

SURFACE CHEMISTRY AND FLOTATION BEHAVIOR OF MONAZITE, APATITE, ILMENITE, QUARTZ, RUTILE, AND ZIRCON WITH OCTANOHYDROXAMIC ACID

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Key Words: Flotation, monazite, apatite, ilmenite, quartz, rutile, zircon.

Global increase in rare earth demand and consumption has led to a further understanding of their beneficiation and recovery. Monazite is the second most important rare earth mineral that can be further exploited. In this study, the surface chemistry of monazite in terms of zeta potential, adsorption density, and flotation response by microflotation using octanohydroxamic acid is determined. Apatite, ilmenite, quartz, rutile, and zircon are minerals that frequently occur with monazite among other minerals, hence were chosen as gangue minerals in this study. The Iso Electric Point (IEP) of monazite, apatite, ilmenite, quartz, rutile, and zircon are 5.3, 8.7, 3.8, 3.4, 6.3, and 50.1 respectively. Thermodynamic parameters of adsorption were evaluated. Ilmenite has highest driving force for adsorption. Adsorption density shows that hydroxamate adsorbs on to monazite and its gangue minerals. This observation was further confirmed by microflotation experiments. Increasing temperature to 80°C raises the adsorption and floatability of monazite and gangue minerals, which does not allow for separation. Appropriate use of depressant is recommended in order to achieve separation of monazite from its gangue.

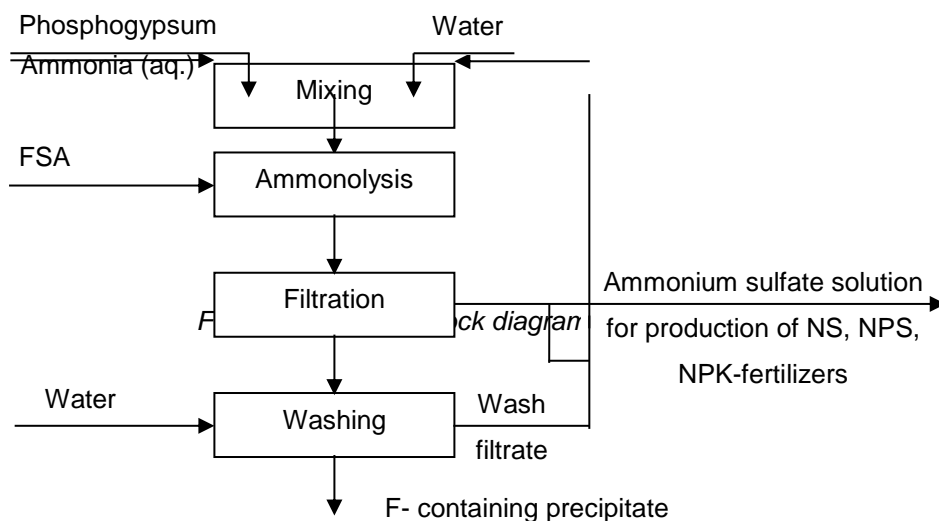
RESOURCE-SAVING TECHNOLOGY FOR PROCESSING BY-PRODUCTS OF THE WET PHOSPHORIC ACID PROCESS – PHOSPHOGYPSUM AND FLUOSILICIC ACID TO PRODUCE AMMONIUM SULFATE

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Key Words: fluosilicic acid; phosphogypsum; ammonium sulfate

At present time, many producers of phosphate fertilizers faced with the challenge of processing and recycling of fluosilicic acid solutions (FSA), obtained in the process of absorption fluorine-containing gases. It is a pollutant with a primary concern and cannot be landfilled without treatment. Existing best available techniques for processing FSA involves production of silicofluoride and fluoride salts which market is very limited. As a result, the acid neutralized with lime. This method of treatment ultimately increases the amount of solid waste sent for storage. A better environmental alternative of this method is the use of technological solutions, which can allow processing FSA with production of substances demanded by the chemical industry. NIUIF has developed a technique (Pat. RU 2462419) of co-processing by-products phosphogypsum and FSA with production of ammonium sulfate without intermediate separation of silica according to the process block diagram shown in Figure 1.



