66μm

This result calculated corresponds with the value obtained in plotted Figure 4.2.3.



Figures 4.2.3: Cum. % Passing Particle Size Analysis of magnetite ore feed after 10 and of grinding.

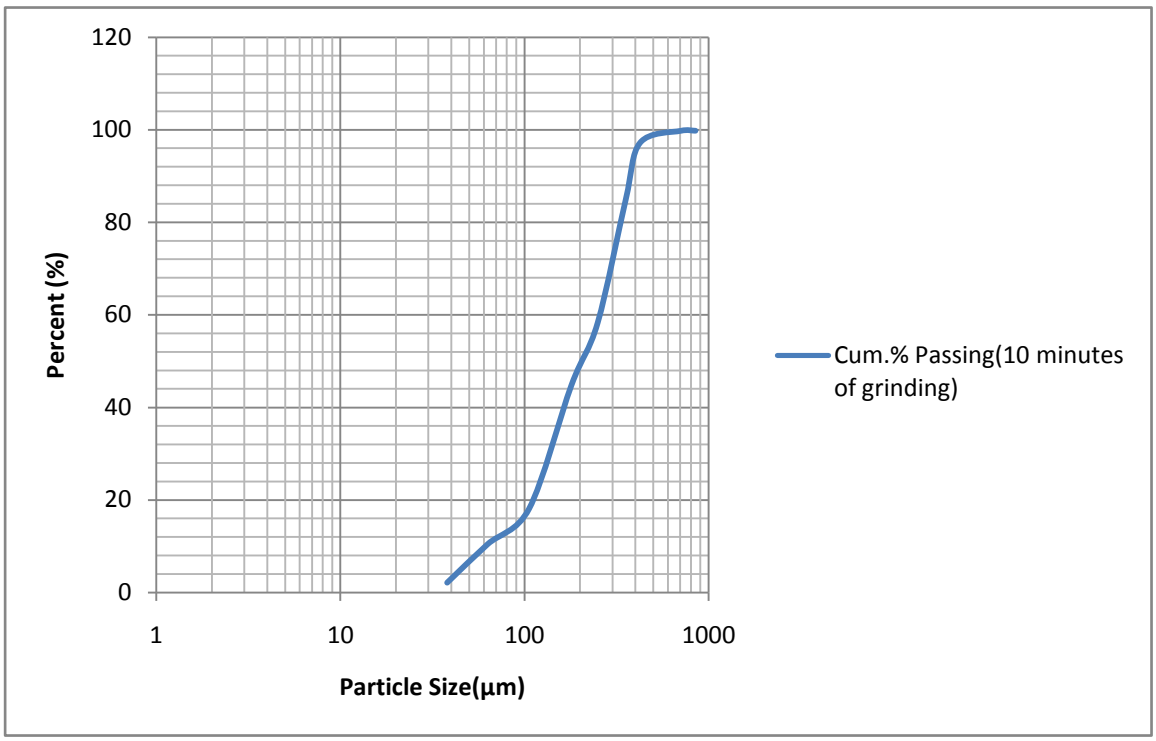
Table 4.2.5: Size Analysis of Magnetite product after 10minutes Grinding

Size Range (μm)	Wt% Retained	Wt%	Sieve Size (μm)	Cum. Wt% Retained	Cum. Wt% Passing
+850	0.2	0.1	850	0.1	99.8
– 850+710	0.0	0.0	710	0.1	99.8
– 710+425	5.2	2.6	425	2.7	97.2
– 425+355	24.6	12.3	355	15.0	84.9
– 355+250	53.2	26.6	250	41.6	58.3
– 250+180	27.1	13.5	180	55.1	44.8
– 180+106	53.4	26.7	106	81.8	18.1
– 106 +63	15.4	7.7	63	89.5	10.4
– 63+38	16.7	8.3	38	97.8	2.1
– 38	4.2	2.1	-	99.9	-
Total	200.0	99.9	-	-	-

If, 355μm = 84.9%

Then 355μm $(80/84.9)^2$= 315.20μm

This result calculated corresponds with the value obtained in plotted Figure 4.2.4.



Figures 4.2.4: Cum. % Passing Particle Size Analysis of Magnetite Product after 10 minutes of grinding.

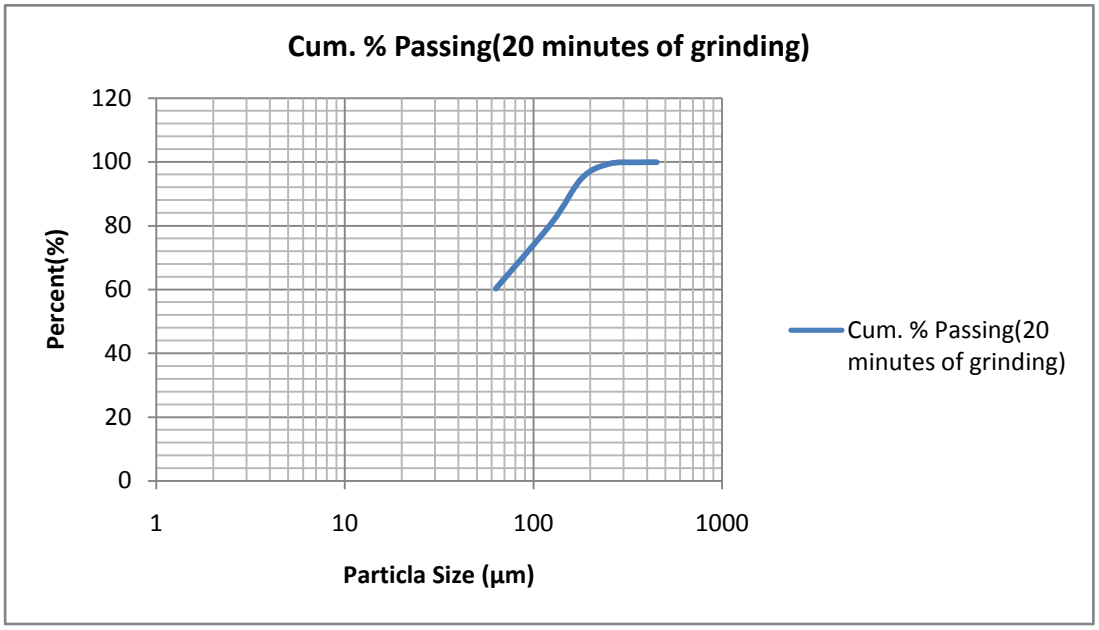
Table 4.2.6: Sieve Analysis of Magnetite product after 20minutes Grinding

Size Range (µm)	Wt(g)	Wt% Retained	Sieve Size (µm)	Cum. Wt% Retained	Cum. Wt% Passing
+450	0.3	0.08	450	0.08	99.93
– 450+355	0.1	0.03	355	0.10	99.90
– 355+250	2.0	0.50	250	0.60	99.40
– 250+180	18.0	4.50	180	5.10	94.90
– 180+125	55.6	13.90	125	19.00	81.00
– 125+63	82.8	20.70	63	39.70	60.30
– 63	241.2	60.30	-	-	-
Total	400.0	100.00	-	-	-

If, 125µm = 81.00%

Then 125µm $(80/81)^2$= 121.95µm

This result calculated corresponds with the value obtained in plotted Figure 4.2.5.



Figures 4.2.5: Gives the Cum. % Passing Particle Size Analysis of Magnetite Product after 20 minutes of grinding.

The Work index is the comminutionparameter which expresses the resistance of material to crushing and grinding; it is the kilowatt hour per ton required to reduce the material from theoretically infinite feed size to 80% passing 100 (Wills and Napier-Munn, 2006).

From Berry and Bruce (1966) using Bond's law in Yaro and Thomas (2001) the work index of an ore can be determined by comparing the grindability:

$$Wr=Wt=Wir10(1/\sqrt{Pr}-1/\sqrt{Fr})=Wit10(1/\sqrt{Pt}-1/\sqrt{Pt}) \quad (4.2)$$

$$Hence,\ Wit=Wir(1/\sqrt{Pr}-1/\sqrt{Fr})/(1/\sqrt{Pt}-1/\sqrt{Ft}). \quad (4.3)$$

Where Wr=Work input of reference ore; Wt=Work input of test ore;

Wir=Work index of reference ore; Wit=Work index of test ore

Pr=80% of Product reference ore passes; Pt=80% of Product test ore passes;

Fr=80% of feed reference ore passes; Ft=80% of feed test ore passes

For 10minutes

Wit= 15.9{(1/√315.20 – 1/√589.66)}/ {(1/√6216.53- 1/√7021.12)}

Wit = 15.9 kwh/t x 0.0151/0.00075= 320.12kwh/t

But 1hr = 60 minutes, therefore a ground of 10 minutes will be equaled to:

10/60 x 1hr= 0.167

Therefore, 320.12kwhr/ton x 0.16= 51.22kwh/t

For 20 minutes grind

Wit= 15.9{(10/√121.95 – 10/√589.66)}/ {(10/√6696.34 - 10/√7021.12)}

Wit = 15.9 kwh/t x 0.049/0.002= 389kwh/t

But 1hr = 60 minutes, therefore a ground of 10 minutes will be equaled to= 10/60 x 1hr= 0.167

Therefore, 389kwhr/tonne x 0.16= 62.24kwh/t.

Average Wit = (51.22+62.24)/2 = (113.46/2)Kwh/t = 56.73Kwh/t

4.3: Flotation Test Results

Table 4.3.1: 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_2O_3	SiO_2	P_2O_5	SO_3	CaO	MgO	TiO_2	MnO	Fe_2O_3	In_2O_3	BaO	CeO_2	Nd_2O_3
Calcite Flota. +38µm/ HA/$_P$H6	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/$_P$ H9	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/$_P$H6	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/$_P$ H9	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/$_P$H6	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/ $_P$H6	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 4.3.2: Scoping Flotation Studies using a conditioning Pulp Density of 60% Solids, pH9, 800g/t reagent dosage and flotation pulp density of 28.5% Solids

Fractions / Parameter	**Reagent S-9849 Promoter(Alkyl Hydroxamates)**								
	- 106 +38 µm			**-180+106µm**			**-250+180µm**		
	Wt (g)	**% P_2O_5**	**% Rec.**	**Wt (g)**	**% P_2O_5**	**% Rec.**	**Wt (g)**	**% P_2O_5**	**% Rec.**
Floats	220.2	19.4	61.5	89.4	21.7	25.0	72.1	21.2	19.4
Tailings	129.8	20.6	38.5	260.6	22.3	75.0	277.9	22.9	80.6
Feed	350.0	19.8	100	350.0	22.1	100	350.0	22.5	100
Recovery (%):[(Cc/Ff)*100]									

Table 4.3.3: Scoping Flotation Studies using a conditioning Pulp Density of 60% Solids, pH9, 800g/t reagent dosage for AERO 704 Promoter (Fatty Acid) and flotation pulp density of 28.5% Solids.

Fractions / Parameter	**AERO®704 Promoter (Distilled Tall-oil Fatty Acids)**								
	- 106 +38 µm			**-180+106µm**			**-250+180µm**		
	Wt (g)	**% P_2O_5**	**% Rec**	**Wt (g)**	**% P_2O_5**	**% Rec.**	**Wt (g)**	**% P_2O_5**	**% Rec.**
Floats	259.8	19.9	76.2	158.2	20.4	43.2	91.5	19.4	24.1
Tailings	74.2	21.7	23.8	191.8	22.1	56.8	258.5	21.7	75.9
Feed	350.0	19.4	100	350.0	21.3	100	350.0	21.1	100
(%)Rec.: [(Cc/Ff)*100]									

Table 4.3.4: 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 / Parameter	**AERO 704 (Fatty Acid)**			**S-9849Promoter(Alkyl Hydroxamates)**			**Diesel / AERO 704 (Fatty Acid)**		
	Wt (g)	**% P_2O_5**	**% Rec.**	**Wt (g)**	**% P_2O_5**	**% Rec.**	**Wt (g)**	**% P_2O_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

Table 4.3.5: Floatation 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)	% P_2O_5	% Rec.	Wt (g)	% P_2O_5	% Rec.	Wt (g)	% P_2O_5	% Rec.	Wt (g)	% P_2O_5	% Rec.
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

Table4.3.6: Floatation Test using 350g of - 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 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

Table 4.3.7: Calcite Flotation at pH9 using Melamine as collector, Dextrin as depressant for phosphate rock and MBIC as frother.

Fraction / Parameter	**- 106+75 (µm)**		**% Rec.**	**– 75+45 (µm)**		**% Rec.**	**– 45+38 (µm)**		**% Rec.**
	Wt (g)	**% P_2O_5**		**Wt (g)**	**% P_2O_5**		**Wt (g)**	**% P_2O_5**	
Float	46.8	25.4	13.0	108.8	23.3	30.4	17.5	22.1	4.7
Tailings	303.2	26.2	87.0	241.2	24.0	69.6	332.5	23.3	95.3
Feed	350.0	26.1	100	350.0	23.8	100	350.0	23.2	100

Table 4.3.8: Results of Incremental Method Flotation Test using 800g/t of AERO 704 (Fatty Acid) at 25% solids and conditioning pulp density of 60% solids and pH9

Fraction / Parameter	**-106+38 (µm)**			**Unit (g)**	**Recovery (%)**	**Cum. Grade (%)**	**Cum. Recovery (%)**
	Wt (g)	**%Wt**	**%Assay (P_2O_5)**				
Float 1	144.1	45.47	23.0	33.14	24.6	23.0	24.6
Float 2	84.1	26.54	22.3	18.75	13.9	22.7	38.5
Float 3	33.5	10.57	22.1	7.40	5.5	22.6	44.0
Float 4	15.5	4.89	22.6	3.50	2.6	22.6	46.6
Float 5	13.2	4.17	23.1	3.05	2.3	22.7	48.9
Float 6	26.5	8.36	21.4	5.67	4.2	22.6	53.1
Sub-Total	316.9	100	22.6	71.51	53.0		-
Total Float	316.9	52.82	22.6	71.51	53.0		-
Tailings	283.1	47.18	22.4	63.41	47.0		
Feed	600.0	100	22.5	134.92	100.0		
%Recovery	**T-Float**	53.0		-	-		
	Tails	47.0		-	-		

Chapter Five

5.0. Discussion of Results

5.1. Chemical Analysis of Sokoto Phosphate Rock

Tables4.1.1 and 4.1.2 respectively show the chemical analysis of the run-of-mine Sokoto phosphate rock material and that of the sorted out phosphate rock nodules. The analysis revealed that the Sokoto Phosphate rock Run-of-Mine Material contains 44.3%SiO2, 12.1% P_2O_5, 19.1%CaO, 3.9%Fe_2O_3, 2.4%Na_2O, 2.2%F^- and 10.5%Al_2O_3 while the sorted out nodules contain14.2%SiO_2, 21.7% P_2O_5, 44.31%CaO, 6.19%Fe_2O_3, and 3.60% Al_2O_3on the average as the major components.

Figures5.1.1 and 5.1.2 respectively show the pictorial bar graphs of the % assay of the chemical analysis of the Run-of-Mine Sokoto phosphate rock material and that of the sorted Phosphate rock nodules. From the graphs, while silica has the highest percentage of 44.3 in the Run-of-mine material followed by lime at 19.1% and P_2O_5 at 12.1%, the sorted material contains lime as the highest component with a percentage of 44.31% followed by phosphate with 21.7%, silica 14.2%, iron hematite 6.19% and alumina with 3.61%.

The result of the chemical analysis shows that the Sokoto phosphate rock material can be upgraded by sorting from 12.1%P_2O_5 to 21.7%P_2O_5. The result also shows that phosphate rock even after sorting, falls in the low grade phosphate rock class because its content falls within the ranges of 14.6 to 29%P_2O_5 as stated in Weiss, 1985 and a high iron content of 6.19% compared to that of Idaho (0.5 to 1.57%Fe_2O_3), North Carolina (1.30%) and Togo (1.20%); the lime content of 44.31% compared favourably with the aforementioned phosphate mineral deposits.

The alumina content of the Sokoto phosphate of 3.61% is higher compared to that of aforementioned United States and that of Togo phosphate mineral deposits. The silica content of 14.2% of the Sokoto phosphate mineral ore is higher than that of Togo, Idaho high grade phosphate but less than that of Idaho low grade phosphate ore as given in Table 2.5. Weiss (1985) stated that deposits with similar characteristics to that of Sokoto phosphate ore could exhibit the percentage of P_2O_5 as low as 5.8%, e.g. the Upper low grade phosphate ore deposit section found in North Carolina mine fields in United States. But this limitation of the deposit does not stop their utilization in producing low grade fertilizer and other phosphorus related products (Weiss, 1985). Hence, the Sokoto phosphate mineral ore can serve as another potential source of phosphate mineral ore for the production of fertilizer and other related phosphorus based products.

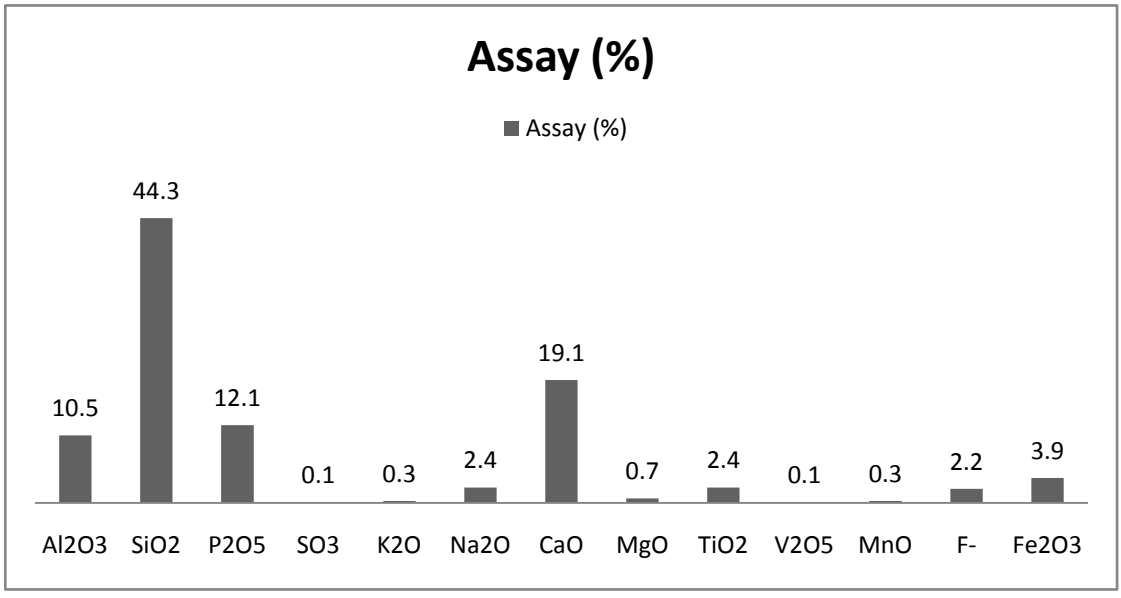


Fig.5.1.1: % Assay of Chemical Analysis of Sokoto Phosphate Rock Run-of-Mine Material

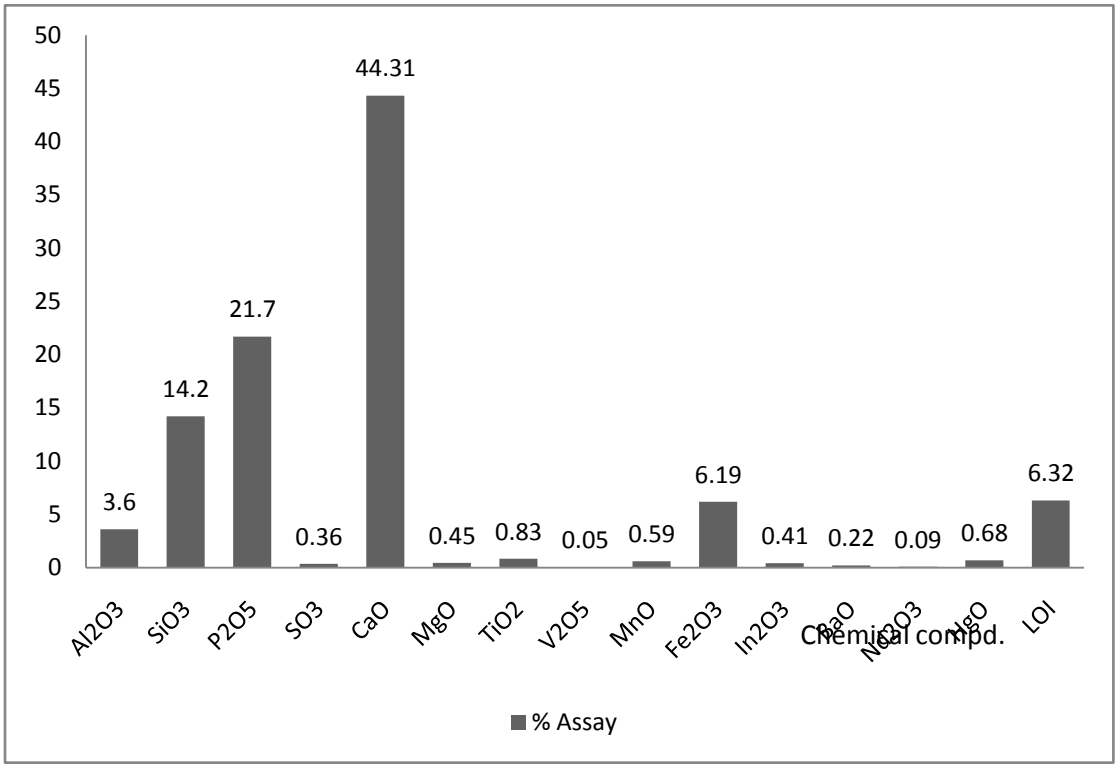


Fig.5.1.2: % Assay of the Chemical Analysis of Sorted Sokoto Phosphate Rock Head Sample

5.2. Mineralogical Analysis of Sokoto Phosphate Mineral Rock

Table4.1.3 shows the mineralogical compositions of the Sokoto phosphate rock deposit. Figures 5.2.1 and 5.2.2 show the XRD diffraction patterns of the Sokoto phosphate rock deposits while the micrographs of the thin slide of the phosphate rock are shown in plates 4.1.1.