This process based on neutralization of the FSA by a phosphogypsum. Gypsum, the main component of phosphogypsum, used for precipitation of F in form of calcium fluoride. After neutralization and precipitation, the suspension separated into solution of ammonium sulfate and mixture of calcium fluoride, gypsum and silica usable in the cement industry. Water solution of ammonium sulfate with low content of F and silica can be used for production of NS, NP, NPS and NPK-fertilizers.

RECOVER OF HIGH PURITY CALCIUM SULFATE FROM PHOSPHOGYPSUM I: THERMODYNAMIC STUDY OF SO_4^{2-} PURIFICATION

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Key Words: Phosphogypsum; purification; thermodynamic analysis.

Chemical agents SO_4^{2-} and Ca^{2+} in phosphogypsum could be recycled to make high-purity calcium sulfate whisker. A key step in this process is to decompose phosphogypsum using NaOH solution to obtain Na_2SO_4 solution and $\text{Ca}(\text{OH})_2$ residue. In this decomposition process, thermodynamic analysis indicates that the majority of impurities reports to the residue phase $\text{Ca}(\text{OH})_2$, with minor amounts of Si and Al impurities end up in Na_2SO_4 solution in forms of Na_2SiO_3 and KAlO_2 . Based on phase diagram at 25°C for the Na_2SO_4 - SiO_3^{2-} - AlO_2^- system, Si and Al impurities may be removed via precipitation by adjusting pH value of the Na_2SO_4 solution. In verification tests on a sodium phosphate solution of pH 13.2 with 17.7mg/L of Al and 3.41mg/L of Si, when pH was adjusted to 12 no Al was detected in the solution with 8.48% Si removal. After solution pH was further lowered to 7, Al was still negligible in the liquid phase, but Si removal was increased to 75.89%..

DRY BENEFICIATION OF PHOSPHATE MINERALS USING A TRIBOELECTRIC BELT SEPARATOR

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Key Words: Electrostatic, Phosphate, Tribocharging, Dolomite, Dry

Beneficiation of phosphate ores by electrostatic processes has been demonstrated by various researchers since the 1940's. Separations have been achieved by removing silica from phosphates, and with fewer examples of calcite and dolomite from phosphate. Limitations on conventional electrostatic systems include low capacity, the need for multiple stages, and operational problems caused by fines. These limitations may be overcome by newer electrostatic processes, such as the triboelectric belt separator developed by ST Equipment & Technology LLC (STET).

In contrast to other electrostatic separation processes that are typically limited to particles greater than 75 μm in size, the STET triboelectric belt separator is ideally suited for separation of very fine (<1 μm) to moderately coarse (500 μm) particles, with very high throughput. The STET triboelectric belt separator technology has been used to process a wide range of industrial minerals and other dry granular powders. Recent bench-top testing using a laboratory scale STET separator has shown the technology to be effective in triboelectric charging and separation of several phosphate ores.

For phosphate ores containing a mostly silicate gangue mineralogy – the STET triboelectric belt separator may offer a high rate, lower cost processing solution, while minimizing or eliminating the need for flotation or other wet processing. For phosphates with more complex gangue mineralogy, or for carbonate / carbonate separations (such as those required for phosphate from dolomite or calcite) – the STET process may be considered as an additional beneficiation step following traditional wet processing steps.

REMOVAL OF COARSE FRACTION FROM PHOSPHATIC CLAYS FOR CLAY SETTLING AREA REDUCTION AND ADDITIONAL PHOSPHATE RECOVERY

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Key Words: Phosphate, dolomite, clay settling area, sizing, consolidation, flotation, BPL recovery

Ore deposits containing more clays and dolomite, and yielding relatively more concentrate than pebble will be encountered as mining advances into the southern extension of the central Florida phosphate district. The permissible clay settling area (CSA) is very limited. Therefore, the ultimate reserve realization would be limited because of lack of CSA storage capacity. The feasibility of increasing reserve recovery by reducing clay tonnages reporting to CSA and recovery of phosphates from the clay stream were investigated. A potential process has been identified that would increase the realized reserve by separating the coarser fractions ("Microfines") of the conventional waste clay stream and removing it, thus freeing up additional CSA storage capacity. The separated Microfines stream, being fine sand and silt, would be disposed with the conventional flotation tailings to mine cuts. In addition, this Microfine fraction has BPL value and can be processed with proven flotation techniques.

In this study, twenty two (22) holes from a southern deposit were selected, drilled and washed to generate -100 micron (conventional Florida phosphatic clays size) samples. The clay slurry from each hole were collected in a 2,000 gallon storage tank, then subjected to two stages sizing using a Krebs 4" gMax cyclone. The settling and consolidation rates of the overflows and the raw clays were determined to determine the potential storage volume reduction.

The feasibility of recovery of phosphate from the coarse fraction materials reported to the 2nd stage cyclone underflow with different flotation processes was explored.

The results showed:

1. Removal of +40 micron fraction from the -100 micron conventional clays could increase storage capacity by about 18.5%.
2. The coarse fraction recovered from the -100 micron clays contained about 12 BPL and 6.5 MgO. After desliming at 400 mesh, the resulting microfine flotation feed contained about 12 BPL and 3.5 MgO.
3. The most promising flotation process was found to be the modified Crago process which produced 62 BPL and 0.8 MgO final concentrate with about 73% BPL recovery and 97% MgO rejection.

UPGRADING PHOSPHOGYPSUM FOR USES AS CONSTRUCTION MATERIALS

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Key Words: Phosphogypsum, phosphate, flotation, gravity separation.

Over 70% of the world's phosphate rock product is consumed by the "wet" process for production of phosphoric acid, which is mainly used for fertilizer manufacturing. In this process, roughly 5 tons of phosphogypsum (PG) are generated per ton of P_2O_5 produced. On that basis, at least 150 million tons of PG are being added annually to the world stockpile of billions of tons. Although the Florida style stacking with lining is the state-of- art PG disposal practice and may stay dominant in the phosphate industry for years to come, this practice is neither cost effective nor environmentally sound. The phosphate companies, particularly those in China, are under great pressure to find uses for PG quickly. Use of PG as construction materials could ease the problem significantly. However, due to its high content of P and other gangue materials such as fluorides and silica, this line of PG use is severely limited. Under this research program, a combination of gravity separation with flotation produced an upgraded PG acceptable for many construction uses. The results summarized in Table 1 indicates this.

Table 1. PG Upgrading Results.

Component, wt%	CaO	P_2O_5	SiO_2
Feed PG	30.44	0.94	8.97
Upgraded PG	32.52	0.59	4.37

RARE EARTHS OCCURRENCE IN FLORIDA PHOSPHATE ORE AND THEIR FATE DURING MINING AND CHEMICAL PROCESSING

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Phosphate minerals have been identified as significant unconventional rare earths resources. World's identified phosphate resources total about 300 billion tons, representing 90 million tons of rare earth elements (REE), assuming an average REE content of 300 ppm. Under a project funded by the Critical Materials Institute (CMI), the Florida Industrial and Phosphate Research Institute (FIPR Institute) conducted detailed chemical and mineralogical characterization of REE in different phosphate mining and processing streams. The research project includes three major parts. Part I covers chemical analysis and some basic properties of different samples. Part II is a detailed process mineralogy study of the amine flotation tails. The third part focuses on isolation and characterization of REE mineral particles in three samples using two advanced techniques, dual energy (DE) rapid scan radiography and high resolution X-ray microtomography (HRXMT).