The XRD diffraction patterns of the Sokoto phosphate rockrevealed the presence flourapatite, hydroxylapatite, calcite and quartz with the XRF result confirming Calcite as the dominant mineral (equivalent of 44.3% CaO), followed by the phosphate group minerals (equivalent of 21.7% P_2O_5), Iron group mineral (equivalent of 6.19%Fe_2O_3) and Silica (equivalent of 3.6%

SiO_2). This explains the high iron content of the Sokoto phosphate rock compared to other apatite type of phosphate mineral ores given in Table 2.5.

The micrographs show that Sokoto Phosphate rock is collophanein nature in view of the fact no mineral grain is visible even under the microscope indicating non-liberalization of associated minerals within the conventional flotation size range of +38microns.

The hydroxylapatite, fluorapatite and carbonate-fluorapatite belong to the apatite family of phosphate mineral ores which are mostly characterized by high percentage of lime with fluorapatite containg high iron content. The presence of these minerals could be attributed to the marine sedimentation of the organic-rich diatomaceous oozes of the West African shelf from bones, teeth and other animal shells.

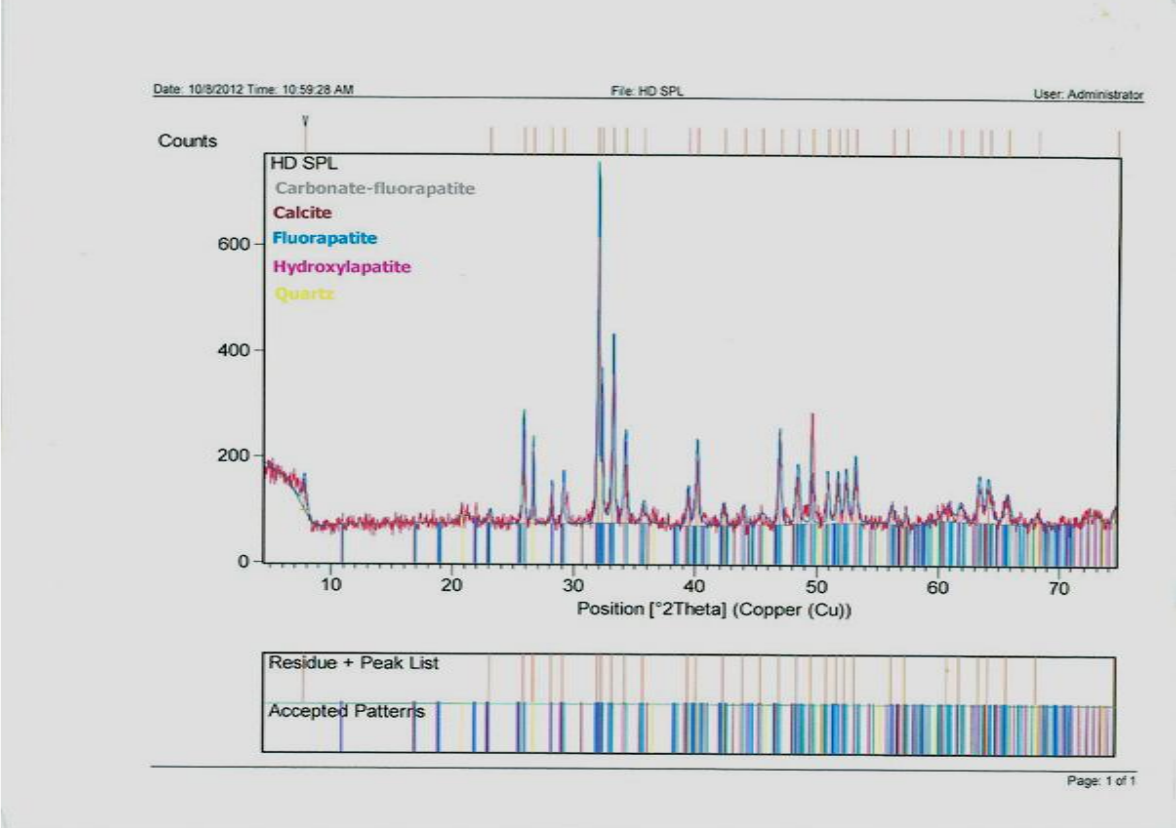


Figure 5.2.1: XRD diffraction pattern of the Sokoto phosphate rock

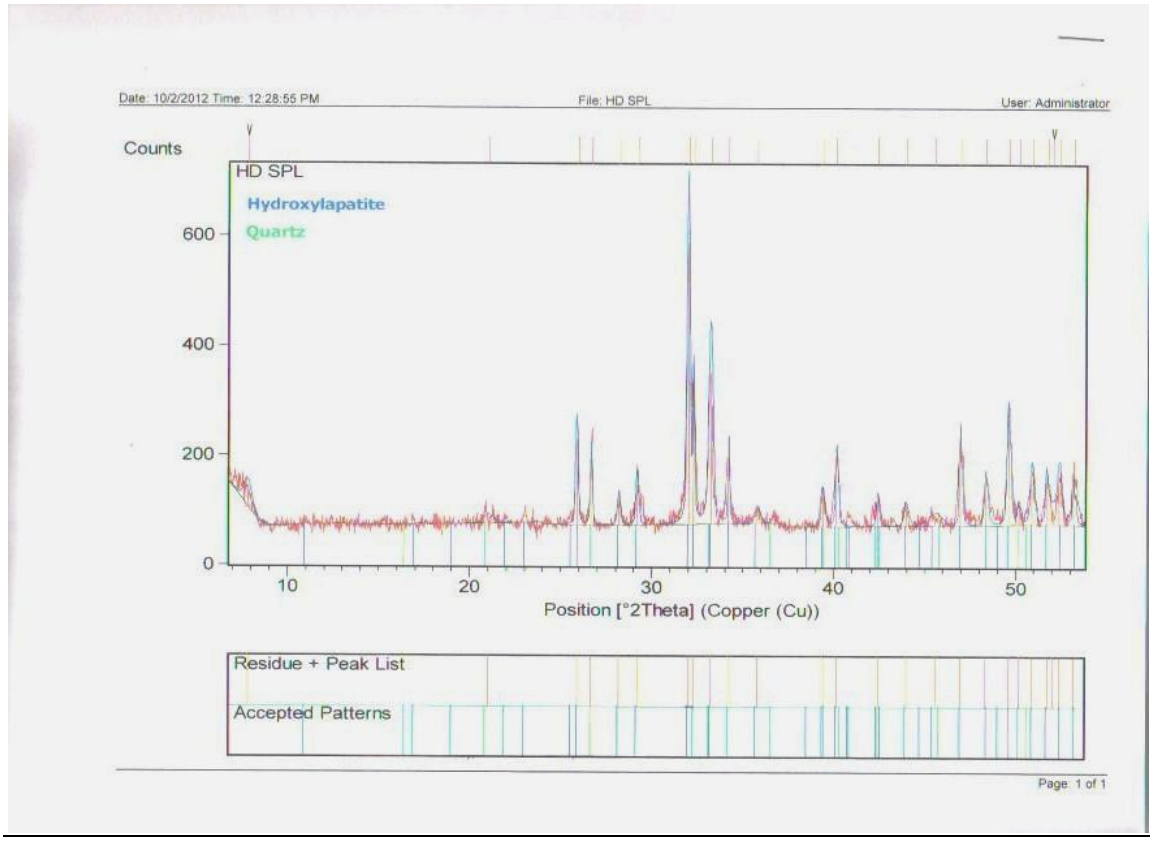


Figure 5.2.2: XRD diffraction pattern of the Sokoto phosphate rock

5.3Particle Size/ Assay Analysis of the Sokoto Phosphate Rock

Table 4.1.4shows the size-assay analysis of the Sokoto phosphate rock head sample while Figure 4.1.2 shows its cumulative weight %. The combined graphs of the size assay analysis of the Sokoto Phosphate Rock are also shown in figure 5.3.1 below.

The graphs show that the 80% cumulative passing size of the phosphate rock is 29,304microns. Scrubbing Sokoto phosphate Rock mined material using a re-enforced screen of 12,700 microns aperture lined scrubber will produce a phosphate rock grade of 24.5% P_2O_5corresponding to a recovery of 72.1% P_2O_5.

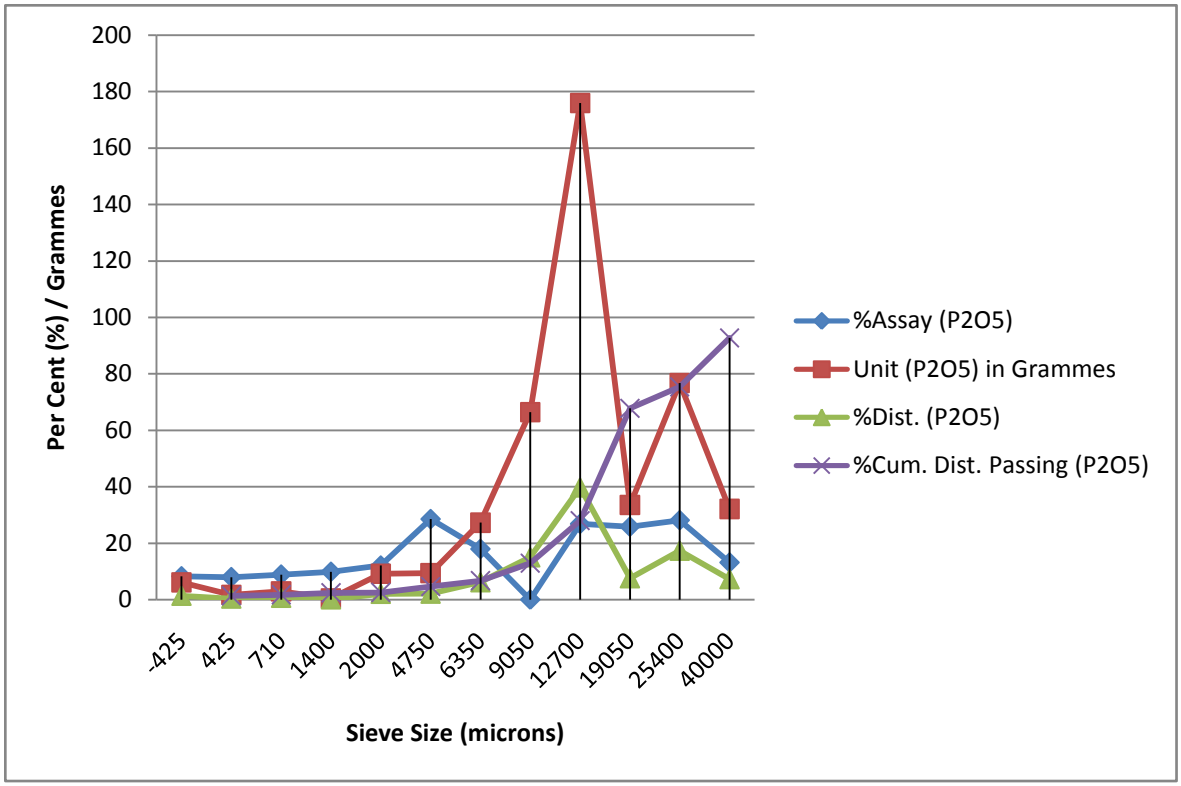


Figure 5.3.1: Graphs of Size Assay Analysis of Sokoto Phosphate RockHeadSample

5.4Work index determination:

Tables 4.2.1 – 4.2.5 give the cumulative Wt% passing of the particle sizes of the test material (Sokotophosphate rock) and a reference ore, Itakpe magnetic iron ore. From the results, the particle size fractions of the test material and the reference ores show a normal distribution pattern, indicating that the particle size fractions of the test and reference ores respond to the theories of comminution. That is, the energy consumed in size reduction is proportional to the area of new surface produced. The 80% passing particle size fraction of the feed to comminution test was 7021.12µm giving product sizes of 6216.53µm and 5233.14µm respectively for 10 and 20 minutes grinding. This gave an average work index of the as-received Sokoto phosphate rock

nodules was computed to be 56.73kwh/ton. The computed work index when compared to the work indexes of other phosphate mineral ores is above the work indexes of 10.13-13.03kwh/ton for phosphate mineral ores sighted in the literatures (Fuerstenau and Han, 2003).This could be attributed to the collophane nature of phosphate nodules that contain no visible grain boundaries indicating an association of very fine mineral grains thus requiring greater amount of energy to effect breakage.

5.5Determination of the Specific Gravity (S.g.) of Sokoto Phosphate Rock using Density Bottle Method

Sample 1:

Weight of empty bottle (w) = 20.8g

Weight of empty bottle + sample (w_1) =30.6g

Weight of empty bottle + sample + water (w_2) =91.8g

Weight of empty bottle + water (w_3) =85.3g

Using the expression Specific gravity $= (w_1-w)/[(w_3-w)-(w_2-w_1)]$

i.e. Specific gravity =(30.6-20.8)/[(85.3-20.8)-(91.8-30.6)]

=9.8/(64.5-61.2)

=9.8/3.3=2.969≈**2.97**

Sample 2:

Weight of empty bottle (w) = 16.7g

Weight of empty bottle + sample (w_1) =26.9g

Weight of empty bottle + sample + water (w_2) =84.7g

Weight of empty bottle + water (w_3) =78.1g

Using the expression Specific gravity = $(w_1-w)/[(w_3-w)-(w_2-w_1)]$

i.e. Specific gravity (s.g) =(26.9-16.7)/[(78.1-16.7)-(84.7-26.9)]

=10.2/(61.4-57.8)

=10.2/3.6

=2.833

≈**2.83**

Average Specific Gravity of the blended phosphate rock (s.g)

= (2.97+2.83)/2

=5.8/2

=2.9

The specific gravity of 2.9 on the average was obtained for the Sokoto phosphate rock. The value obtained is slightly higher compared to that of 2.65 sighted in table 2.1 but agrees with a range of 2.9 – 3.2 reported for apatite group minerals (Encyclopaedia Britanica, 2010). The slightly high value could be attributed to the presence of other impurity minerals like iron, silica etc associated with the Sokoto phosphate rock that increase its specific gravity.

5.6. Discussion of the Flotation Test Results

5.6.1 Discussion of the Flotation of Calcite and Phosphate bearing minerals of the Sokoto phosphate rock.

Table 4.3.1: 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. 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 4.3 shows 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 in Weiss, 1985. Weiss, 1985 explained that froth flotation process is the widely adoptable method in the beneficiating of phosphate mineral ores in United States as given in Table 2.6.

The average value of 22.6%P_2O_5content 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_5contents 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

as shown in Figure 2.1 where silica that transformed to $Si(OH)_4$ seems to be active at pH ranging from 4 to 12.

Figures 5.6.1 and 5.6.2 below show % assay of the chemical compounds in the products recovered from the froth flotation of Sokoto phosphate ore sample using Reagent S-9849 Promoter (Alkyl Hydroxamates) and melamine reagents respectively.

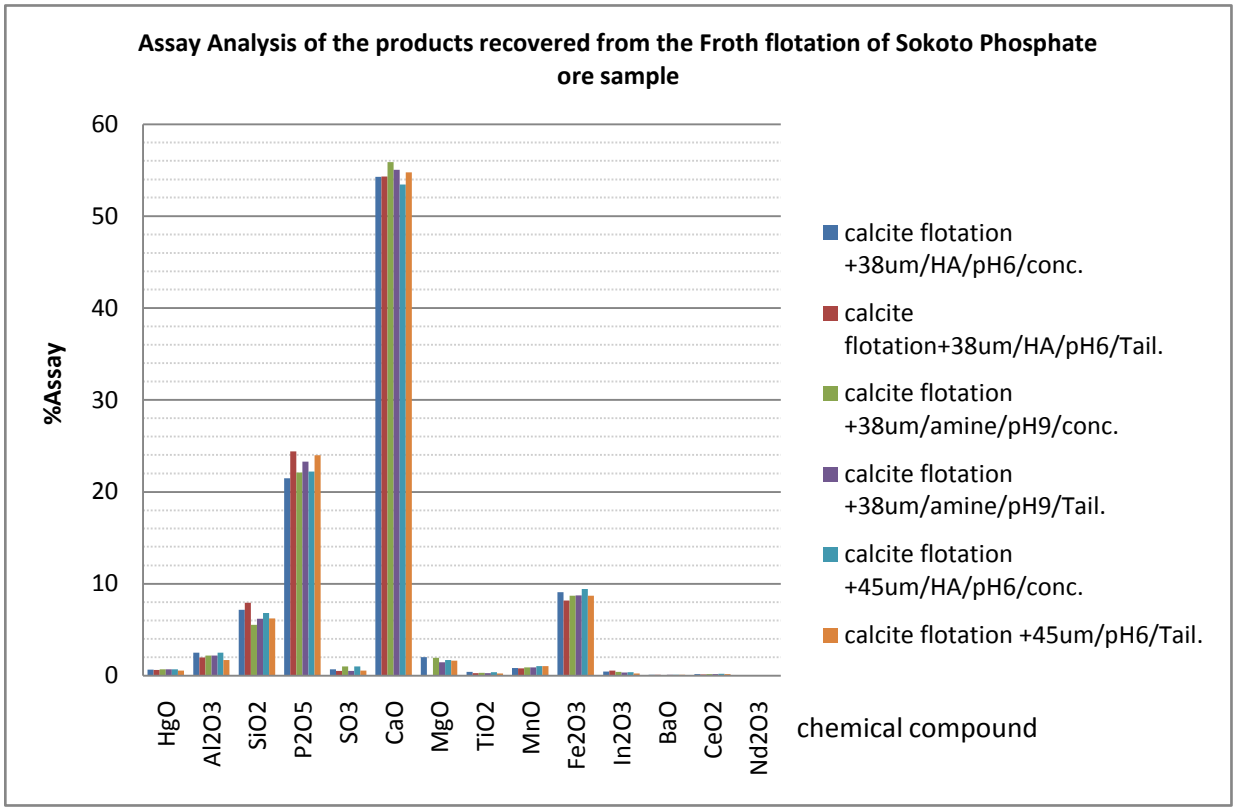


Fig.5.6.1: % assay of the chemical compound in the products recovered from the froth flotation of Sokoto phosphate Rock sample using Alkyl Hydroxamates (HA) and Melamine reagents.

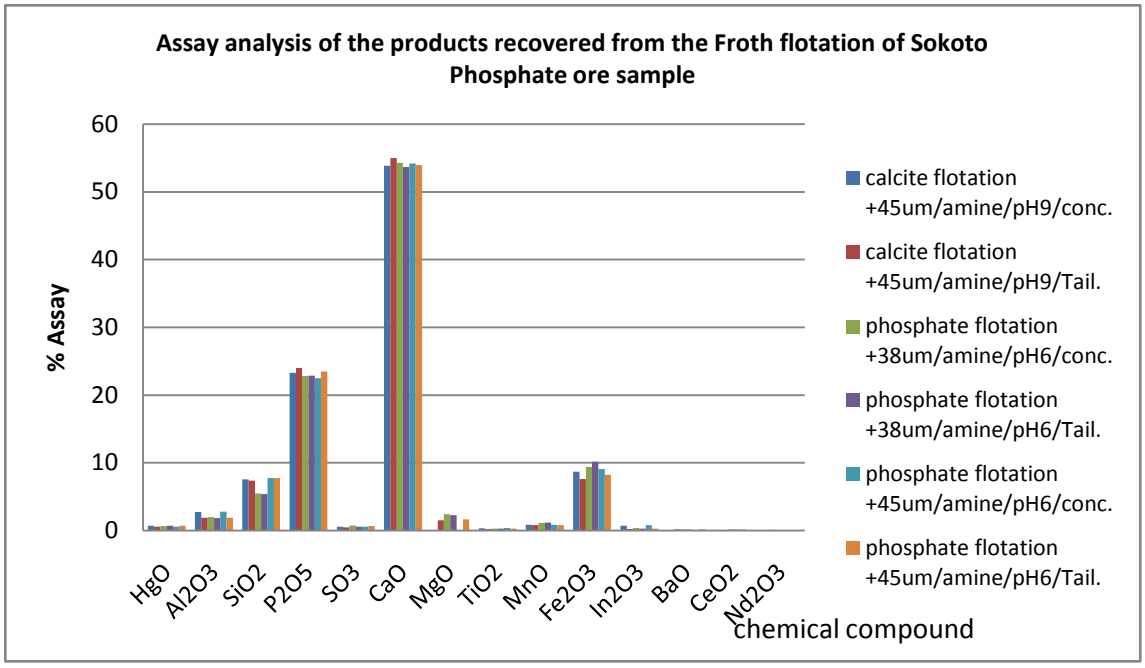


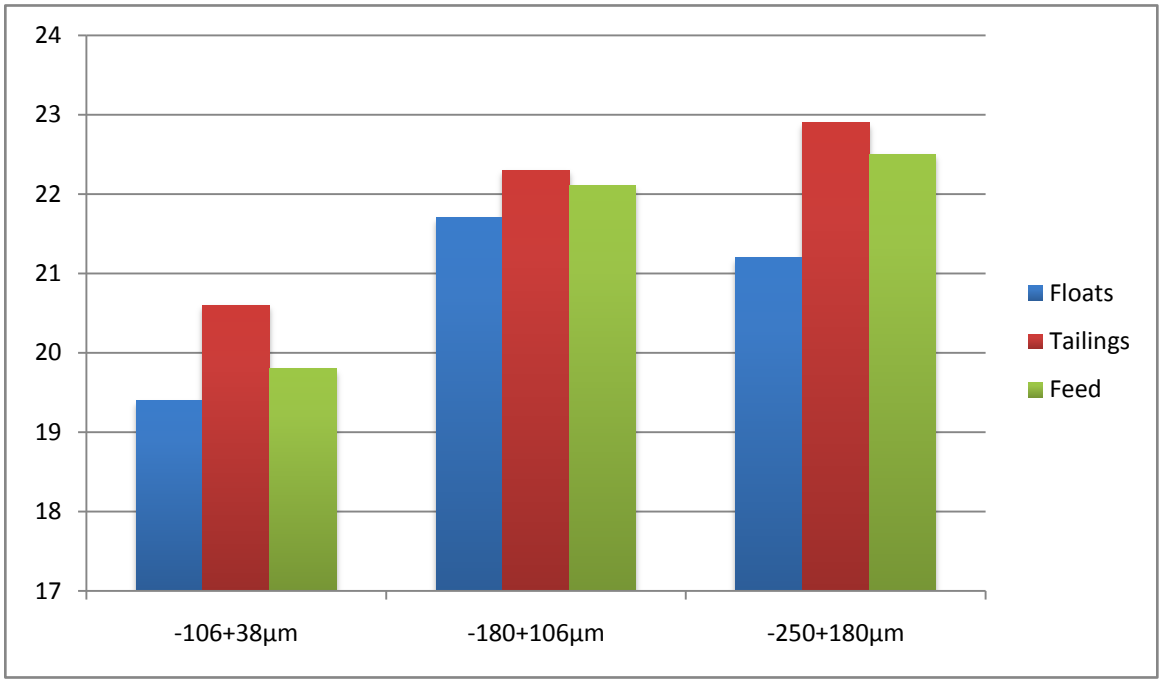
Figure 5.6.2: % assay of the chemical compound in the products recovered from the froth flotation of Sokoto phosphate ore sample using Melamine reagent only.