Five samples were collected from a central Florida phosphate operation, including amine flotation tails, waste clay, phosphate rock, phosphoric acid, wet phosphogypsum (PG), and phosphoric acid sludge. These samples were analyzed for rare earth elements, uranium, thorium, routine chemical compositions, and radioactivity. Results show total REE of 70-500 ppm in the samples with uranium ranging from 25-120 ppm. Radium-226 analyzed about 20 pCi/g in phosphogypsum, 28 in phosphate rock, and 0.2 in phosphoric acid, and the corresponding uranium-238 numbers are 2.8, 20 and 36 pCi/g. Simple sizing and chemical analysis of phosphogypsum revealed an extremely encouraging piece of information on REE in PG. About 65% of the REE in PG is concentrated in the minus 500 mesh (approximately 30 microns) fraction that represents less than 10% of the total PG mass. Another fact is that the finest fraction also contains most of the thorium but little uranium.

A detailed process mineralogy study was conducted on the amine flotation tails sample using a Mineral Liberation Analyzer, the most advanced instrument for this type of study. Two major rare earth minerals were detected in the amine tails including monazite and xenotime. The monazite monomers average 1.27% CaO, 13.73% La₂O₃, 29.28% Ce₂O₃, 12.26% Nd₂O₃, 0.63% UO₂, 6.2% ThO₂, 3.55% Pr₂O₃, 0.46% Al₂O₃, 1.69% SiO₂, and 30.92% P₂O₅. Xenotime is composed of the following chemicals: 46.44% Y₂O₃, 2.29% Gd₂O₃, 5.24% Dy₂O₃, 3.93% Yb₂O₃, 0.31% Nd₂O₃, 4.61% Er₂O₃, 0.66% Sm₂O₃, 1.06% UO₂, 0.19% CaO, and 35.26 P₂O₅. Other major minerals in the amine tails include quartz, fluoapatite, feldspar, rutile, pseudorutile and zircon.

In Part III, dual energy (DE) rapid scan radiography was used to first identify potential RE particles, followed by a more detailed quantified liberation analysis by high resolution X-ray microtomography (HRXMT). Three sample streams, Shaking Table Concentrate, Acid Plant Feed, and Phosphogypsum, were separated into three size classes: >106 μm, 75-106 μm, and 53-75 μm. DE radiographs were taken at two energy levels and the ratio calculated. The images were thresholded to show only potential rare earth particles and then those particles were removed to prepare HRXMT samples. The samples were digitally reconstructed and the concentration of rare earth particles found using digital processing software. Based on the degree of liberation, the best particle size to find fully liberated monazite particles is 75-106 μm, although other sizes can reasonably be considered for Acid Plant Feed and Phosphogypsum.

Based on chemical analyses and minerals characterization, the following conclusions may be made about the occurrence and fate of REE in phosphate mining and processing: 1) REE in the flotation tails exist primarily in monazite with some in xenotime and heavy minerals such as zircon; 2) In the phosphoric acid manufacturing process over 70% of the REE in phosphate rock is dissolved, but a majority (about 70%) of which is either re-precipitated with PG or get absorbed onto PG; 3) the REE in phosphoric acid is mostly precipitated as the acid is concentrated from about 30% P₂O₅ to 54%; 4) REE in the waste clay occur in two major forms, xenotime and calcium substitution in phosphate crystals.

THE RECOVERY OF RARE EARTH ELEMENTS FROM PHOSPHATE ROCK AND PHOSPHATE MINING WASTE PRODUCTS USING A NOVEL WATER-INSOLUBLE ADSORPTION POLYMER

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Key Words: Rare earth elements (REE), phosphate, phosphogypsum, poly (maleic anhydride-alt-1-octadecene) sodium salt, solvent extraction

The rare earth elements (REE) or rare earth metals are vital components in many modern electronics and are critical to the advances in several high technology fields, including green energy. While numerous procedures to extract and recover rare earth elements from phosphate waste products have been reported, none have seen widespread commercial acceptance due to various limitations, such as high cost and low efficiency, and the inability to economically extend the technology to large-scale operations. One way to achieve a commercially viable separation scheme is to employ a material that will economically and selectively bind various REEs in the presence of potential interfering ions, such as sodium, calcium, and silicon. In this study, the extraction and recovery of rare earth elements and phosphorus from phosphate rock and three phosphate fertilizer waste by-products, phosphogypsum, amine tailings, and waste clay, using 2.5% nitric acid and a novel water-insoluble adsorption polymer, poly (maleic anhydride-alt-1-octadecene) sodium salt, are examined. Overall extraction and recovery yields were between 80% for gadolinium and 8% for praseodymium from amine tailings, between 70% for terbium and 7% for praseodymium from phosphogypsum, between 56% for scandium and 15% for praseodymium from phosphate rock, and between 77% for samarium and 31% for praseodymium from waste clay. Average REE extraction and recovery yields were 50% to 60%. Poly (maleic anhydride-alt-1-octadecene) sodium salt effectively bound 100% of the rare earth elements extracted from the solids. Phosphorus was not chelated by the polymer and was isolated from the extract solution in yields of 16% to 34%. These results suggest that this process may be an efficient means of recovering rare earth elements and phosphorus from phosphate mining waste products.

A NOVEL PROCESS TO RECOVER SULFUR, LIME AND RARE EARTHS FROM GYPSUM

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Key Words: Gypsum, sulfur, lime, carbo-thermal reduction.

A novel process is described to recover elemental sulfur, high-grade calcium carbonate and a concentrate of rare earths. The process entails carbo-thermal reduction of gypsum to form calcium sulfide using ordinary bituminous coal as both energy and carbon source, followed by extraction of calcium sulfide as an aqueous solution of calcium bisulfide. Calcium bisulfide is then converted to hydrogen sulfide that can be converted to elemental sulfur using any one of a number of existing commercial processes and high-purity (>99%) calcium carbonate.

The overall mass and energy balances as well as indicative economic parameters shows that it should be economically viable to replace about 95% of sulfur purchased by a typical merchant-grade phosphoric acid producer with recycled sulfur and to produce high-quality calcium carbonate byproduct that should be suitable for use in many applications such as agricultural lime, lime for cement manufacturing and even in higher-value precipitated calcium carbonate filler markets. Rare-earths in the residue is concentrated to about seven times the concentration in waste phosphogypsum, making the viability to recover it much more lucrative.

Radioactive elements are concentrated as well and report to the same stream as the rare earth elements.

HYDROTHERMAL PRE-TREATMENT PROCESS OF PHOSPHOGYPSUM FOR ENHANCED RARE EARTHS RECOVERY

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Key Words: phosphogypsum, rare earths, recovery, structure, modification

Phosphogypsum (PG) is an industrial waste of phosphoric acid (H_3PO_4) production from phosphate ore by sulphuric acid (H_2SO_4) digestion, commonly known as the “wet acid” process. Phosphate rock often contains some valuable rare earth elements (REE) and it is well-established that from 70% to 85% of REE originally present in the phosphate rock end up in the PG, with an average REE content of ~0.4 wt. % [1-4]. PG is considered to be an attractive secondary resource of REE. However, processes available currently for REE recovery are often very expensive, sometimes impractical, and in other cases are characterised by highly variable and low overall REE recoveries due to the complexity of the REE association within the PG structure [5]. Mintek developed a novel hydrothermal process which modifies the microstructure of PG and release entrapped REE phases as shown in Figure 1 for subsequent leaching/recovery. Modification of the PG structure resulted in an increase of REE recovery from 5% (m/m) to 80% (m/m). This process was verified on various PG samples and carries a potential to unlock substantial REE value associated with the enormous size of PG stockpiles globally.