5.6.2Discussion on the Scoping Flotation Test Results

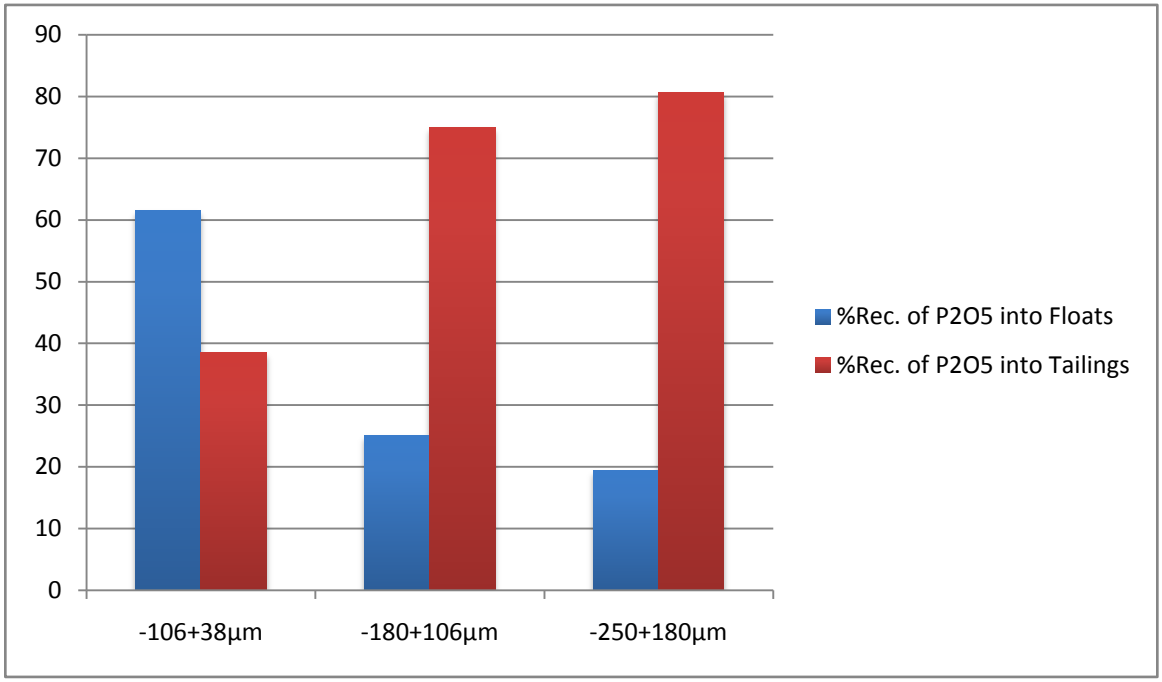
(i) Table 4.3.2gives the result of the Scoping flotation studies of the Sokoto phosphate mineral ore using a conditioning Pulp Density of 60% Solids, pH9, 800g/t Alkyl Hydroxamates dosage and flotation pulp density of 28.5% Solids. Figures 5.6.3, 5.6.4, 5.6.5 and 5.6.6 below show the % Assay of P_2O_5, %Recovery and the weights of floated phosphate sample against sieve size fractions using reagent S-9849 promoter, 800g/t, flotation pulp density of 28.5%solids, at pH9, pulp density of 60%solids.

Figure 5.6.12 shows that the scoping test did not produce a clean separation of the P_2O_5 mineral content from the associated minerals as shown by the marginal differences [19.4% for float against 20.6% for Tailings in case of +38µm size range fraction; 21.7% for floats against 22.3%

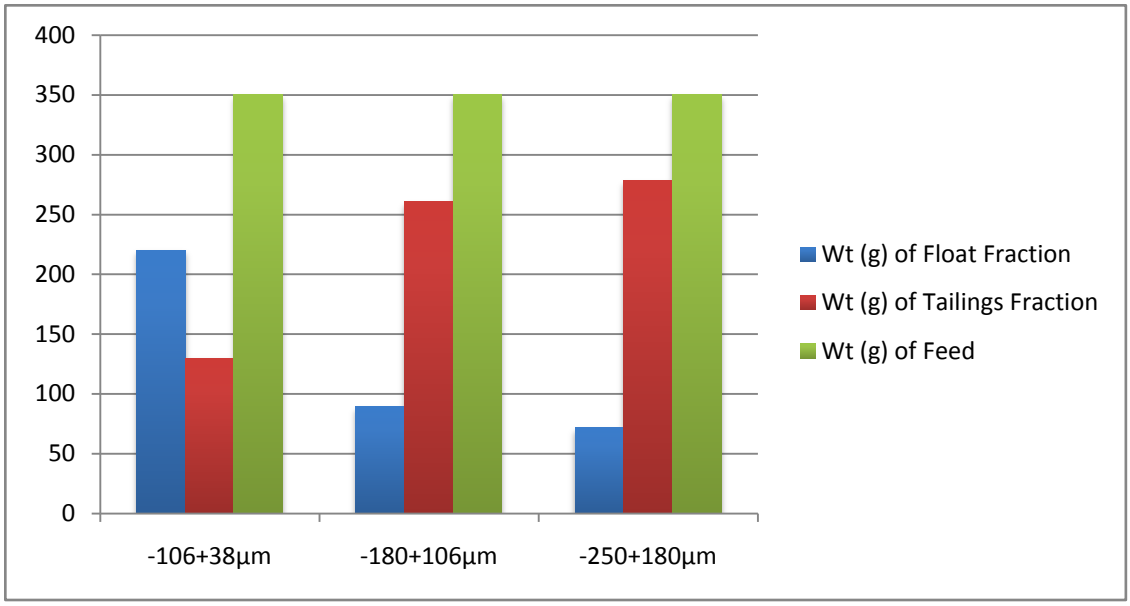
for tailings in the case of +106µm size fraction; and 21.2% for float against 22.9% for tailings in the case of +180 fraction]. This is attributable to the collophane nature of the Sokoto phosphate rock which makes liberation of associated minerals at the flotation sizes impossible. The results also showed that +38µm gave the highest recovery figure of 61.5% as against 25% and 19.4% recorded for +106µmand +180 fractions respectively. The significant difference in the recovery figures is attributable to the difficulty associated in lifting mineral particles into the float fraction by air bubbles with increasing particle size.



Figures 5.6.3: % Assay of P_2O_5 of floated phosphate sample against sieve size fractions using reagent S-9849 promoter (Alkyl Hydroxamates), 800g/t, flotation pulp density of 28.5%solids, at pH9, pulp density of 60%solids.



Figures 5.6.4: %Recovery of floated phosphate sample against sieve size fractions using reagent S-9849 promoter (Alkyl Hydroxamates), 800g/t, flotation pulp density of 28.5%solids, at pH9, pulp density of 60%solids.



Figures 5.6.5: Weights of floated phosphate sample against sieve size fractions using reagent S-9849 promoter (Alkyl Hydroxamates), 800g/t, flotation pulp density of 28.5%solids, at pH9, pulp density of 60%solids.

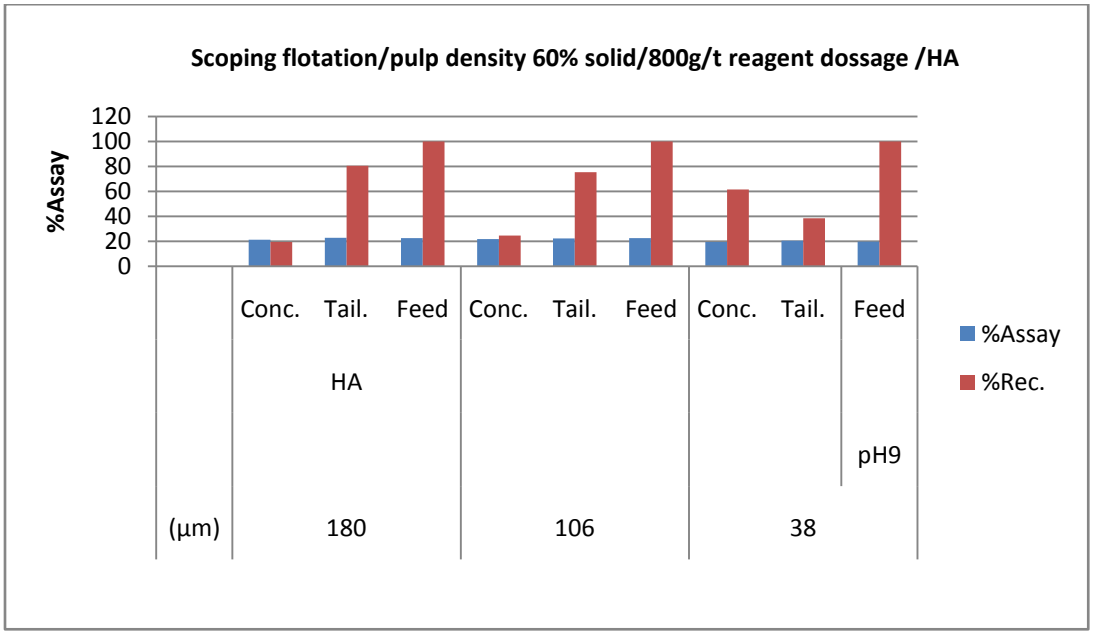


Figure 5.6.6: %Assay and Recovery of the scoping flotation/pulp density 60% solid/800g/t/HA reagent dosage at pH9 against sieve size fractions.

(iii) Table 4.3.3 and Figures 5.6.7, 5.6.8, 5.6.9 and 5.6.10 show the results of the scoping flotation studies using a conditioning Pulp Density of 60%Solids, pH9, 800g/t reagent dosage for AERO 704 Promoter (Fatty Acid) and flotation pulp density of 28.5% Solids. From the result, the +106umsizegave the highest assay content of 20.4%$P_2O_5$with a recovery of 76.2% compared to +38µm (19.9%$P_2O_5$and recovery of 43.2%) and +180um (19.4%$P_2O_5$and 24.1% recovery) in their floats (concentrates) but with no clean separation as the tailings fraction also contained similar grades with marginal differences.. This is also attributable to non-liberation of associating minerals and the difficulty in lifting mineral particles into the float fraction with increasing particle size.

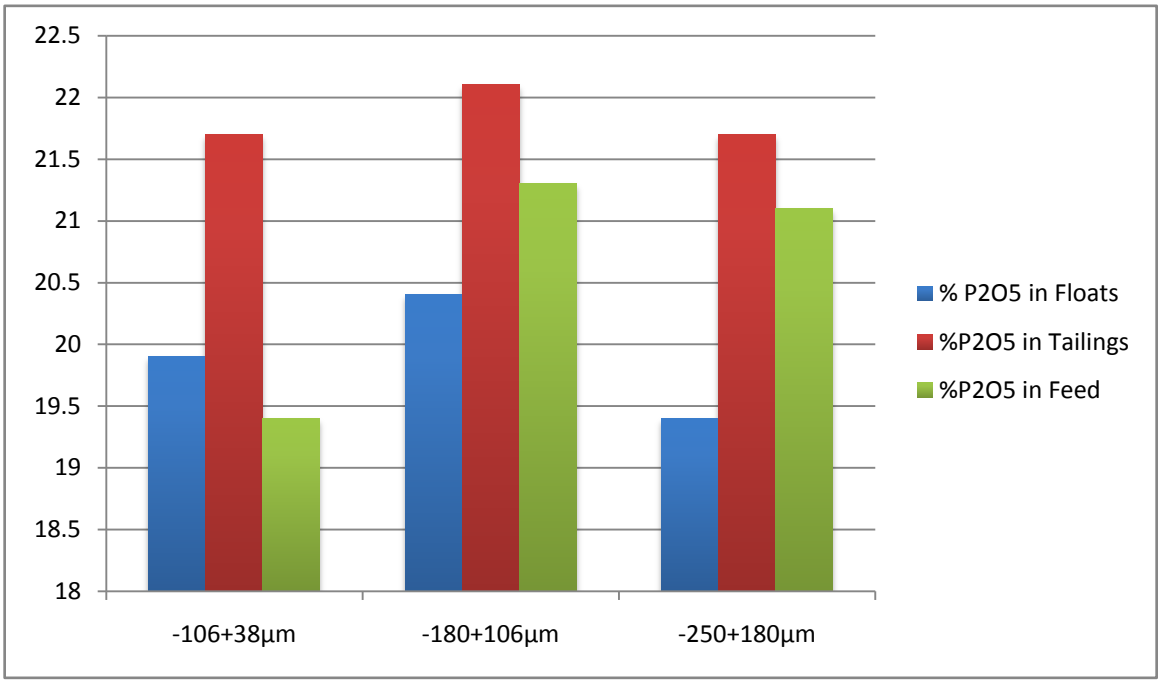


Figure 5.6.7: % Assay of $P_2O_5$of the Flotation Fractions using AERO704 Promoter

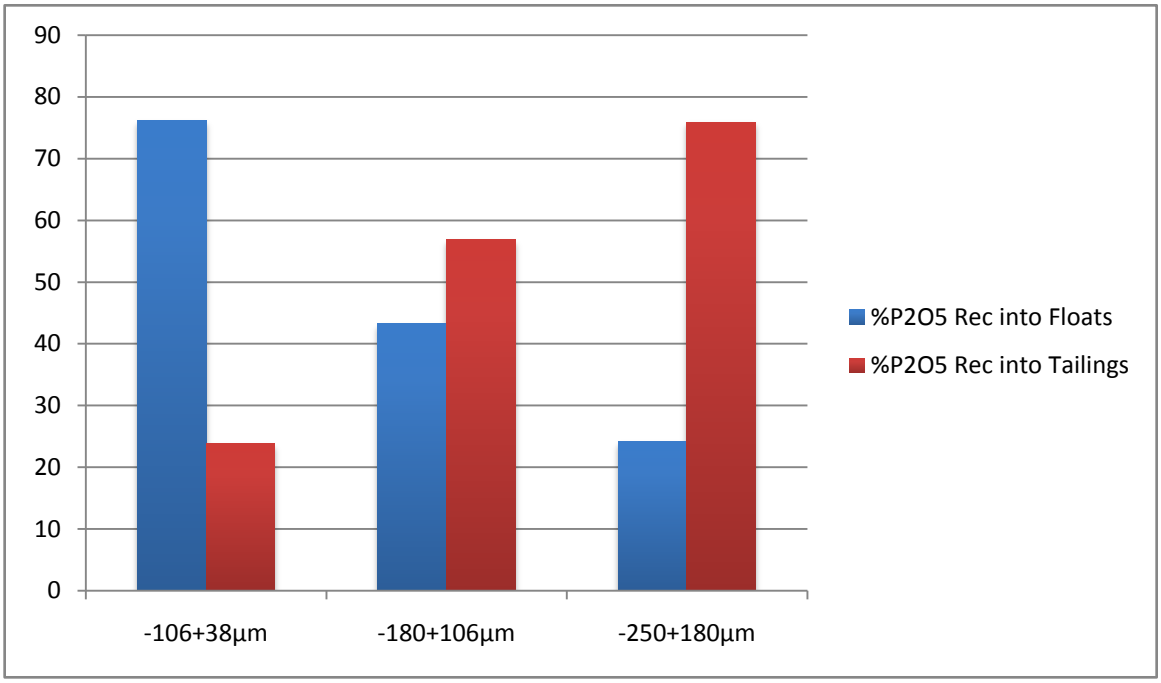


Figure 5.6.8: % Recovery of P_2O_5 into flotation Fractions using AERO 704Promoter

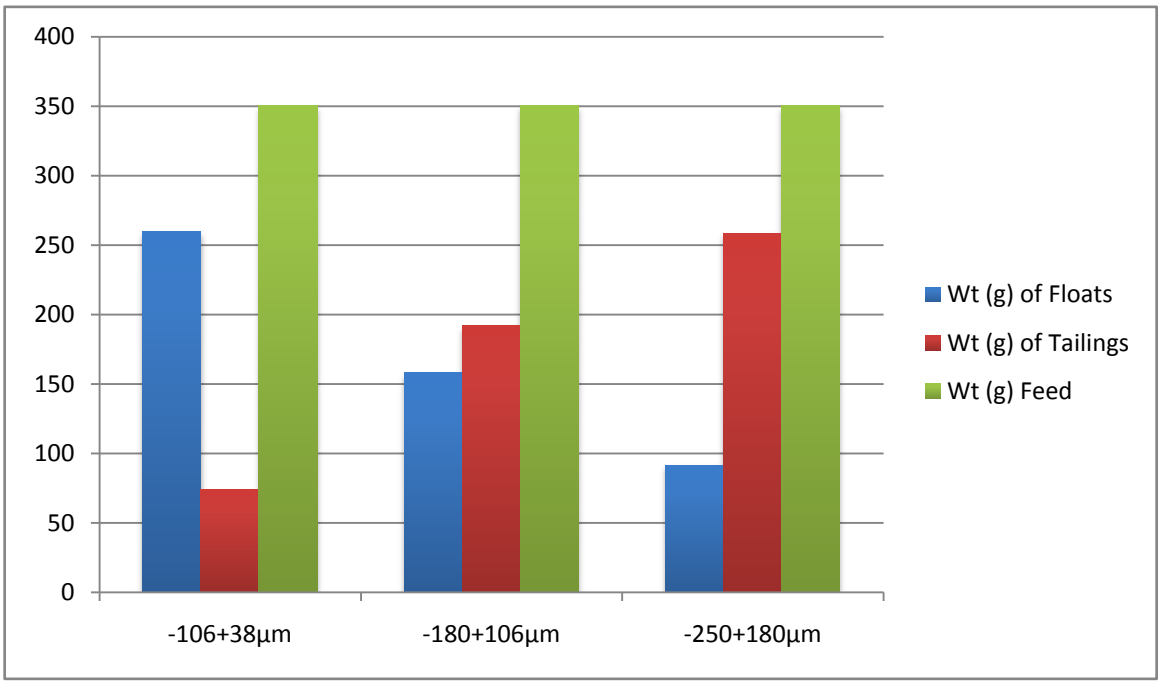
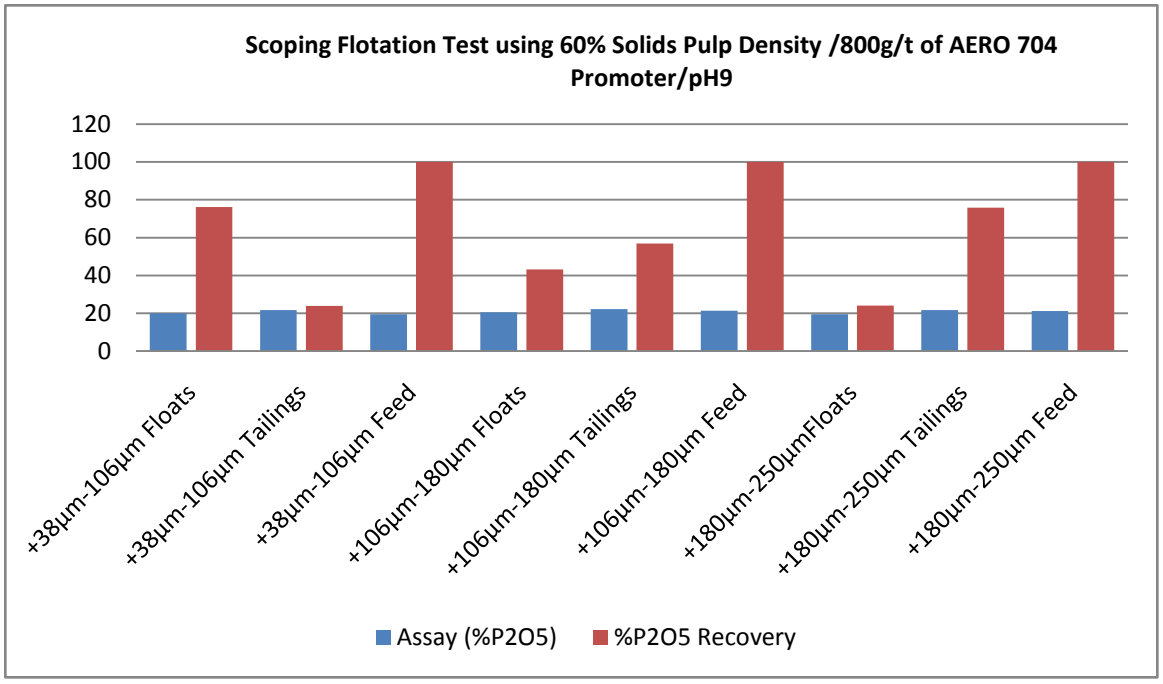


Figure 5.6.9: wt (g) of the flotation samples of various sieve size fractions using AERO704 promoter reagent.