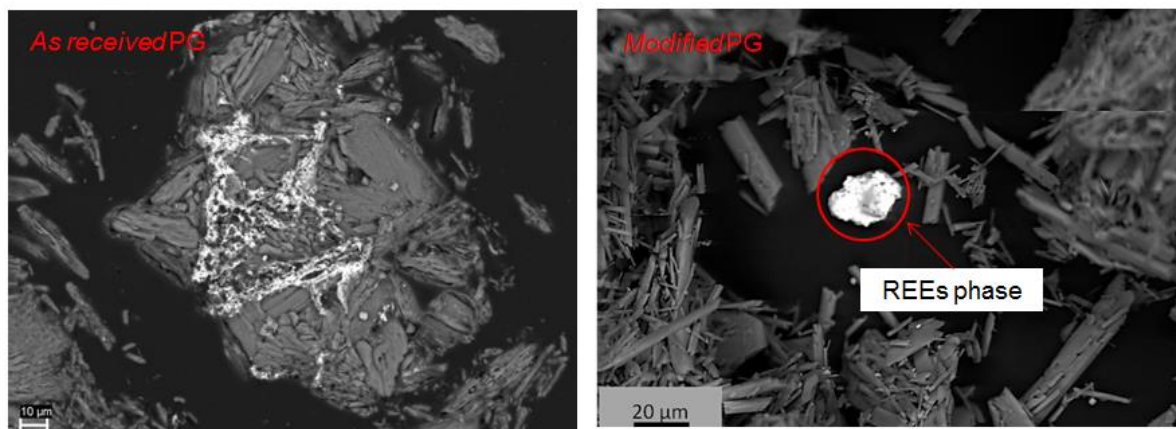


Figure 1 – Cross-sectional SEM image of phosphogypsum with REE phases (a) “as received” (b) modified

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SIMULTANEOUS RECOVERY OF RARE EARTHS AND URANIUM FROM WET PROCESS PHOSPHORIC ACID USING SOLVENT EXTRACTION WITH D2EHPA

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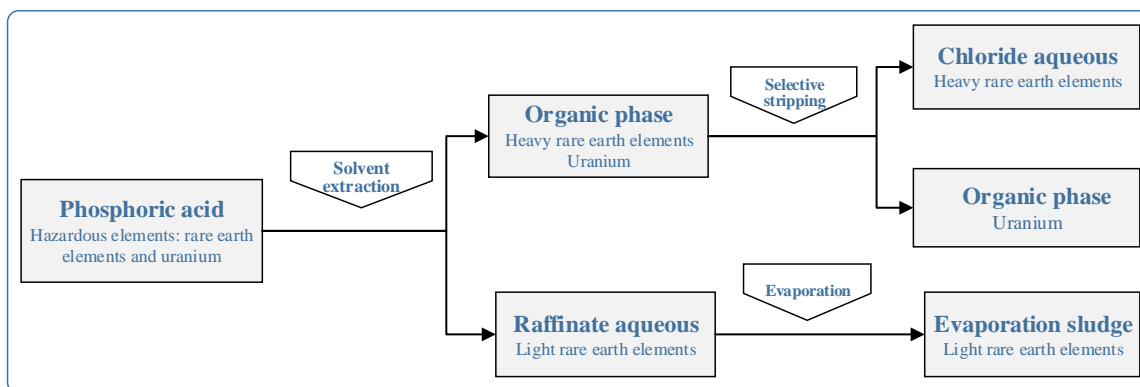
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Key Words: phosphoric acid, rare earth elements, uranium, solvent extraction, selective stripping, evaporation enrichment

In most cases, wet-process phosphoric acid (WPA) contains trace amount of rare earth elements and uranium, which ultimately enter and accumulate in soil and may cause environmental damages. In order to remove and recover these elements, studies about solvent extraction of heavy rare earth elements (HREEs) and uranium (U) with D2EHPA, selective stripping of HREEs from U, and evaporation precipitation of light rare earth elements (LREEs) were investigated. Extraction efficiency of U from WPA was greatly enhanced, but HREEs extraction were significantly retarded when using the solvent mixture of di- (2-ethylhexyl) phosphoric acid (D2EHPA) and tri-butyl-phosphate (TBP) or tri-octyl-phosphine oxide (TOPO) compared with D2EHPA alone. 89.4% and 94.2% accumulative two-stage extraction efficiencies of HREEs and U, respectively, were achieved with 1.0 M D2EHPA alone. And more than 98.1% HREEs were selectively stripped from loaded D2EHPA with 6.0 M HCl via three stages. In addition, LREEs were enriched to 1204.3 $\mu\text{g/g}$ in evaporation sludge during the concentration process of phosphoric acid from 30% to 50% P_2O_5 , and the filtration performance was greatly improved. Based on the studies, an alternative process was developed to recover and group LREEs, HREEs and U during the phosphoric acid production.