Figures 5.6.10: %Assay and Recovery of the scoping flotation using pulp density of 60% solid/800g/t AERO 704 reagent dosage at pH9

Figure 5.6.11 shows the recovery figures for P_2O_5 components floated into the float fractions using AERO 704 and Alkyl Hydroxamates. The higher recovery figures recorded for P_2O_5component recovered into the floated fractions when the various sized fractions were floated using AERO 704 Promoter [76.2%, 43.2% and 24.1% respectively recorded for +38µm, +106µm and +180 size fractions] as compared to figures obtained when floated with reagent S-9849 Promoter(Alkyl Hydroxamates) [61.5%, 25.0% and 19.4% respectively for +38µm, +106µm and +180µm size fractions] show that AERO 704 is a better collector for the Sokoto Phosphate rock than reagent S-9849 Promoter(Alkyl Hydroxamates).

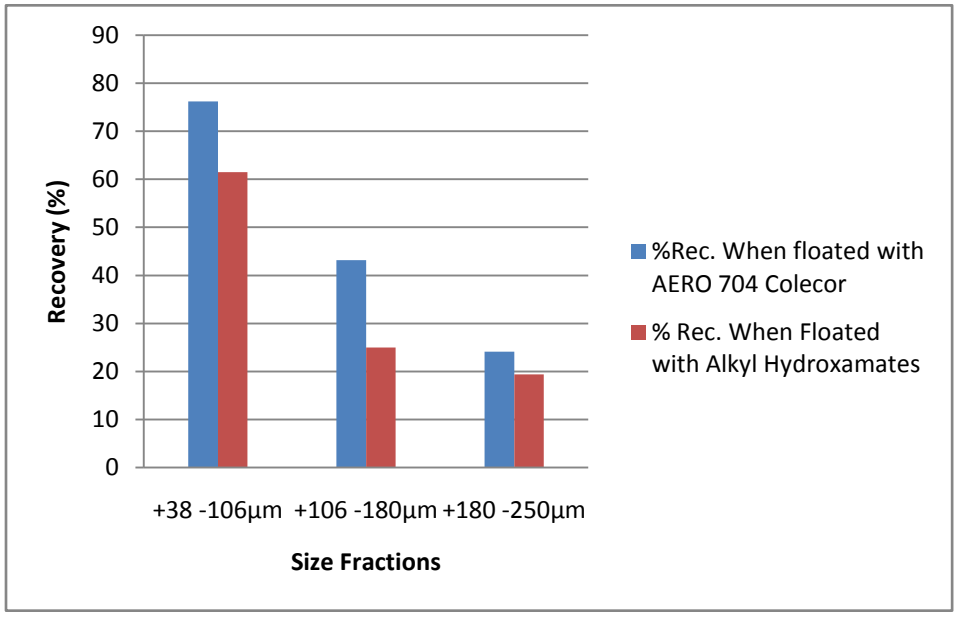


Figure: 5.6.11: Recovery figures for P_2O_5 Components recovered into Float Fractions collected using AERO 704 and Alkyl Hydroxames as collectors at pH9

iii. Table 4.3.4 shows the results of Scoping Studies on -106+38µm size fraction using 800g/t of various Reagent Types with Sodium Silicate and Aluminum Chloride as depressants forcalcite at 25% solids and conditioning pulp density of 60% solids and pH9. The results show that diesel/AERO 704 had a better recovery of 60.4% when compared with the recoveries of 55.4% and 48.8% respectively recorded for AERO 704 alone and Alkyl Hydroxamates. These values are however lower than the recovery figures recorded when AERO 704 (76.2%) and Alkyl Hydroxamates (61.5%) were used without any depressants. The grades of the various fractions collected showed marginal 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 (Weiss, 1985).

These results show that using AERO 704 collector alone and without any depressant in floating Sokoto phosphate rock will give better recovery results than either Alkyl Hydroxamates or AERO 704 combined with Diesel.

Figures5.6.7 and 5.6.8 respectively show the assays of $\%P_2O_5$ and % recoveries of P_2O_5 into the floats and tailings for the Scoping Studies of -106+38µm size fraction using 800g/t of with various reagent types, sodium silicate and aluminum chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9. The trends of the graphs illustrate the behavioral patterns of the reagents actions as regards the values obtained for the floats and tailings of the flotation study of the Sokoto phosphate mineral ore.

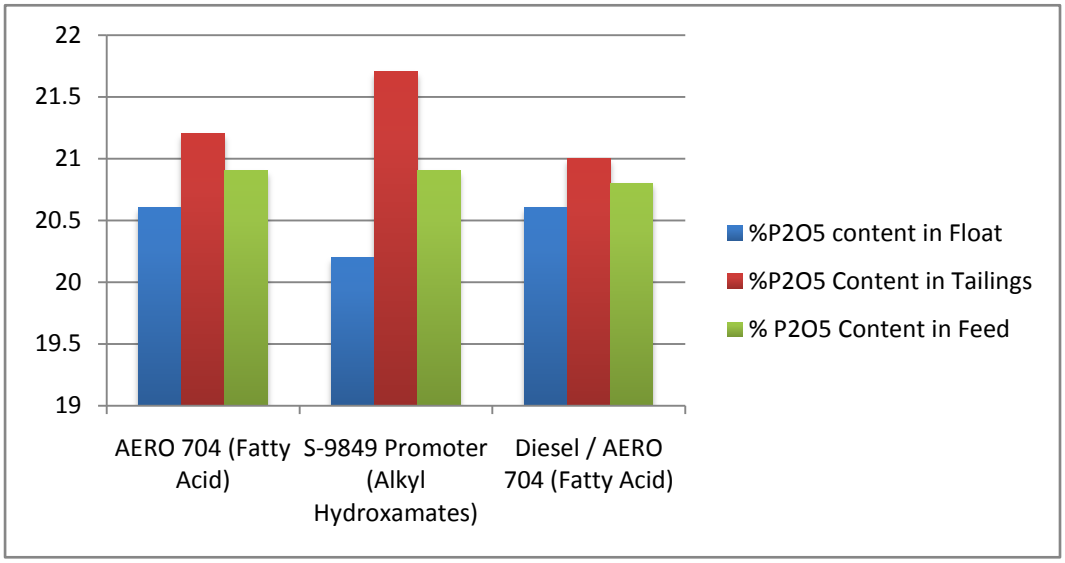


Figure 5.6.12: assays of $\%P_2O_5$infloats and tailings for Scoping Studies on -106+38µm size fraction using 800g/t of various Reagent Types with Sodium Silicate and Aluminum Chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9

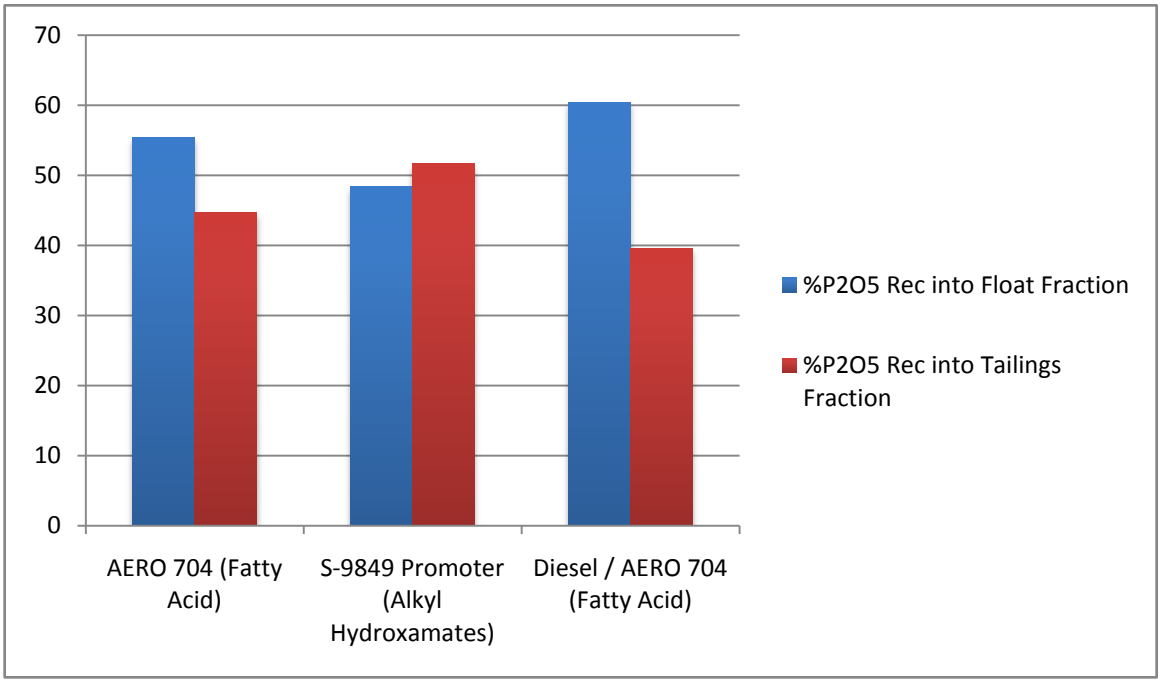


Figure 5.6.13: %Recovery of P_2O_5into flotation fractions of Scoping Studies on -106+38µm size fraction using 800g/t of various Reagent Types with Sodium Silicate and Aluminum Chloride as depressants for calcite at 25% solids and conditioning pulp density of 60% solids and pH9

(iv) Table 4.3.3 gives the result of the 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 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). Figures 5.6.14, 5.6.15 and 5.6.16 show %Assay of P_2O_5 and Recovery in the flotation products of -106+38μm at pH8/AERO 704 Promoter/MBIC (frother). The graphs illustrate the behavioral trends of the Sokoto phosphate bearing minerals in the flotation system as earlier explained above.

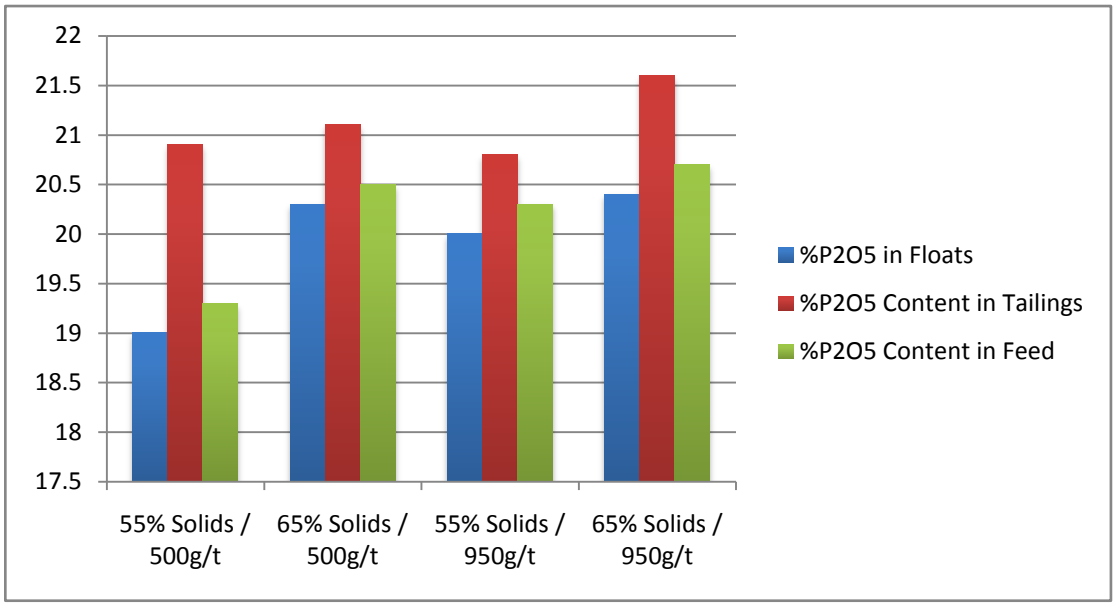


Figure 5.6.14: % Assay of P_2O_5 of the flotation Fractions of -106+38um at pH8/AERO 704 Promoter/MBIC (frother)

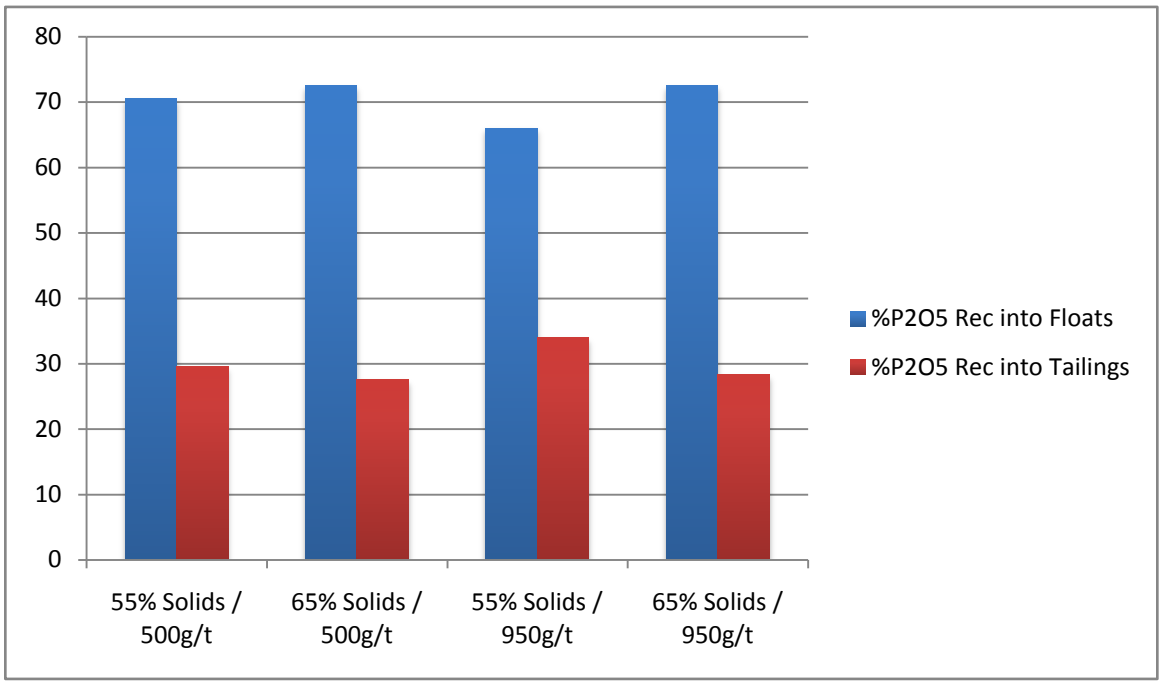


Figure 5.6.15: % Recovery of P_2O_5 of the flotation fractions of -106+38um at pH8/AERO 704 Promoter/MBIC (frother).

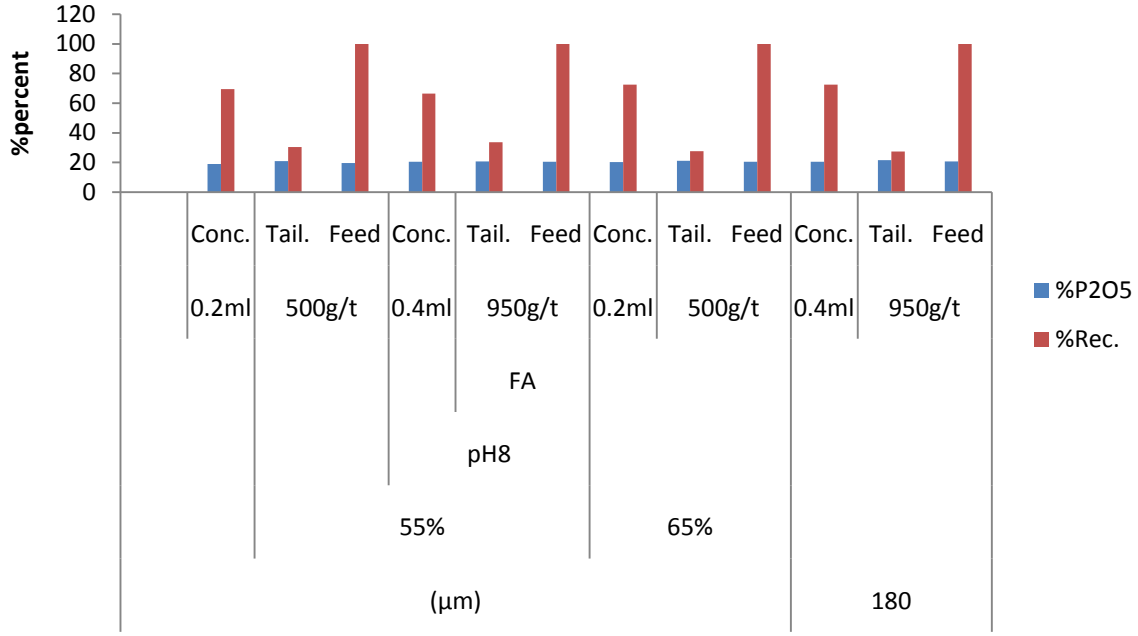


Figure 5.6.16: %Assay and Recovery of the flotation test result of 350g of

-106+38µm size fractions at 28.5% solids with FA collector and MBIC frother at pH8.

(v) Table 4.3.4 gives the result of the 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 reagents at pH10. 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 could be attributed 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). Figures 5.6.17,5.6.18 and 5.6.19 show %Assay of P_2O_5contents and Recovery of the flotation products of -106+38µm at pH10/AERO 704 Promoter/MBIC (frother). The graphs illustrate the behavioral trends of the Sokoto phosphate bearing minerals in the flotation system as earlier explained above.

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 attributed to the fact that the Sokoto phosphate bearing minerals are

sensitive to low basicity pH condition and higher above that, their qualities (%assay) reduce while their recoveries increase.