RECOVERY OF THORIUM FROM PHOSPHOGYPSUM BY ACIDIC LEACHING

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Key Words: Thorium, radioactivity, acid leaching, phosphogypsum.

Phosphogypsum (PG), a waste material readily available in large quantities, is generated in the wet process of phosphoric acid production. Due to the contaminants in the material and the radioactivity of PG, the use of this material has limitations. South African phosphogypsum (from Phalaborwa) contains high levels of thorium that could be recovered for industrial use. The study was conducted to determine the best acid for leaching thorium from phosphogypsum; to investigate the effect of solid to liquid ratios and of concentration on the efficiency of the process; and to study the effect of contact time on the thorium recovered from the leaching process. Thorium was recovered from phosphogypsum by leaching the samples with strong acids under specified controlled and manipulated variables. 98% sulphuric, 32% hydrochloric and 55% nitric acid were compared to identify the best leaching reagent. The solid to liquid ratios studied were 2%, 5% and 10%. The acid concentrations investigated were 2 M, 5 M and 10 M. Nitric acid proved to be the best leaching reagent among the investigated acids, recovering 0.07486 g of Th/ kg of PG. The lowest concentration of 2 M recovered more thorium (0.06117 g of Th/ kg of PG); while a higher solid loading yielded more thorium (10% loading recovered 0.04453 g of Th/ kg of PG).

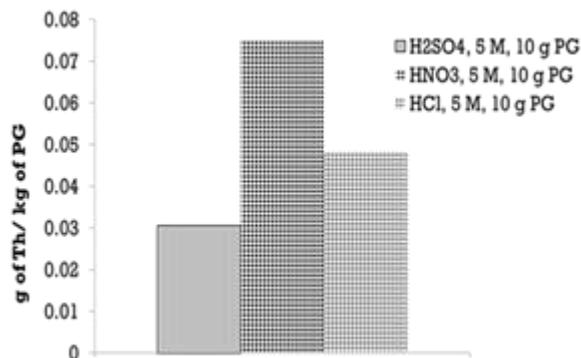


Figure 1 – Thorium leached with different acids

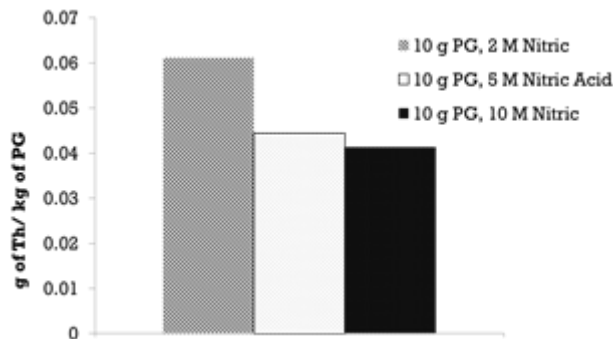


Figure 2 – Effect of nitric acid concentration on the recovery of Thorium

HISTORY AND FUTURE OF PHOSPHATE MINING AND BENEFICIATION IN SOUTH AFRICA

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The major igneous phosphate deposit in Phalaborwa was discovered in 1904. The establishment of the Phosphate Development Corporation (Foskor) in 1951 catalyzed the development and growth of phosphate mining and phosphate fertilizer manufacturing in South Africa as well as associated businesses. This keynote address covers four major topics: 1) chemical and mineralogical properties of major phosphate deposits in South Africa, 2) mining and beneficiation practices, 3) technical challenges and evolution of processing technologies, and 4) future trends.