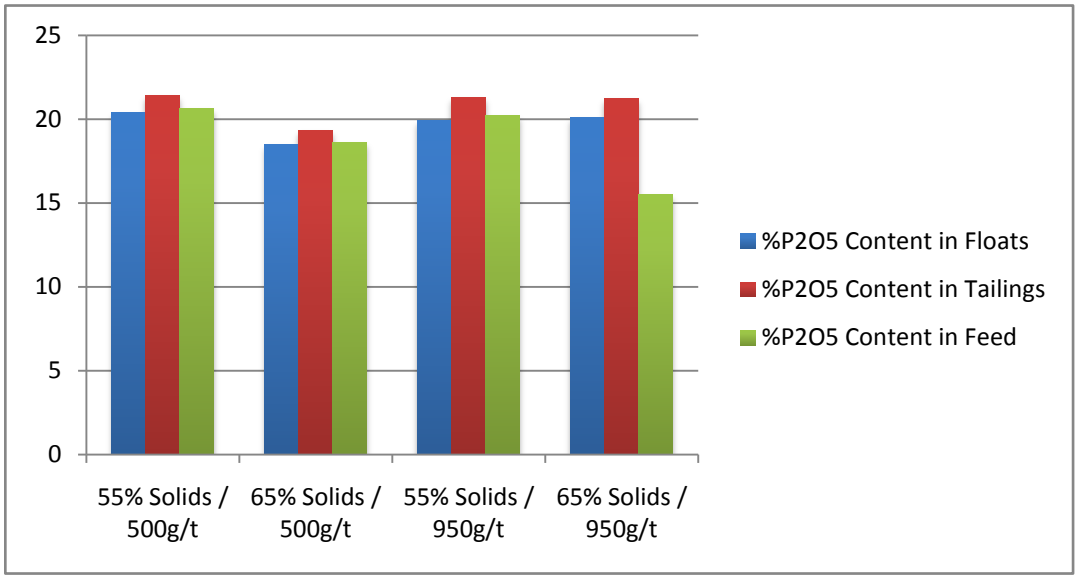


Figure 5.6.17: % Assay of P_2O_5 in the flotation samples of -106+38um at pH10/AERO 704Promoter/MBIC(frother)

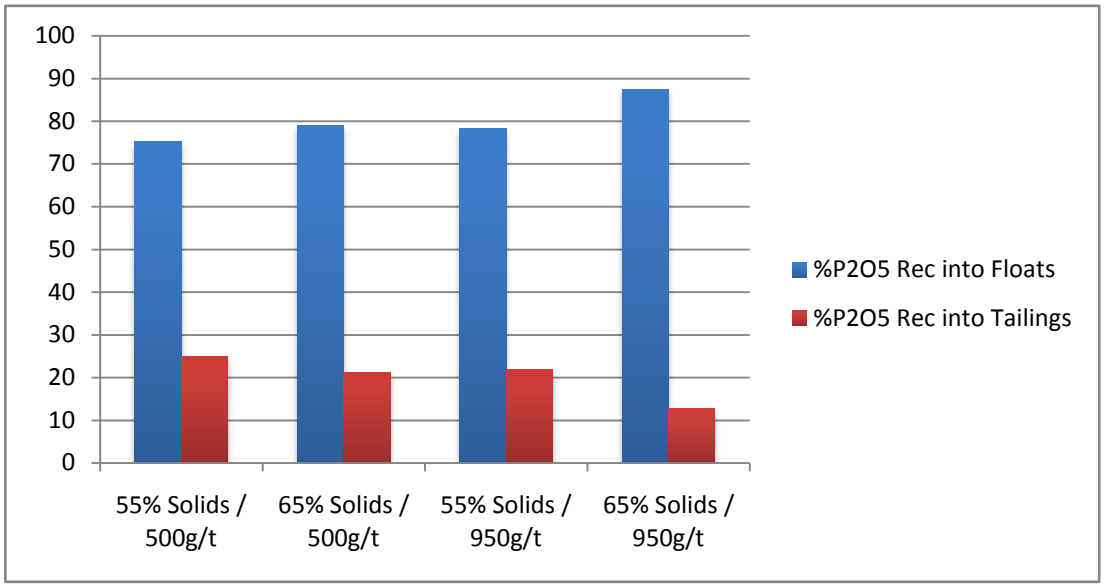


Figure 5.6.18: % recoveries of P_2O_5 into floats produced during floatation test using 350g of -106+38 µm size fraction at 28.5% solids with AERO®704 Promoter (Distilled Tall-oil Fatty Acids) and Methyl isobutyl carbinol (MBIC) frother at pH10

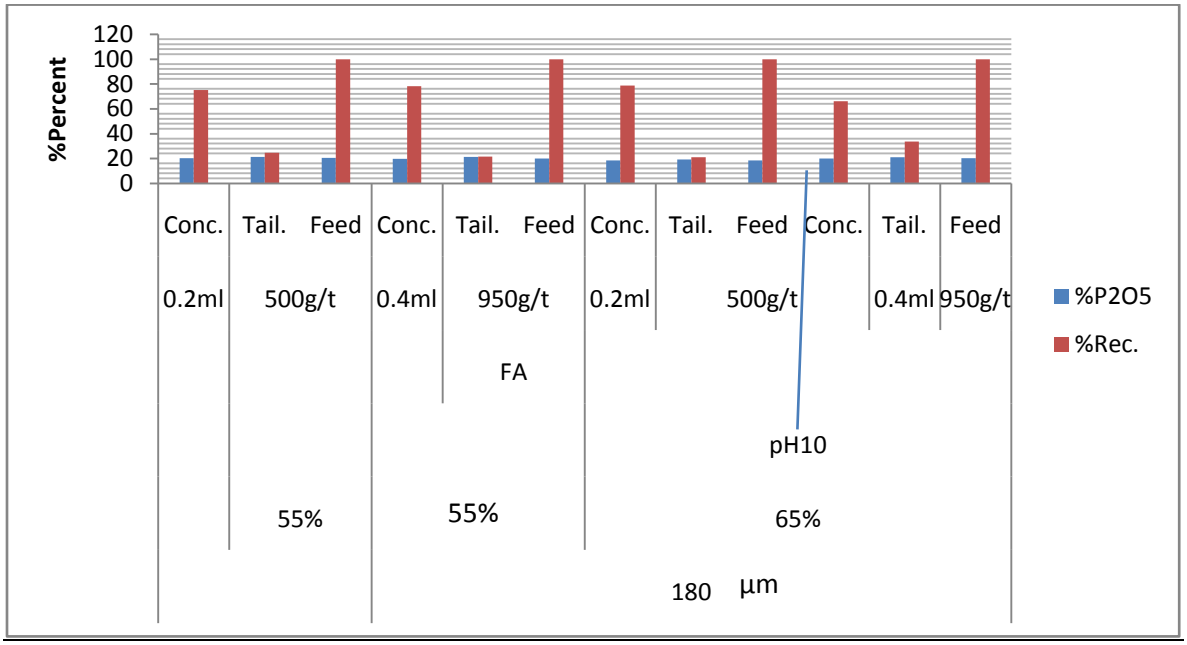


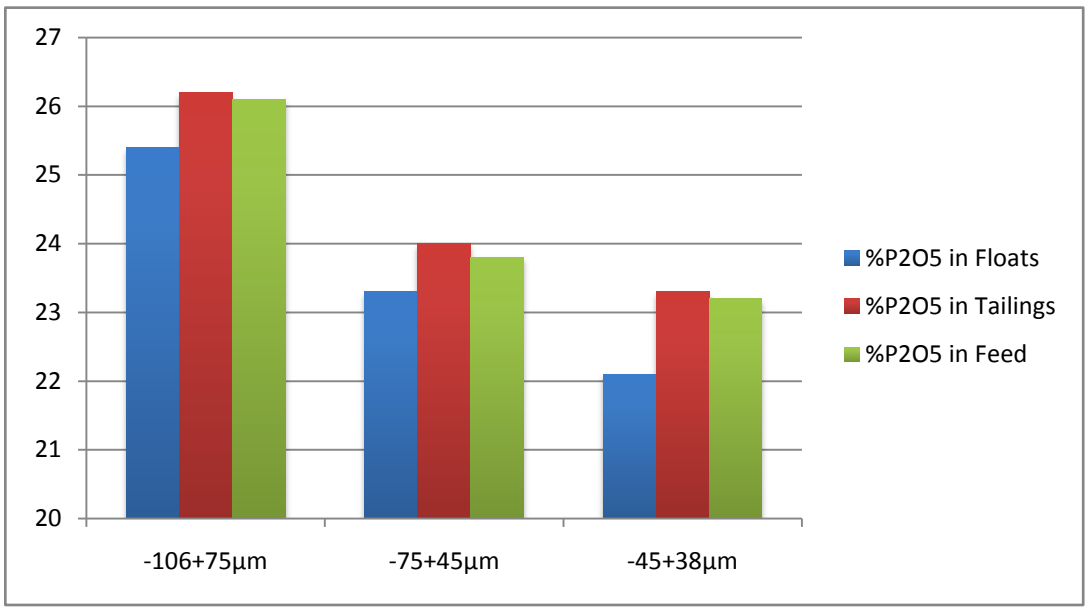
Figure 5.6.19: %Assay and Recovery of the flotation test result of 350g of -106+38µm size fractions at 28.5% solids with FA collector and MBIC frother at pH10

5.7 Discussion on the Result of the Calcite flotation Test

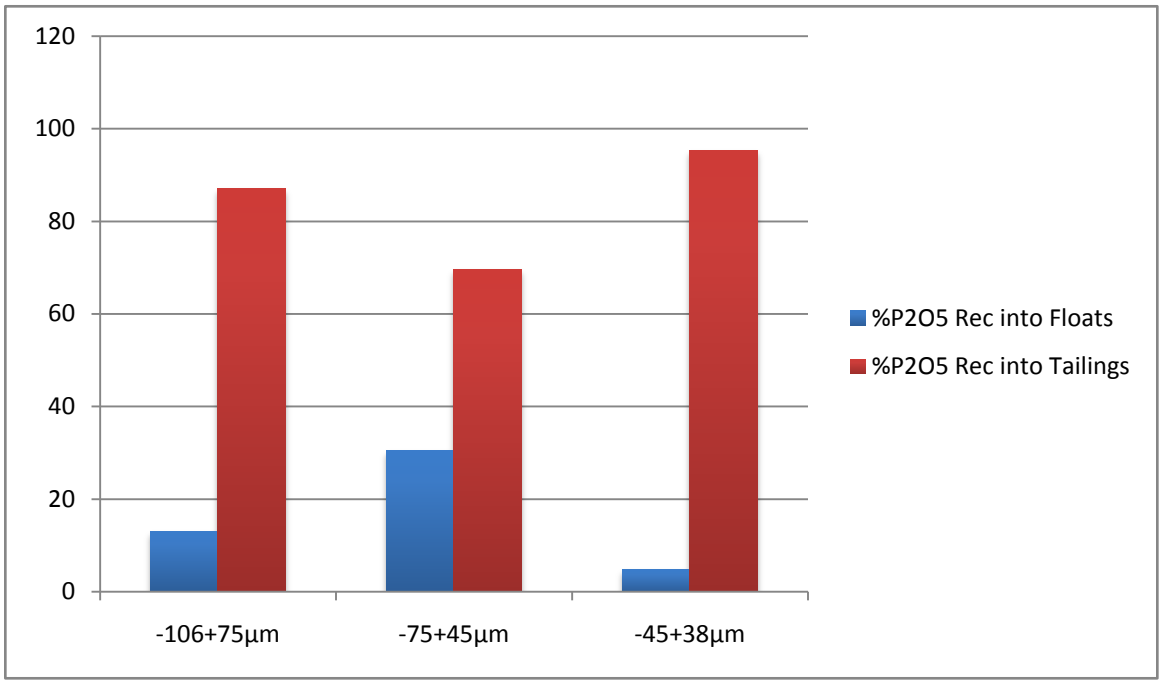
Table 4.3.5 gives the result of Calcite Flotation of Sokoto phosphate rock at pH9 using Melamine as collector, Dextrin as depressant for phosphate rock and MBIC as frother. The result shows that the flotation test on the -75+45um size fraction produced a concentrate (float) product weighing 31.1% of the feed, assaying 23.3%P_2O_5 with a recovery of 30.4% of the P_2O_5 content. The test on the -106+75µm sieves size fraction produced a float product weighing 13.4% of the feed assaying 25.4%P_2O_5 with a recovery of 13.0% of the P_2O_5 content while the test on the -45+38µm fraction produced a float product assaying 22.1%P_2O_5 containing 4.7% of the P_2O_5 contained in the feed.

Figures 5.7.1; 5.7.2 and 5.7.3 showthe behavioral trends of the % assays and recoveries of P_2O_5 into floats and the tailings recovered during Scoping Flotation Tests of Calcite Flotation at pH9 using Melamine as collector, Dextrin as depressant for phosphate rock and MBIC as frother. These reveal low froth collection as shown by the various weight percentages for all the size fractions tested as well as no clean separation of the valuable P_2O_5 content as revealed by the grades of the floats and tailings products produced.

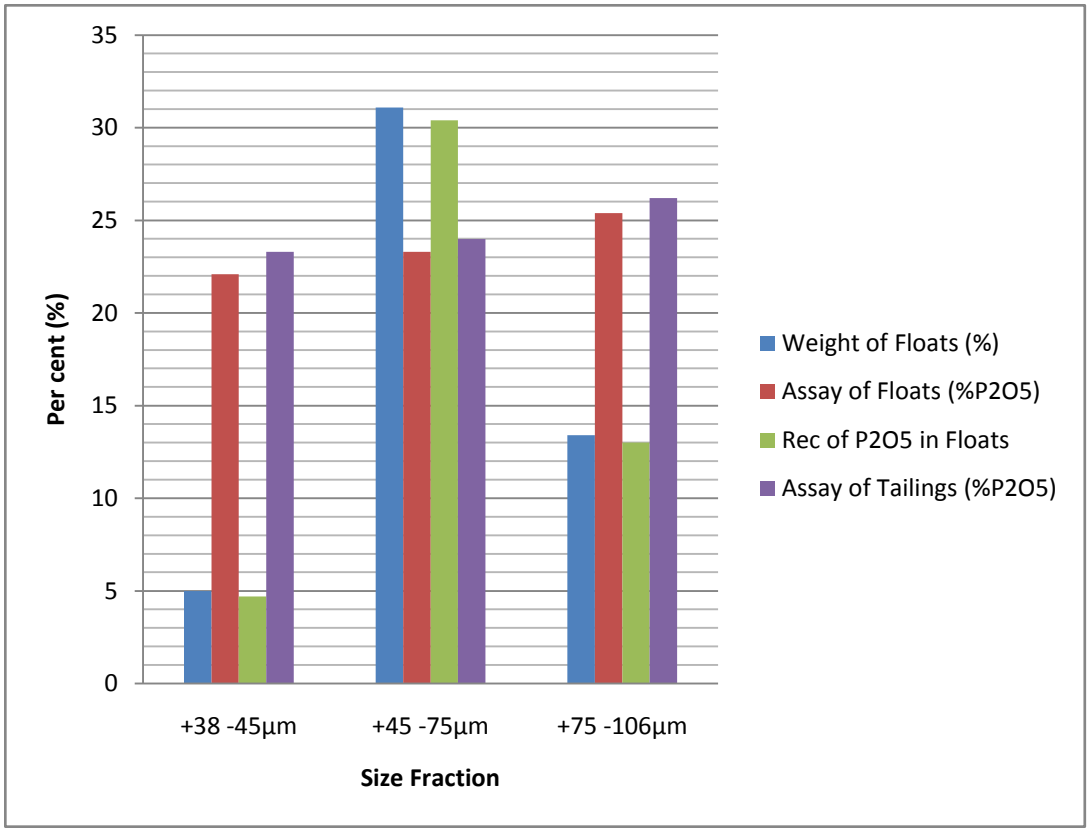
The lack of clean separation can be explained by inadequate liberation of the P_2O_5 valuable content arising from the collophane nature of the test material which made liberation at the test sizes impossible.



Figures 5.7.1: % Assay of P_2O_5 in floats recovered during Scoping Flotation Tests of Calcite Flotation at pH9 using Melamine as collector, Dextrin as depressant for phosphate rock and MBIC as frother



Figures 5.7.2: Recoveries of P_2O_5 in floats recovered during Scoping Flotation Tests of Calcite Flotation at pH9 using Melamine as collector, Dextrin as depressant for phosphate rock and MBIC as frother



Figures 5.7.3:Weights of Float Fraction (%); Assay (%P_2O_5) and Recoveries of P_2O_5 into floats recovered during Scoping Flotation Tests of Calcite Flotation at pH9 using Melamine as collector, Dextrin as depressant for phosphate rock and MBIC as frother.

5.8 Discussion on the Result of Incremental Flotation Test

Table 4.3.6 gives the results of incremental flotation test carried out using 800g/t of AERO 704 (Fatty Acid) at 25% solids and conditioning pulp density of 60% solids and pH9. From the result, float1has an assay content of 23.0% P_2O_5, float 2 (22.3%P_2O_5), float 3 (22.1%P_2O_5), float 4 (22.6%P_2O_5), and float 6 (21.4%P_2O_5) floated from a feed assaying 22.5%.

Figures5.8.1 and 5.8.2 show that the incremental test produced a composite float assaying 22.6% P_2O_5 with 53.1% P_2O_5 recovery. The results show non-clean separation arising from non-

significant differences between the assays of P_2O_5 of the various fractions collected caused by the intimate association of P_2O_5 minerals with the carbonate minerals that inhibit phosphate flotation.

The grades of the float fractions collected show that the Roughing and Scavenging stages can be separated after two minutes flotation, which is the point at which the grade of the feed is equal to the grade of the float.

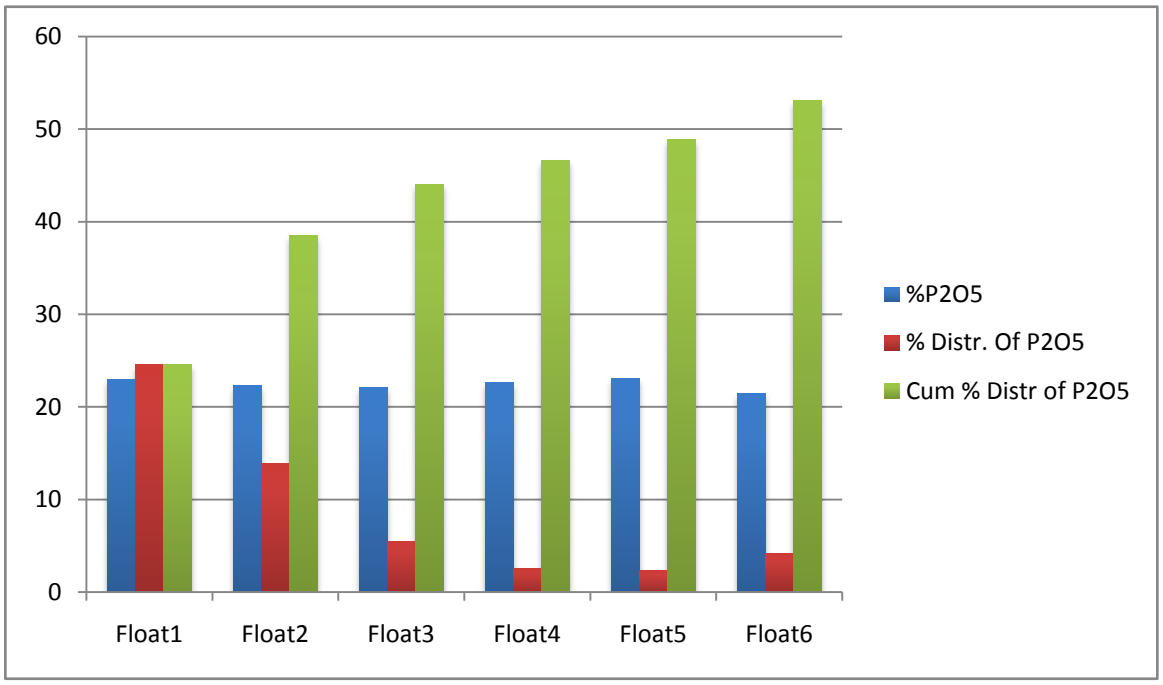


Figure 5.8.1:%P_2O_5, % distribution of $P_2O_5$and cumulative distribution of P_2O_5 in the various floats of the incremental method flotation test of 800g/t/AERO704 at 25% solid/pulp density 60% at pH9 of particle size fractions of -106+ 38µm

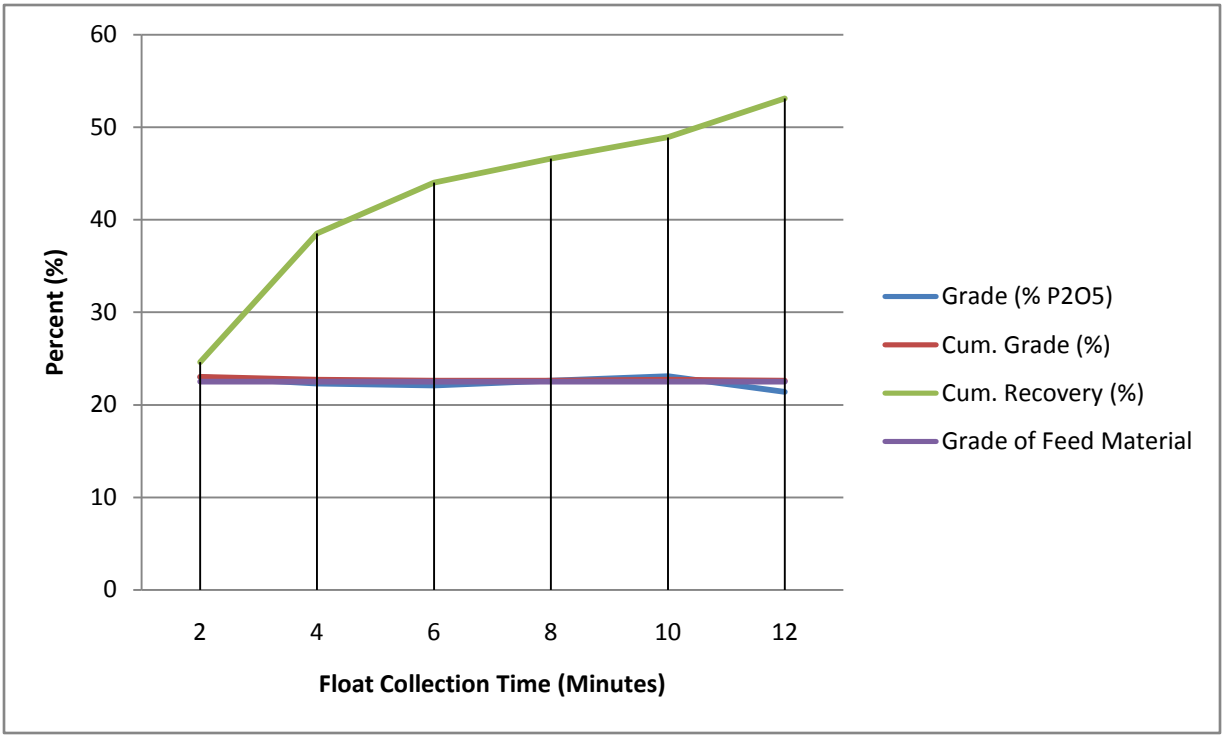


Figure 5.8.2: %P_2O_5 of float fractions, distribution and cumulative distribution of P_2O_5 in the various floats fractions and Feed Grade used in the incremental method flotation test of 800g/t/AERO704 at 25% solid/pulp density 60% at pH9 of particle size fractions of -106+ 38µm

5.9. The Design Philosophy of the Proposed Process flowsheet for the Beneficiation of the Sokoto Phosphate Mineral Ore Deposit

5.9.1 Introduction

The results of the chemical analysis showed that the Run-of-Mine contained 44.3%SiO_2, 12.1% P_2O_5, 19.1%CaO, 3.9%Fe_2O_3, and 10.5% Al_2O_3 while the sorted nodules contained 14.2%SiO_2, 21.7% P_2O_5, 44.31%CaO, 6.19%Fe_2O_3, and 3.60% Al_2O_3. These results show that the P_2O_5 content could be upgraded by sorting out the phosphate nodules from the shale matrix.

The mineralogical analysis showed that the Sokoto phosphate rock contained hydroxylapatite, flourapatite and carbonate-flouapatite, which are phosphate bearing minerals in association with Calcite and Quartz. The chemical analysis showed that calcite constituted the major mineral.

The hydroxylapatite, flourapatite and carbonate-flourapatite belong to the apatite family of phosphate bearing minerals, which the thin section photomicrographs showed to be intimately associated with the other mineral assemblage in very fine texture with no visible grain boundaries seen under the microscope.

Sokoto Phosphate rock is also characterized by high percentage of lime (44.3%), a high iron ore content (6.2%) and having an average specific gravity of 2.9.

The results of the size- Assay analysis indicate that Sokoto phosphate nodules could be upgraded using a screen mesh having 12.7mm apertures to produce a Phosphate nodule fraction grading 24.5% P_2O_5 with a corresponding P_2O_5 recovery of 72.1%. This fraction could then be ground to the required particle size of 80% passing 75µm which falls within the less than 150µm size proposed for phosphate rock product by Agriculture and Food Organization (2002) using Roller Mills.

Flotation tests results show no clean separation of the phosphate bearing minerals from its associated mineral assemblage due to non-liberation arising from very fine mineral association. This indicates that Sokoto phosphate rock is not economically amenable to upgrading by flotation due to the need to grind very fine below economic flotation range to achieve liberation.

However, flotation with AERO 704 (fatty acid) was observed to have given the best recovery of 87.3% at 950g/t of reagent, 65% solid conditioning pulp density and pH10 albeit with poor separation.

In view of the above observations, the flowsheet for upgrading Sokoto Phosphate Rock is proposed to comprise a scrubber with a retaining screen having 12700µm apertures and Milling System containing a Gyratory Crushing Unit, Ring Roll Pulverizer and Cyclone Classification Unit for Powdered Material Collection fitted with Air Cleaning system as shown in figure 5.9.1.

(i) **Scrubbing**

This entails using a rotating washing drum that has a 12.7mm retaining screen that separates +12.7mm material grading 24.5%P_2O_5 from -12.7mm material and water which will be recycle via a settling pond. The +12.7mm material will be dried using solar and then pulverized.

(ii) **Comminution/ Classification Unit (Crushing, grinding and Air Classification)**

This comprises a gyratory Crushing unit that produces phosphate nodule chippings that would be fed into a ring roll mill to produce 100% passing 75µm, 24.5% P_2O_5 product that can be used to produce feed material for fertilizer production using a cyclone fitted with air cleaner to prevent dust pollution

Run-of-Mine (12.1% P_2O_5, 80% passing 29,300um) Plus Water



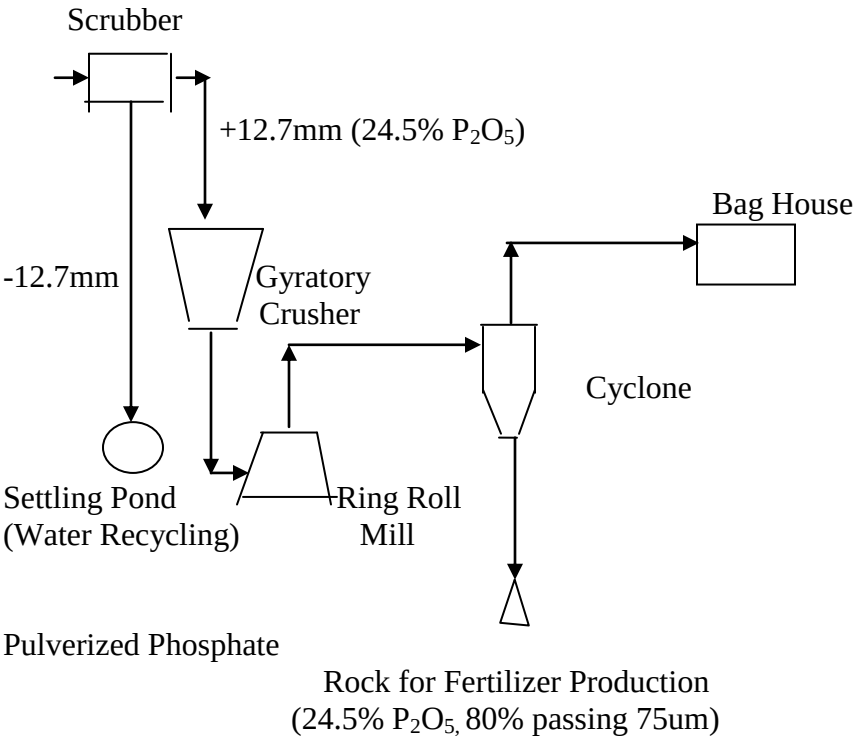


Figure 5.9.1: Process Flow Sheet for the Beneficiation of Sokoto Phosphate Rock

Chapter six

6.0 Conclusion and Recommendations

6.1 Conclusions

The characterization and beneficiation parameters of Sokoto phosphate mineral ore have been investigated and determined for the design of the process flow-sheet 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 are:

a. That Sokoto phosphate rock Run-of-Mine Ore contained44.3%SiO_2, 12.1% P_2O_5, 19.1%CaO, 3.9%Fe_2O_3, and 10.5% Al_2O_3 while the sorted nodules without scalping contained 14.2%SiO_2, 21.7% P_2O_5, 44.31%CaO, 6.19%Fe_2O_3, and 3.60% Al_2O_3. This makes the mineral ore a low grade phosphate deposit containing high iron and silica percent contents in line with the categorization system used in Weiss, 1985;

b. That the Sokoto phosphate rock contains calcite as the predominant mineral with hydroxylapatite, flourapatite and carbonate-flouapatiteasas the phosphate bearing minerals;

c. That Sokoto phosphate rock has an average specific gravity of 2.9 which agrees with the range of 2.9 – 3.2 sited in Encyclopedia Britanica (2010);

d. That Sokoto phosphate mineral ore has a work index of 56.7kwh/t which is higher than the range of 10.13 –13.03 Kwh/t reported in Fuerstenau and Han, 2003 arising from very fine intimate mineral association;

e. That Sokoto phosphate rock nodules show very fine intimate association of the phosphate bearing minerals with the other mineral assemblage;

f. That Sokoto phosphate mineral exists in nodules disseminated in shale /clay matrix;

g. That 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;

h. That AERO 704 (fatty acid) gave the best collector response; and

i. That a Process Flowsheet comprising a scrubber with a retaining screen of 12.7mm, a Milling Assembly made up of a Crusher, Grinder and Classifier fitted to a bag house will successfully produce about 24.5% P_2O_5milled low grade 80% passing 75µm

phosphate feed for fertilizer manufacture and other uses as observed by Weiss, 1985 with very minimal particulate matter discharged to the environment.

6.2 Recommendations

It is recommended that Pre-thermal treatment measures like calcination or roasting be applied prior to the beneficiation of Sokoto phosphate mineral ore to find out if liberation of the associating minerals could be enhanced.

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APPENDICES

APPENDIX 1

Experimental Design for the Design of Process Flowsheet For Sokoto Phosphate Deposit

Experimental works carried out are as outlined below.

1. **Mineral Characterization**
   a. Size –Assay Analysis using sieves and XRF for chemical analysis
   b. Mineralogical analysis of head sample using XRD
   c. Density Determination using density bottle method
   d. Comminution studies:
      i. Work index determination using Berry and Bruce Method
      ii. Mesh of grind determination by grinding to different size products
      iii. Sieve analysis on ground material to determine 80% passing sizes

2. **Flotation Tests on Sokoto Phosphate Rock using Denver Laboratory Flotation Machine**

   Reagents used comprised AERO 704 (Fatty Acid), Sodium Carbonate, Calcium Hydroxide, Reagent S-9849, (an alcoholic solution of alkyl hydroxamic acid from Cytec), Aerofroth 70 (methylisobutylcarbinol - MIBC), Dextrin, Phosphoric Acid and Melamine.)

i. **Determining the economic mesh of grind and reagent scoping**

Flotation tests carried out on samples milled in d (ii) above using a conditioning pulp density of 60%, reagent doses of 800g/t of selected reagents and a flotation pulp density of about 28.5% solids.

ii. **Reagent dosage scoping with 60% pulp density and 28.5% flotation pulp density.**

Flotation tests carried out using various reagent dosages (500, 650, 800, 950g/l) of the better of the two reagents.

iii. **Conditioning Pulp Density scoping**

Flotation tests to be carried out using various conditioning pulp densities (55, 60, 65) using the best reagent dosage obtained in 'B' above and 28.5% flotation pulp density.

iv. **Determination of flotation kinetics**

Flotation Kinetics test carried out using incremental method by conditioning 350g of phosphate rock with 800g/t of AERO 704 (Fatty Acid) at 25% solids and conditioning pulp density of 60% solids and PH9. Six samples collected every two (2) minutes (2, 4, 6, 8, & 10)

APPENDIX 2

XRD Results

Name and formula

Reference code:	01-085-0798
Mineral name:	Quartz, syn
Compound name:	Silicon Oxide
PDF index name:	Silicon Oxide
Empirical formula:	O_2Si
Chemical formula:	SiO_2

Crystallographic parameters

Crystal system:	Hexagonal
Space group:	P3221
Space group number:	154
a (Å):	4.9140
b (Å):	4.9140
c (Å):	5.4050
Alpha (°):	90.0000
Beta (°):	90.0000
Gamma (°):	120.0000
Volume of cell (10^6 pm^3):	113.03
Z:	3.00
RIR:	3.33

Status, subfiles and quality

Status:	Alternate Pattern
Subfiles:	Alloy, metal or intermetalic Cement and Hydration Product Common Phase Excipient Forensic ICSD Pattern Inorganic Mineral Pharmaceutical
Quality:	Blank (B)

Comments

ANX:	AX2
Creation Date:	11/20/2008
Modification Date:	1/21/2009
ANX:	AX2
Analysis:	O2 Si1
Formula from original source:	Si O2
ICSD Collection Code:	27834
Calculated Pattern Original Remarks:	Stable up to 846 K, above P6222, above 1143 K tridymite is stable
Significant Warning:	15%<R factor (for powder)
Wyckoff Sequence:	c a(P3221)
Unit Cell Data Source:	Powder Diffraction.

References

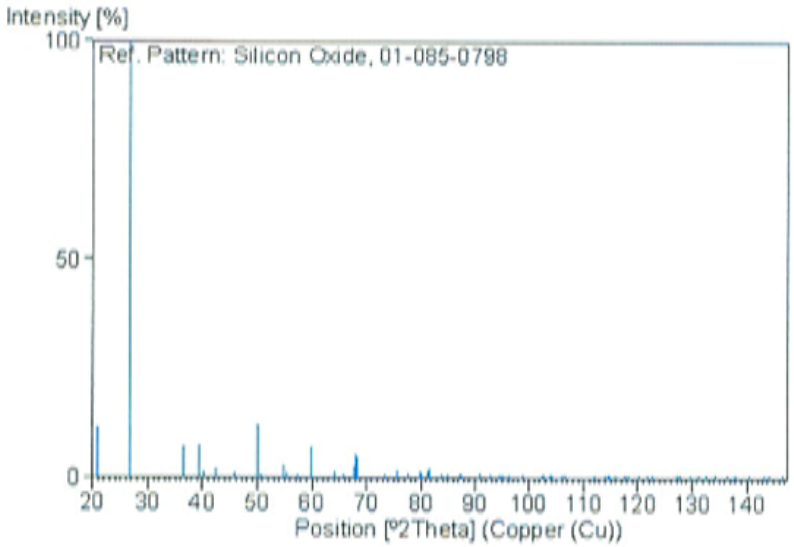
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Structure: Young, R.A., Mackie, P.E., von Dreele, R.B., *J. Appl. Crystallogr.*, **10**, 262, (1977)

Peak list

No.	h	k	l	d [A]	2Theta[deg]	I [%]
1	1	0	0	4.25560	20.857	11.8
2	0	1	1	3.34360	26.639	100.0
3	1	1	0	2.45700	36.542	7.3
4	1	0	2	2.28140	39.467	7.6
5	1	1	1	2.23670	40.289	1.8
6	2	0	0	2.12780	42.448	2.5
7	0	2	1	1.97990	45.792	1.4
8	1	1	2	1.81800	50.138	12.3
9	0	0	3	1.80170	50.623	0.3
10	0	2	2	1.67180	54.872	3.0
11	0	1	3	1.65910	55.328	1.2
12	2	1	0	1.60850	57.226	0.7
13	2	1	1	1.54170	59.953	7.2
14	1	1	3	1.45290	64.035	1.6
15	3	0	0	1.41860	65.776	0.2
16	1	2	2	1.38220	67.739	2.7
17	2	0	3	1.37500	68.142	5.3
18	0	3	1	1.37210	68.306	4.9
19	1	0	4	1.28790	73.469	1.1
20	0	3	2	1.25600	75.656	2.1
21	2	2	0	1.22850	77.662	1.2
22	2	1	3	1.19990	79.878	1.8
23	2	2	1	1.19800	80.030	1.3
24	1	1	4	1.18400	81.172	1.5
25	3	1	0	1.18030	81.480	2.5
26	1	3	1	1.15310	83.830	0.9
27	2	0	4	1.14070	84.953	0.3
28	2	2	2	1.11840	87.063	0.1
29	0	3	3	1.11450	87.444	0.1
30	1	3	2	1.08160	90.826	1.2
31	4	0	0	1.06390	92.777	0.1
32	0	1	5	1.04770	94.653	0.8
33	0	4	1	1.04390	95.107	0.8
34	2	1	4	1.03460	96.239	1.1
35	2	2	3	1.01500	98.738	0.3
36	0	4	2	0.98950	102.242	0.5
37	1	1	5	0.98950	102.242	0.5
38	1	3	3	0.98730	102.559	0.8
39	0	3	4	0.97840	103.869	0.3
40	3	2	0	0.97630	104.184	0.5
41	0	2	5	0.96380	106.114	0.1
42	2	3	1	0.96080	106.591	0.9
43	4	1	0	0.92870	112.082	0.1
44	3	2	2	0.91820	114.053	0.4
45	0	4	3	0.91610	114.459	0.7
46	1	4	1	0.91520	114.635	0.8
47	2	2	4	0.90900	115.863	0.3
48	0	0	6	0.90080	117.548	0.1
49	1	2	5	0.89720	118.311	0.7
50	1	3	4	0.88890	120.126	0.3

51	1	0	6	0.88130	121.865	0.1
52	1	4	2	0.87830	122.574	0.4
53	0	3	5	0.85980	127.250	0.2
54	3	2	3	0.85840	127.628	0.1
55	5	0	0	0.85110	129.664	0.1
56	1	1	6	0.84580	131.214	0.2
57	0	5	1	0.84080	132.740	0.1
58	0	4	4	0.83590	134.299	0.1
59	0	2	6	0.82960	136.410	0.6
60	1	4	3	0.82550	137.857	0.9
61	3	3	0	0.81900	140.284	0.2
62	5	0	2	0.81180	143.201	0.9
63	2	2	5	0.81180	143.201	0.9
64	3	3	1	0.80980	144.062	0.5
65	4	2	0	0.80420	146.609	0.2

Stick Pattern



Name and formula

Reference code:	01-070-7718
Mineral name:	Hydroxylapatite, syn
Compound name:	Calcium Oxide Phosphate Deuteriooxide
Common name:	calcium phosphate(V) oxide deuteriooxide
PDF index name:	Calcium Oxide Phosphate Deuteriooxide
Empirical formula:	$Ca_{4.79}DO_{13}P_{2.97}$
Chemical formula:	$Ca_{4.79}(PO_4)_{2.97}O_{0.12}(OD)$

Crystallographic parameters

Crystal system:	Hexagonal
Space group:	P63/m
Space group number:	176
a (Å):	9.4183
b (Å):	9.4183
c (Å):	6.8843
Alpha (°):	90.0000
Beta (°):	90.0000
Gamma (°):	120.0000
Volume of cell (10^6 pm^3):	528.85
Z:	2.00
RIR:	1.07

Subfiles and Quality

Subfiles:	ICSD Pattern Inorganic Mineral
Quality:	Blank (B)

Comments

ANX:	A3B5X13
Creation Date:	11/20/2008
Modification Date:	1/21/2009
ANX:	A3B5X13
Analysis:	D1 Ca4.79 O13 P2.97
Formula from original source:	Ca4.79 (P O4)2.97 O0.12 (O D)
ICSD Collection Code:	093531
Test from external database:	Deviation of the charge sum from zero tolerable
Minor Warning:	Minor test comments from ICSD exist
Significant Warning:	ICSD Warning on inter-atomic distances exist
Wyckoff Sequence:	i h4 f e(P63/M)
Unit Cell Data Source:	Powder Diffraction.

References

Primary reference:	*Calculated from ICSD using POWD-12++*
Structure:	Leventouri, T., Chakoumakos, B.C., Papanearchou, N., Perdikatsis, B., *J. Mater. Res.*,

16, 2600, (2001)

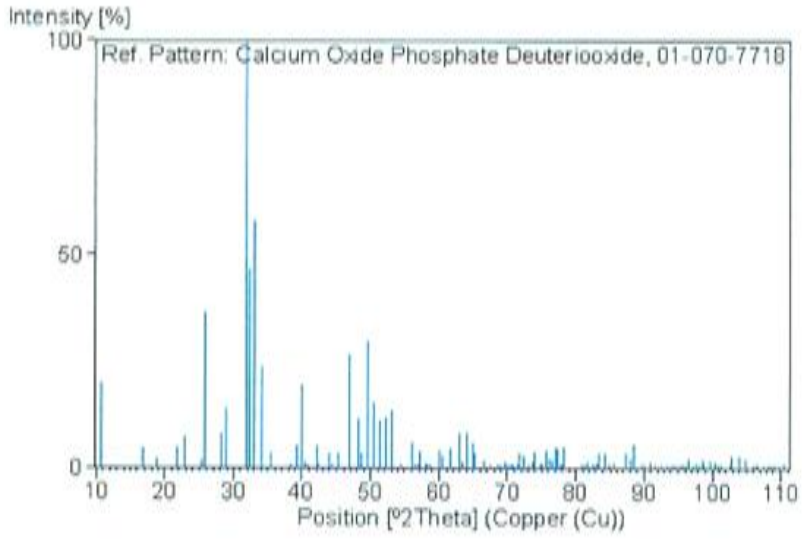
Peak list

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3	1	1	0	4.70920	18.829	2.3
4	2	0	0	4.07820	21.775	5.0
5	1	1	1	3.88680	22.862	7.5
6	2	0	1	3.50880	25.363	2.0
7	0	0	2	3.44220	25.862	36.5
8	1	0	2	3.17130	28.115	8.2
9	2	1	0	3.08290	28.939	14.2
10	2	1	1	2.81360	31.778	100.0
11	1	1	2	2.77890	32.186	46.4
12	3	0	0	2.71880	32.917	57.9
13	2	0	2	2.63040	34.057	24.2
14	3	0	1	2.52880	35.470	3.9
15	2	2	0	2.35460	38.191	0.3
16	2	1	2	2.29650	39.197	5.5
17	1	3	0	2.26220	39.816	19.6
18	2	2	1	2.22790	40.455	1.6
19	1	0	3	2.20900	40.817	0.3
20	1	3	1	2.14910	42.008	5.5
21	3	0	2	2.13360	42.327	1.1
22	1	1	3	2.06290	43.852	3.9
23	4	0	0	2.03910	44.391	0.8
24	2	0	3	1.99990	45.308	3.8
25	2	2	2	1.94340	46.703	26.7
26	1	3	2	1.89050	48.091	11.6
27	2	3	0	1.87120	48.618	3.6
28	2	1	3	1.84080	49.475	29.8
29	3	2	1	1.80570	50.503	15.3
30	1	4	0	1.77990	51.288	11.2
31	4	0	2	1.75440	52.089	11.9
32	3	0	3	1.75440	52.089	11.9
33	1	4	1	1.72110	53.175	13.8
34	0	0	4	1.72110	53.175	13.8
35	1	0	4	1.68400	54.442	0.9
36	3	2	2	1.64400	55.881	6.3
37	5	0	0	1.63130	56.354	0.3
38	1	1	4	1.61650	56.917	0.3
39	3	1	3	1.61100	57.129	4.0
40	5	0	1	1.58730	58.063	1.4
41	2	0	4	1.58730	58.063	1.4
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44	2	4	0	1.54140	59.966	4.4
45	3	3	1	1.53040	60.441	3.2
46	2	4	1	1.50420	61.608	4.8
47	1	2	4	1.50420	61.608	4.8
48	5	0	2	1.47410	63.008	8.2
49	5	1	0	1.46500	63.445	1.5
50	3	0	4	1.45420	63.971	6.9
51	3	2	3	1.45020	64.169	8.4
52	5	1	1	1.43290	65.038	6.3
53	3	3	2	1.42820	65.279	3.9
54	4	2	2	1.40680	66.399	2.1

55	1	4	3	1.40680	66.399	2.1
56	2	2	4	1.38950	67.335	0.1
57	3	1	4	1.36970	68.442	0.4
58	6	0	0	1.35940	69.033	0.2
59	5	1	2	1.34800	69.701	1.8
60	4	3	0	1.34090	70.124	0.4
61	6	0	1	1.33370	70.559	0.1
62	5	0	3	1.32960	70.809	0.2
63	1	1	5	1.32150	71.309	0.3
64	4	3	1	1.31620	71.641	3.6
65	4	0	4	1.31620	71.641	3.6
66	5	2	0	1.30610	72.282	3.0
67	3	3	3	1.29560	72.961	0.3
68	5	2	1	1.28320	73.782	1.8
69	2	4	3	1.27960	74.024	4.0
70	2	3	4	1.26680	74.900	1.0
71	6	0	2	1.26440	75.067	1.3
72	2	1	5	1.25720	75.572	4.5
73	3	4	2	1.24950	76.120	2.5
74	6	1	0	1.24380	76.532	1.9
75	1	4	4	1.23720	77.015	5.0
76	5	1	3	1.23480	77.192	4.7
77	3	0	5	1.22830	77.677	0.3
78	1	6	1	1.22400	78.001	0.7
79	2	5	2	1.22110	78.222	5.0
80	2	2	5	1.18860	80.793	0.2
81	5	0	4	1.18400	81.172	0.5
82	4	4	0	1.17730	81.732	1.7
83	1	6	2	1.16980	82.369	0.5
84	6	0	3	1.16980	82.369	0.5
85	3	5	0	1.16520	82.766	0.6
86	4	4	1	1.16040	83.184	1.3
87	3	3	4	1.16040	83.184	1.3
88	4	3	3	1.15780	83.413	3.7
89	2	4	4	1.14820	84.269	3.8
90	0	0	6	1.14820	84.269	3.8
91	4	0	5	1.14110	84.916	0.1
92	5	2	3	1.13510	85.472	1.3
93	1	1	6	1.11480	87.415	3.8
94	4	4	2	1.11480	87.415	3.8
95	2	3	5	1.10900	87.989	2.2
96	3	5	2	1.10370	88.522	5.9
97	1	6	3	1.09350	89.568	0.3
98	4	1	5	1.08900	90.039	0.1
99	1	7	0	1.08040	90.955	1.7
100	6	2	2	1.07460	91.586	0.9
101	1	7	1	1.06680	92.451	0.3
102	6	0	4	1.06680	92.451	0.3
103	4	3	4	1.05780	93.473	0.4
104	5	0	5	1.05220	94.123	0.3
105	4	4	3	1.04750	94.677	0.1
106	4	5	0	1.04430	95.059	0.1
107	5	2	4	1.04040	95.529	1.0
108	7	0	3	1.03900	95.699	0.5
109	3	3	5	1.03510	96.177	0.9
110	4	5	1	1.03250	96.499	1.7
111	2	2	6	1.03140	96.636	2.4
112	7	1	2	1.03140	96.636	2.4
113	2	4	5	1.02690	97.202	0.8
114	1	3	6	1.02330	97.660	1.4

115	8	0	0	1.01960	98.137	0.9
116	6	3	1	1.01640	98.554	2.3
117	6	2	3	1.01460	98.790	1.2
118	1	6	4	1.00810	99.656	2.0
119	5	1	5	1.00330	100.307	1.8
120	4	0	6	1.00000	100.762	1.0
121	4	5	2	1.00000	100.762	1.0
122	7	2	0	0.99650	101.249	0.7
123	7	2	1	0.98620	102.719	0.5
124	3	6	2	0.98470	102.938	2.9
125	8	0	2	0.97760	103.989	3.0
126	1	7	3	0.97760	103.989	3.0
127	4	4	4	0.97170	104.884	2.2
128	3	5	4	0.96490	105.940	0.8
129	4	1	6	0.96490	105.940	0.8
130	1	1	7	0.96270	106.288	0.4
131	4	3	5	0.96060	106.623	0.9
132	8	1	0	0.95460	107.595	0.1
133	5	4	3	0.95050	108.273	0.6
134	5	2	5	0.94760	108.760	0.7
135	8	1	1	0.94520	109.167	0.9
136	2	6	4	0.94520	109.167	0.9
137	5	5	0	0.94180	109.751	0.5
138	5	0	6	0.93850	110.325	1.1
139	6	3	3	0.93790	110.431	1.2

Stick Pattern



Name and formula

Reference code:	01-073-9797
Mineral name:	Fluorapatite
Compound name:	Calcium Iron Phosphate Fluorine Hydroxide
Common name:	Calcium iron phosphate(V) fluoride hydroxide
PDF index name:	Calcium Iron Phosphate Fluorine Hydroxide
Empirical formula:	$Ca_{9.8}F_{1.6}Fe_{0.2}H_{0.4}O_{24.4}P_6$
Chemical formula:	$(Ca_{9.8}Fe_{0.2}) (PO_4)_6 (F_{1.6} (OH)_{0.4})$

Crystallographic parameters

Crystal system:	Monoclinic
Space group:	P21/m
Space group number:	11
a (Å):	9.3260
b (Å):	9.3960
c (Å):	6.8460
Alpha (°):	90.0000
Beta (°):	90.0000
Gamma (°):	119.9500
Volume of cell (10^6 pm^3):	519.79
Z:	1.00
RIR:	0.57

Subfiles and Quality

Subfiles:	ICSD Pattern Inorganic Mineral
Quality:	Blank (B)

Comments

ANX:	A3B5XY12
Creation Date:	11/20/2008
Modification Date:	1/21/2009
ANX:	A3B5XY12
Analysis:	H0.4 Ca9.8 F1.6 Fe0.2 O24.4 P6
Formula from original source:	(Ca9.8 Fe0.2) (P O4)6 (F1.6 (O H)0.4)
ICSD Collection Code:	77139
Significant Warning:	ICSD Warning on inter-atomic distances exist
Wyckoff Sequence:	f5 e16(P1121/M)
Unit Cell Data Source:	Single Crystal.

References

Primary reference:	*Calculated from ICSD using POWD-12++*
Structure:	Hughes, J.M., Fransolet, A.-M., Schreyer, W., *Neues Jahrb. Mineral., Monatsh.*, **1993**, 504, (1993)

Peak list

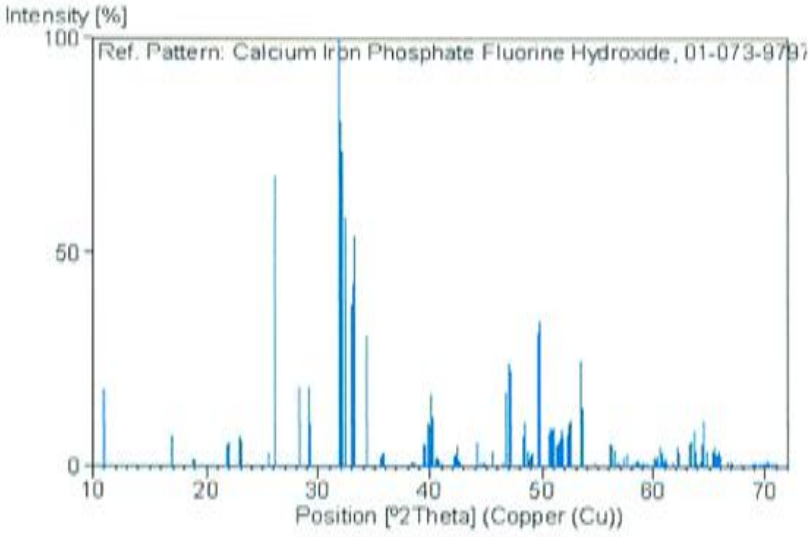
No.	h	k	l	d [A]	2Theta[deg]	I [%]
1	-1	1	0	8.08060	10.940	17.8
2	1	0	0	8.08060	10.940	17.8
3	-1	1	1	5.22990	16.940	7.2
4	1	0	1	5.22990	16.940	7.2
5	-1	2	0	4.69790	18.874	2.1
6	1	1	0	4.69790	18.874	2.1
7	-2	1	0	4.66300	19.017	1.6
8	0	2	0	4.07060	21.816	4.2
9	-2	2	0	4.05230	21.916	5.2
10	2	0	0	4.04030	21.982	5.9
11	-1	2	1	3.87360	22.940	7.1
12	1	1	1	3.87360	22.940	7.1
13	-2	1	1	3.85390	23.059	6.3
14	-2	2	1	3.48720	25.523	3.1
15	2	0	1	3.47950	25.580	3.3
16	0	0	2	3.42300	26.010	68.1
17	0	1	2	3.15540	28.260	18.7
18	1	0	2	3.15540	28.260	18.7
19	-2	3	0	3.07180	29.046	18.7
20	1	2	0	3.07180	29.046	18.7
21	-3	2	0	3.05160	29.242	10.1
22	-3	1	0	3.05160	29.242	10.1
23	-2	3	1	2.80210	31.912	100.0
24	1	2	1	2.80210	31.912	100.0
25	2	1	1	2.79470	31.999	80.8
26	-3	2	1	2.78720	32.087	73.9
27	-3	1	1	2.78720	32.087	73.9
28	-1	2	2	2.76650	32.334	55.2
29	1	1	2	2.76650	32.334	55.2
30	-2	1	2	2.75930	32.421	58.2
31	0	3	0	2.71380	32.980	37.8
32	-3	3	0	2.70150	33.134	42.6
33	3	0	0	2.69350	33.235	54.0
34	-2	2	2	2.61170	34.308	30.7
35	2	0	2	2.61170	34.308	30.7
36	0	3	1	2.52280	35.557	2.5
37	-3	3	1	2.51290	35.701	2.9
38	3	0	1	2.50650	35.796	3.5
39	-2	4	0	2.34900	38.286	0.2
40	2	2	0	2.34200	38.405	0.2
41	-4	2	0	2.33150	38.585	0.2
42	-2	3	2	2.28600	39.384	5.4
43	1	2	2	2.28600	39.384	5.4
44	-3	2	2	2.27780	39.532	5.1
45	-3	1	2	2.27780	39.532	5.1
46	-1	4	0	2.25880	39.878	10.2
47	1	3	0	2.25510	39.946	9.7
48	-3	4	0	2.25510	39.946	9.7
49	3	1	0	2.24480	40.138	17.0
50	-4	3	0	2.24480	40.138	17.0
51	-4	1	0	2.23940	40.239	11.6
52	-2	4	1	2.22180	40.571	2.0
53	2	2	1	2.21590	40.684	2.5
54	-4	2	1	2.20700	40.855	1.7
55	0	1	3	2.19730	41.044	0.6
56	1	0	3	2.19730	41.044	0.6

57	-1	4	1	2.14500	42.092	2.6
58	1	3	1	2.13960	42.203	3.1
59	-3	4	1	2.13960	42.203	3.1
60	3	1	1	2.13190	42.363	5.2
61	-4	3	1	2.13190	42.363	5.2
62	-4	1	1	2.12840	42.436	3.9
63	0	3	2	2.12840	42.436	3.9
64	-3	3	2	2.12060	42.599	1.5
65	3	0	2	2.11680	42.680	1.0
66	1	1	3	2.05150	44.108	5.7
67	-2	1	3	2.05150	44.108	5.7
68	0	4	0	2.03530	44.478	0.8
69	-4	4	0	2.02610	44.691	0.8
70	4	0	0	2.02020	44.828	1.0
71	0	2	3	1.99060	45.532	3.8
72	2	0	3	1.99060	45.532	3.8
73	0	4	1	1.95090	46.512	0.6
74	4	0	1	1.93680	46.871	17.6
75	-2	4	2	1.93680	46.871	17.6
76	2	2	2	1.93290	46.971	24.0
77	-4	2	2	1.92700	47.124	22.4
78	1	3	2	1.88160	48.332	7.2
79	-3	4	2	1.88160	48.332	7.2
80	3	1	2	1.87710	48.456	10.6
81	-4	1	2	1.87710	48.456	10.6
82	-2	5	0	1.86770	48.715	3.8
83	2	3	0	1.86300	48.846	1.8
84	3	2	0	1.85940	48.947	2.7
85	-5	3	0	1.85380	49.105	3.4
86	-5	2	0	1.85380	49.105	3.4
87	-2	3	3	1.83170	49.737	31.2
88	1	2	3	1.83170	49.737	31.2
89	-3	2	3	1.82750	49.859	34.0
90	-3	1	3	1.82750	49.859	34.0
91	-2	5	1	1.80190	50.617	7.8
92	-3	5	1	1.80190	50.617	7.8
93	2	3	1	1.79760	50.747	9.2
94	3	2	1	1.79440	50.844	8.4
95	-5	3	1	1.78940	50.996	9.1
96	-5	2	1	1.78940	50.996	9.1
97	-1	5	0	1.77720	51.371	5.2
98	1	4	0	1.77490	51.443	5.3
99	-4	5	0	1.77150	51.549	6.4
100	-5	4	0	1.76550	51.737	8.4
101	4	1	0	1.76550	51.737	8.4
102	-5	1	0	1.76210	51.844	6.9
103	0	4	2	1.74940	52.249	7.5
104	-4	4	2	1.74360	52.436	10.0
105	-3	3	3	1.74360	52.436	10.0
106	3	0	3	1.73980	52.559	10.9
107	4	0	2	1.73980	52.559	10.9
108	0	0	4	1.71150	53.497	24.8
109	4	1	1	1.71150	53.497	24.8
110	-5	1	1	1.70650	53.666	13.6
111	-1	1	4	1.67440	54.780	0.9
112	1	0	4	1.67440	54.780	0.9
113	-3	5	2	1.63800	56.103	5.3
114	-2	4	3	1.63800	56.103	5.3
115	2	3	2	1.63640	56.163	5.2
116	2	2	3	1.63390	56.257	3.5

117	3	2	2	1.63390	56.257	3.5
118	-5	2	2	1.62910	56.437	4.1
119	0	5	0	1.62910	56.437	4.1
120	-5	5	0	1.62090	56.749	0.4
121	5	0	0	1.61610	56.933	0.3
122	1	3	3	1.60400	57.402	2.3
123	-3	4	3	1.60400	57.402	2.3
124	-4	3	3	1.59830	57.626	3.1
125	-4	1	3	1.59830	57.626	3.1
126	0	5	1	1.58410	58.191	0.9
127	-5	5	1	1.57730	58.467	1.2
128	1	4	2	1.57730	58.467	1.2
129	-4	5	2	1.57290	58.646	1.6
130	5	0	1	1.57290	58.646	1.6
131	-5	4	2	1.56910	58.802	0.7
132	4	1	2	1.56910	58.802	0.7
133	-5	1	2	1.56600	58.930	0.7
134	-3	6	0	1.56600	58.930	0.7
135	3	3	0	1.56130	59.125	0.7
136	-6	3	0	1.55430	59.418	0.4
137	-2	6	0	1.53890	60.073	2.4
138	-4	6	0	1.53590	60.203	1.6
139	2	4	0	1.53590	60.203	1.6
140	4	2	0	1.53070	60.428	2.7
141	-3	6	1	1.52660	60.608	4.7
142	-6	2	0	1.52660	60.608	4.7
143	3	3	1	1.52220	60.801	3.5
144	0	4	3	1.51890	60.947	1.4
145	-6	3	1	1.51580	61.085	2.2
146	-4	4	3	1.51580	61.085	2.2
147	4	0	3	1.51260	61.229	1.2
148	-2	6	1	1.50140	61.735	1.2
149	-3	2	4	1.49320	62.112	4.7
150	-3	1	4	1.49320	62.112	4.7
151	-6	4	1	1.49100	62.213	3.3
152	0	5	2	1.47040	63.185	5.5
153	-5	5	2	1.46500	63.445	6.1
154	5	0	2	1.46140	63.619	8.5
155	1	5	0	1.46140	63.619	8.5
156	-5	6	0	1.45760	63.804	3.9
157	-6	5	0	1.45370	63.996	0.8
158	5	1	0	1.45370	63.996	0.8
159	-6	1	0	1.45040	64.159	1.4
160	0	3	4	1.44760	64.298	5.5
161	3	0	4	1.44460	64.448	10.9
162	2	3	3	1.44460	64.448	10.9
163	-5	2	3	1.43820	64.769	3.8
164	-1	6	1	1.42900	65.238	3.7
165	1	5	1	1.42900	65.238	3.7
166	-5	6	1	1.42560	65.413	4.9
167	-3	6	2	1.42560	65.413	4.9
168	-6	5	1	1.42200	65.599	2.9
169	5	1	1	1.42200	65.599	2.9
170	3	3	2	1.42050	65.677	2.6
171	-6	1	1	1.41890	65.761	3.9
172	-6	3	2	1.41520	65.954	2.6
173	-1	5	3	1.40220	66.645	1.3
174	1	4	3	1.40220	66.645	1.3
175	-5	4	3	1.39650	66.953	1.3
176	-6	4	2	1.39650	66.953	1.3

177	-6	2	2	1.39360	67.111	0.9
178	-4	2	4	1.37970	67.878	0.1
179	1	3	4	1.36330	68.808	0.3
180	-3	4	4	1.36330	68.808	0.3
181	-4	3	4	1.35980	69.010	0.3
182	-4	1	4	1.35980	69.010	0.3
183	0	6	0	1.35690	69.179	0.3
184	-6	6	0	1.35080	69.536	0.3
185	1	0	5	1.35080	69.536	0.3
186	6	0	0	1.34680	69.772	0.4
187	-1	6	2	1.34500	69.879	0.8
188	1	5	2	1.34380	69.951	0.7
189	-5	6	2	1.34110	70.112	0.9
190	-6	5	2	1.33810	70.293	1.6
191	-4	7	0	1.33810	70.293	1.6
192	-6	1	2	1.33550	70.450	1.1
193	3	4	0	1.33550	70.450	1.1
194	0	6	1	1.33100	70.724	0.4
195	-7	4	0	1.32750	70.938	0.3
196	-7	3	0	1.32750	70.938	0.3
197	0	5	3	1.32540	71.068	0.2
198	-6	6	1	1.32540	71.068	0.2

Stick Pattern



Name and formula

Reference code:	01-086-2342
Compound name:	Calcium Carbonate
Common name:	calcite high
PDF index name:	Calcium Carbonate
Empirical formula:	$CCaO_3$
Chemical formula:	$Ca(CO_3)$

Crystallographic parameters

Crystal system:	Rhombohedral
Space group:	R-3c
Space group number:	167
a (Å):	4.9780
b (Å):	4.9780
c (Å):	17.4620
Alpha (°):	90.0000
Beta (°):	90.0000
Gamma (°):	120.0000
Volume of cell (10^6 pm^3):	374.74
Z:	6.00
RIR:	2.72

Status, subfiles and quality

Status:	Diffraction data collected at non ambient temperature Alternate Pattern
Subfiles:	Excipient ICSD Pattern Inorganic Mineral Pharmaceutical
Quality:	Star (S)

Comments

ANX:	ABX3
Creation Date:	11/20/2008
Modification Date:	1/21/2009
ANX:	ABX3
Analysis:	C1 Ca1 O3
Formula from original source:	Ca (C O3)
ICSD Collection Code:	40115
Calculated Pattern Original Remarks:	Also rigid body refinement of CO3-group (R=0.052). Stable up to 1260 K (3rd ref., Tomaszewski), above R3-m, m.p. 1520 K
Temperature of Data Collection:	1023 K
Wyckoff Sequence:	e b a(R3-CH)
Unit Cell Data Source:	Single Crystal.

References

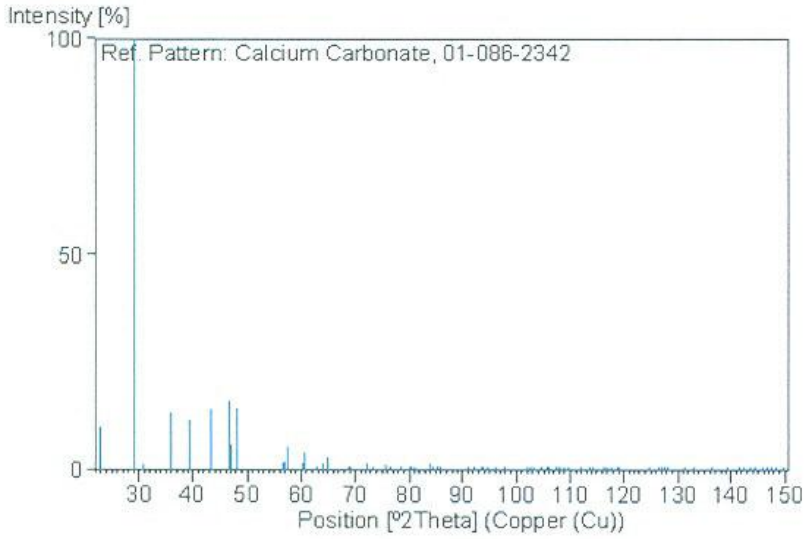
Primary reference: *Calculated from ICSD using POWD-12++*, (2004)
Structure: Markgraf, S.A., Reeder, R.J., *Am. Mineral.*, **70**, 590, (1985)

Peak list

No.	h	k	l	d [A]	2Theta[deg]	I [%]
1	0	1	2	3.86550	22.989	9.8
2	1	0	4	3.06740	29.088	100.0
3	0	0	6	2.91030	30.696	1.4
4	1	1	0	2.48900	36.056	13.5
5	1	1	3	2.28850	39.339	11.8
6	2	0	2	2.09270	43.196	14.2
7	0	1	8	1.94740	46.601	16.0
8	0	2	4	1.93280	46.974	5.9
9	1	1	6	1.89160	48.061	14.4
10	2	1	1	1.62240	56.691	1.8
11	1	0	10	1.61850	56.840	1.9
12	1	2	2	1.60180	57.488	5.3
13	2	0	8	1.53370	60.298	1.2
14	1	1	9	1.53020	60.450	1.7
15	2	1	4	1.52660	60.608	4.0
16	1	2	5	1.47660	62.889	1.0
17	0	0	12	1.45520	63.922	1.6
18	3	0	0	1.43700	64.830	2.9
19	2	1	7	1.36420	68.756	0.4
20	0	2	10	1.35680	69.185	0.7
21	1	2	8	1.30570	72.307	1.8
22	0	3	6	1.28850	73.429	0.5
23	1	1	12	1.25620	75.642	1.2
24	2	2	0	1.24450	76.481	0.9
25	2	2	3	1.21700	78.536	0.1
26	0	1	14	1.19820	80.014	0.1
27	2	1	10	1.19130	80.572	0.9
28	3	1	2	1.18460	81.123	0.2
29	1	3	4	1.15320	83.821	1.7
30	2	2	6	1.14430	84.623	0.5
31	1	2	11	1.13700	85.295	0.1
32	3	1	5	1.13120	85.837	0.1
33	2	0	14	1.07960	91.042	0.3
34	0	4	2	1.06960	92.138	0.1
35	1	0	16	1.05800	93.450	0.3
36	1	1	15	1.05450	93.855	0.2
37	3	1	8	1.04860	94.547	1.0
38	4	0	4	1.04640	94.808	1.1
39	2	1	13	1.03650	96.005	0.1
40	0	3	12	1.02250	97.763	0.6
41	1	2	14	0.99040	102.113	0.2
42	3	2	1	0.98740	102.545	0.1
43	1	3	10	0.98660	102.661	0.1
44	2	3	2	0.98270	103.231	0.2
45	0	2	16	0.97370	104.578	0.2
46	0	4	8	0.96640	105.705	0.4
47	3	2	4	0.96460	105.987	0.4
48	3	1	11	0.95510	107.513	0.1
49	2	3	5	0.95160	108.090	0.1
50	2	2	12	0.94580	109.065	0.3
51	4	1	0	0.94080	109.924	0.4
52	1	4	3	0.92870	112.082	0.1

53	3	2	7	0.91940	113.823	0.1
54	4	0	10	0.91710	114.266	0.1
55	2	1	16	0.90680	116.308	0.2
56	1	1	18	0.90390	116.903	0.1
57	2	3	8	0.90090	117.527	0.2
58	1	4	6	0.89510	118.762	0.1
59	1	3	13	0.89310	119.197	0.1
60	1	2	17	0.86890	124.879	0.1
61	3	1	14	0.86310	126.373	0.1
62	3	2	10	0.86060	127.036	0.1
63	5	0	2	0.85800	127.737	0.1
64	0	1	20	0.85570	128.368	0.1
65	0	5	4	0.84590	131.184	0.1
66	2	3	11	0.83940	133.178	0.1
67	3	3	0	0.82970	136.376	0.1
68	3	3	3	0.82140	139.367	0.1
69	0	4	14	0.81550	141.669	0.1
70	2	4	1	0.81380	142.364	0.1
71	4	2	2	0.81120	143.457	0.1
72	2	0	20	0.80920	144.325	0.1
73	1	3	16	0.80610	145.720	0.2
74	0	3	18	0.80400	146.705	0.1
75	5	0	8	0.80190	147.723	0.1
76	2	4	4	0.80090	148.222	0.1
77	2	1	19	0.80090	148.222	0.1
78	3	3	6	0.79790	149.772	0.1

Stick Pattern



Name and formula

Reference code:	01-073-9697
Mineral name:	Carbonate-fluorapatite
Compound name:	Calcium Phosphate Fluoride Carbonate
PDF index name:	Calcium Phosphate Fluoride Carbonate
Empirical formula:	$C_{0.53}Ca_{9.74}F_{2.05}O_{23.39}P_{5.45}$
Chemical formula:	$Ca_{9.74}(PO_4)_{5.45}F_{2.05}(CO_3)_{0.53}$

Crystallographic parameters

Crystal system:	Hexagonal
Space group:	P63/m
Space group number:	176
a (Å):	9.3593
b (Å):	9.3593
c (Å):	6.8919
Alpha (°):	90.0000
Beta (°):	90.0000
Gamma (°):	120.0000
Volume of cell (10^6 pm^3):	522.82
Z:	1.00
RIR:	1.01

Subfiles and Quality

Subfiles:	ICSD Pattern Inorganic Mineral
Quality:	Indexed (I)

Comments

Creation Date:	11/19/2008
Modification Date:	1/21/2009
Analysis:	C0.53 Ca9.74 F2.05 O23.39 P5.45
Formula from original source:	Ca9.74 (P O4)5.45 F2.05 (C O3).53
ICSD Collection Code:	71856
Calculated Pattern Original Remarks:	Sample tempered at 1023 K
Test from external database:	A site occupation is implausible or meaningless but agrees with the paper
Minor Warning:	Minor test comments from ICSD exist. Reported site occupancy factor>1.0
Wyckoff Sequence:	i h5 f a(P63/M)
Unit Cell Data Source:	Powder Diffraction.

References

Primary reference:	*Calculated from ICSD using POWD-12++*
Structure:	Perdikatsis, B., *Materials Science Forum*, **79**, 809, (1991)

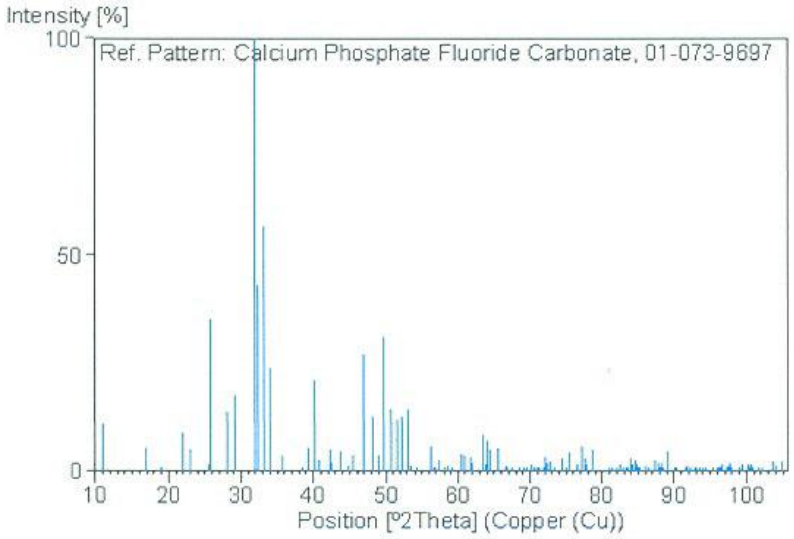
Peak list

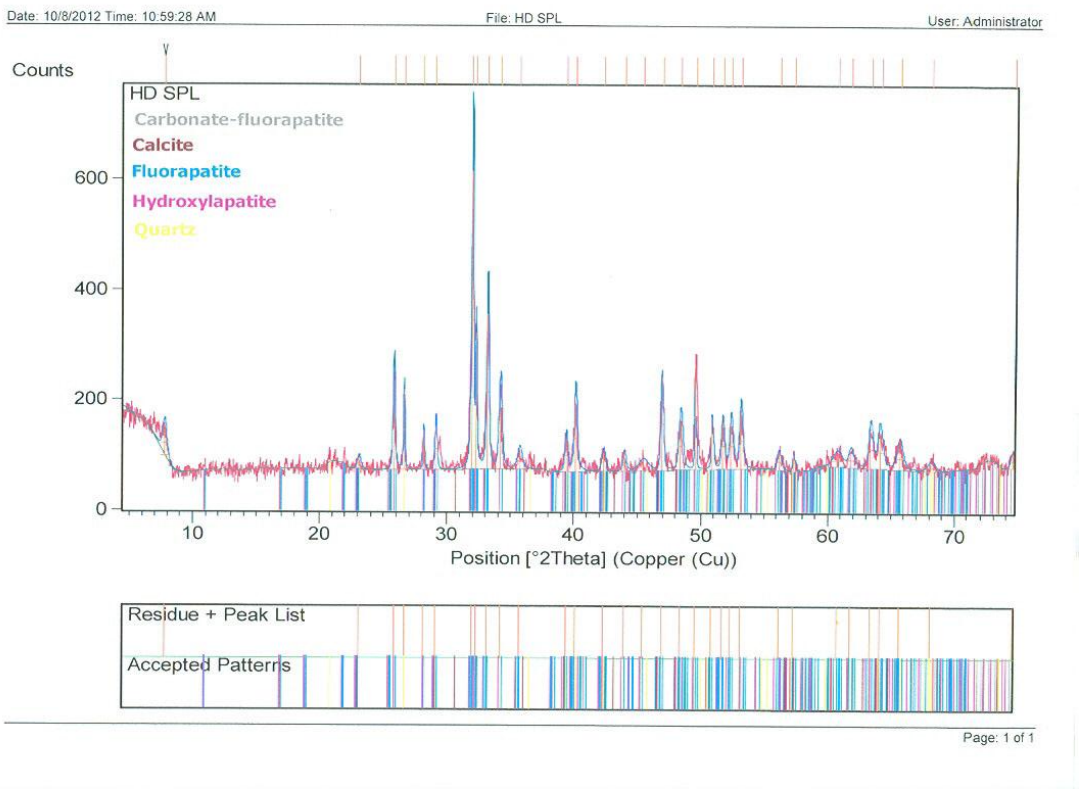
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4	2	0	0	4.05270	21.914	8.8
5	1	1	1	3.87150	22.953	5.0
6	2	0	1	3.49350	25.476	1.8
7	0	0	2	3.44600	25.833	35.1
8	1	0	2	3.17120	28.116	13.7
9	2	1	0	3.06360	29.125	17.4
10	2	1	1	2.79940	31.944	100.0
11	1	1	2	2.77480	32.235	43.0
12	3	0	0	2.70180	33.130	56.7
13	2	0	2	2.62520	34.126	23.9
14	3	0	1	2.51540	35.665	3.6
15	2	2	0	2.33980	38.442	0.4
16	2	1	2	2.28960	39.320	5.4
17	1	3	0	2.24800	40.078	21.0
18	2	2	1	2.21560	40.690	2.6
19	1	0	3	2.21020	40.794	1.8
20	1	3	1	2.13720	42.253	5.1
21	3	0	2	2.12620	42.482	1.9
22	1	1	3	2.06220	43.867	4.9
23	4	0	0	2.02640	44.684	1.2
24	2	0	3	1.99850	45.342	3.7
25	2	2	2	1.93580	46.897	26.9
26	1	3	2	1.88280	48.300	12.6
27	2	3	0	1.85950	48.944	3.7
28	2	1	3	1.83790	49.558	31.0
29	3	2	1	1.79530	50.816	14.6
30	1	4	0	1.76870	51.636	11.9
31	4	0	2	1.74670	52.336	12.6
32	0	0	4	1.72300	53.112	14.3
33	4	1	1	1.71320	53.439	1.3
34	1	0	4	1.68530	54.396	0.4
35	3	2	2	1.63640	56.163	5.9
36	5	0	0	1.62110	56.741	0.3
37	1	1	4	1.61690	56.902	0.5
38	3	1	3	1.60670	57.296	2.7
39	2	0	4	1.58560	58.131	0.2
40	5	0	1	1.57800	58.438	1.3
41	4	1	2	1.57360	58.617	1.2
42	3	3	0	1.55990	59.183	0.6
43	2	4	0	1.53180	60.380	4.1
44	3	3	1	1.52140	60.837	3.6
45	4	0	3	1.52140	60.837	3.6
46	1	2	4	1.50180	61.717	3.3
47	2	4	1	1.49530	62.015	2.0
48	5	0	2	1.46690	63.353	8.5
49	5	1	0	1.45580	63.893	1.6
50	3	0	4	1.45270	64.045	7.1
51	3	2	3	1.44540	64.408	5.2
52	5	1	1	1.42430	65.480	5.4
53	3	3	2	1.42110	65.646	4.2
54	4	1	3	1.40150	66.683	1.4
55	4	2	2	1.39970	66.780	1.1
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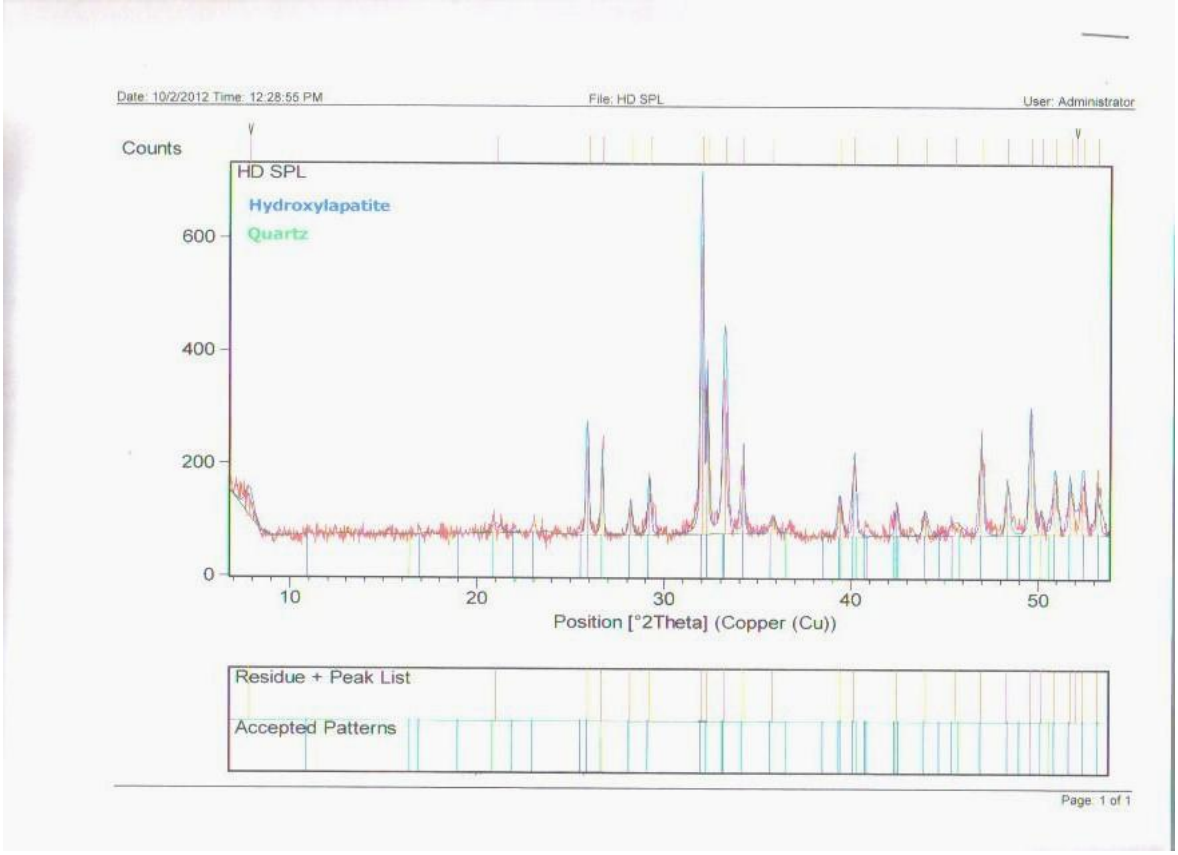
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63	5	0	3	1.32450	71.123	0.2
64	1	1	5	1.32220	71.266	0.3
65	4	0	4	1.31260	71.868	0.5
66	4	3	1	1.30830	72.141	3.4
67	2	0	5	1.30500	72.352	1.9
68	5	2	0	1.29790	72.811	2.5
69	3	3	3	1.29050	73.296	0.3
70	2	5	1	1.27440	74.377	3.1
71	2	4	3	1.27440	74.377	3.1
72	2	3	4	1.26380	75.108	0.8
73	2	1	5	1.25700	75.586	4.5
74	3	4	2	1.24280	76.605	1.7
75	6	1	0	1.23610	77.096	2.3
76	1	4	4	1.23420	77.237	5.7
77	5	1	3	1.22970	77.572	3.2
78	3	0	5	1.22780	77.715	1.5
79	1	6	1	1.21660	78.567	1.2
80	2	5	2	1.21460	78.721	5.2
81	2	2	5	1.18760	80.875	0.3
82	5	0	4	1.18070	81.447	0.6
83	1	3	5	1.17510	81.918	0.5
84	4	4	0	1.16990	82.361	1.5
85	1	6	2	1.16350	82.913	0.6
86	3	5	0	1.15790	83.404	0.6
87	3	3	4	1.15640	83.536	0.7
88	4	3	3	1.15260	83.874	3.1
89	0	0	6	1.14860	84.233	1.5
90	2	4	4	1.14480	84.578	2.8
91	5	3	1	1.14190	84.843	1.5
92	4	0	5	1.13970	85.045	0.2
93	1	0	6	1.13730	85.267	0.4
94	5	2	3	1.13000	85.950	1.4
95	2	6	0	1.12400	86.522	0.3
96	1	1	6	1.11550	87.346	2.6
97	5	1	4	1.11200	87.691	1.9
98	2	6	1	1.10940	87.949	1.0
99	2	3	5	1.10730	88.159	2.1
100	2	0	6	1.10510	88.380	1.1
101	5	3	2	1.09760	89.144	4.8
102	1	6	3	1.08850	90.091	0.4
103	4	1	5	1.08720	90.229	0.1
104	1	2	6	1.07550	91.487	0.2
105	1	7	0	1.07360	91.696	1.3
106	6	2	2	1.06860	92.249	0.8
107	6	0	4	1.06310	92.868	0.4
108	7	1	1	1.06080	93.129	0.2
109	3	0	6	1.05710	93.554	0.1
110	4	3	4	1.05410	93.901	0.3
111	5	0	5	1.05010	94.370	0.3
112	4	4	3	1.04250	95.275	0.1
113	5	2	4	1.03670	95.980	0.7
114	7	0	3	1.03400	96.313	0.7
115	3	3	5	1.03290	96.450	1.0
116	2	2	6	1.03110	96.674	1.8
117	4	5	1	1.02620	97.291	0.9
118	7	1	2	1.02500	97.443	1.4

119	2	4	5	1.02500	97.443	1.4
120	1	3	6	1.02290	97.711	1.9
121	3	6	0	1.02120	97.930	0.8
122	8	0	0	1.01320	98.975	0.6
123	6	3	1	1.01020	99.374	1.7
124	2	6	3	1.01020	99.374	1.7
125	1	6	4	1.00430	100.171	1.8
126	8	0	1	1.00240	100.431	1.1
127	5	1	5	1.00090	100.637	1.5
128	4	0	6	0.99930	100.859	1.1
129	5	4	2	0.99370	101.643	0.6
130	7	2	0	0.99020	102.142	0.5
131	3	6	2	0.97910	103.764	2.2
132	3	2	6	0.97720	104.049	1.2
133	8	0	2	0.97200	104.838	2.2
134	1	7	3	0.97200	104.838	2.2

Stick Pattern




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HD SPL
Carbonate-fluorapatite
Calcite
Fluorapatite
Hydroxylapatite
Quartz
600
400
200
0
10
20
30
40
50
60
70
Position [°2Theta] (Copper (Cu))
Residue + Peak List
Accepted Patterns
Page: 1 of 1


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HD SPL
Hydroxylapatite
Quartz
600
400
200
0
10
20
30
40
50
Position [°2Theta] (Copper (Cu))
Residue + Peak List
Accepted Patterns
Page: 1 of 1

APPENDIX 3

Chemical Analysis Result of Sokoto Phosphate Rock Deposit Run-of-Mine

OXIDES	% CONTENT
Al_2O_3	10.50
SiO_2	44.30
P_2O_5	12.10
SO_3	0.14
K_2O	0.34
Na_2O	2.42
CaO	19.10
MgO	0.73
TiO_2	2.44
V_2O_5	0.11
MnO	0.34
F^-	2.24
Fe_2O_3	3.91
Eu_2O_3	0.23

APPENDIX 4

Chemical Analysis Result of Sorted Sokoto Phosphate Nodules

OXIDES	% CONTENT
Al2O3	3.60
SiO2	14.20
P2O5	21.70
SO3	0.36
CaO	44.31
MgO	0.45
TiO2	0.83
V2O5	0.05
MnO	0.59
Fe2O3	6.1.
In2O3	0.41
BaO	0.22
Nd2O3	0.09
HgO	0.68
LOI	6.32

APPENDIX 5

Chemical Analysis Result of Flotation Tests on Sokoto Phosphate Rock

S/No.	SAMPLE ID	P2O5 CONTENT (%)
1.	F/38/55%/PH8/0.2ml/FA	19.00
2.	T/38/55%/PH8/0.2ml/FA	20.90
3.	F/38/55%/PH8/0.4ml/FA	20.00
4.	T/38/55%/PH8/0.4ml/FA	20.80
5.	F/38/55%/PH10/0.2ml/FA	20.40
6.	T/38/55%/PH10/0.2ml/FA	21.40
7.	F/38/55%/PH10/0.4ml/FA	19.90
8.	T/38/55%/PH10/0.4ml/FA	21.30
9.	F/38/65%/PH8/0.2ml/FA	20.30
10.	T/38/65%/PH8/0.2ml/FA	21.10
11.	F/38/65%/PH8/0.4ml/FA	20.40
12.	T/38/65%/PH8/0.4ml/FA	21.60
13.	F/38/65%/PH10/0.2ml/FA	18.50
14.	T/38/65%/PH10/0.2ml/FA	19.30
15.	F/38/65%/PH10/0.4ml/FA	20.10
16.	T/38/65%/PH10/0.4ml/FA	21.20
17.	F/180/800HA/PH9/60%	21.20
18.	T/180/800HA/PH9/60%	22.9
19.	F/106/800HA/PH9/60%	21.700
20.	T/106/800HA/PH9/60%	22.30
21.	F/38/800HA/PH9/60%	19.40
22.	T/38/800HA/PH9/60%	20.60
23.	F/180/800FA/PH9/60%	19.40
24.	T/180/800FA/PH9/60%	21.70
25.	F/106/800FA/PH9/60%	20.40
26.	T/106/800FA/PH9/60%	22.10
27.	F/38/800FA/PH9/60%	19.90
28.	T/38/800FA/PH9/60%	20.90
29.	F1/38/800/FA	23.00
30.	F2/38/800/FA/2	22.30
31.	F3/38/800/FA/2	22.10
32.	F4/38/800/FA/2	22.60
33.	F5/38/800/FA/2	23.10
34.	F6/38/800/FA	21.40
35.	38/800/FA/T	22.40
36.	+38-106um/SS-AC/FA/PH9/F	20.60
37.	+38-106um/SS-AC/FA/PH9/T	21.20
38.	+38-106um/SS-AC/HA/PH9/F	20.20
39.	+38-106um/SS-AC/HA/PH9/T	21.70
40.	+38-106um/Di-FA/PH9/F	20.60
41.	+38-106um/Di-FA/PH9/T	21.00