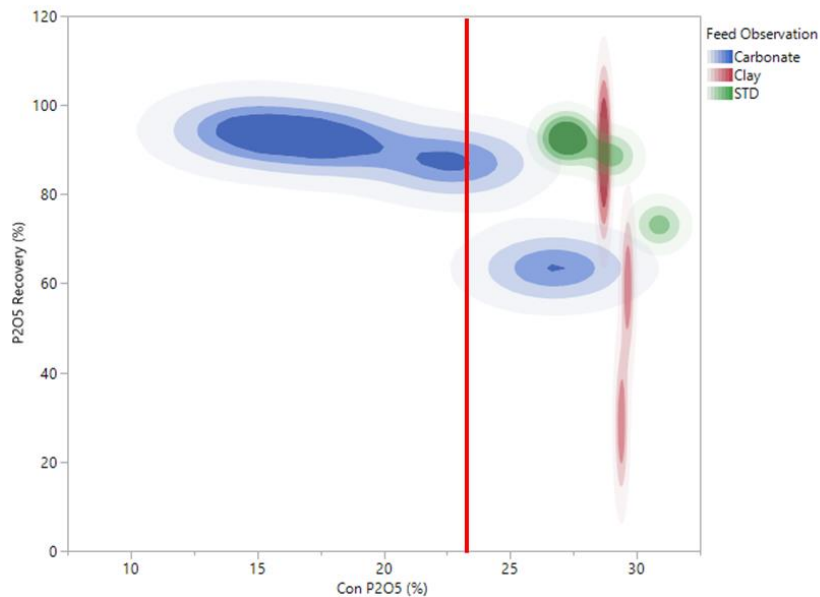
HOW TO TRULY OPTIMIZE PHOSPHATE FLOTATION, WHEN FEED GRADE IS EVER-CHANGING

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Key words: phosphate flotation, calcium carbonate, clay, beneficiation, collector

Optimizing a reagent package or beneficiation process on an average feed experienced at a float plant is one of the key objectives in successfully achieving an optimal, average grade/recovery for any mineral being processed. However, unlike the quality controlled processing chemistries, or engineered process, the mineralogy is minimally controlled by selective mining at best. The change in mineralogy can be a blessing or curse, and can even devastate a plant in matter of minutes. Therefore, applying techniques such as using the core samples from mine planning, or sophisticated analytical instrumentation can assist with planning when a detrimental change will occur. However, seasoned and informed flotation operators are necessary for ensuring the plant is prepared to handle these changes. This paper focuses on the results obtained from the application of a technical service program, which allowed for the investigation of various problematic feeds that plague a phosphate flotation plant. The purpose of this investigation was to investigate the cause, and develop a response plan for the flotation operators and engineers for when these difficult feeds reach the plant.

This paper will introduce three feed types, with all three requiring significantly different processing requirements in the plant. All three feeds were investigated to provide the most optimal mill conditions, which will be provided. The graph below shows how all three respond to the current controls in place, thus the need for change.



NEW REAGENT DEVELOPMENT FOR THE BENEFICIATION OF VARIOUS PHOSPHATE ORES

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Key Words: Phosphate Beneficiation, Froth Flotation, Cationic Collectors, Anionic Collectors

As the world population continues to increase, the demand for fertilizers is expected to remain strong. The growth in fertilizer demand is driven not only by population growth, but also by changes in diets, as more of the world's population is consuming more animal based protein. This trend drives the need for even more crops to feed cattle and higher fertilizer demand.

The phosphate that is used to make fertilizers continues to be sourced from mining, and new deposits are becoming increasingly more complex to beneficiate. Clariant Mining Solutions works closely with engineering houses and mining companies around the globe to develop novel reagents to solve the unique challenges of each deposit. In this presentation, some examples of the solutions developed by Clariant Mining Solutions will be highlighted.

Additionally, even existing phosphate operations face challenges to adapt to changing ore characteristics and to increase the sustainability of their operations. Some examples of the reagent development that Clariant Mining Solutions has conducted to improve existing operations will also be presented, including novel amines that reduce the water demand at the beneficiation plant.

MINE ARNAUD PROJECT: FLOTATION CIRCUIT ADJUSTMENT AND COLLECTOR REDUCTION

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Michel Lafontaine, Mine Arnaud

Key Words: apatite, column flotation, collector reduction

The Mine Arnaud open deposit is located in Sept-Iles in the province of Quebec, Canada. The project, which is currently under development, consists of igneous apatite ore grading around 5% P_2O_5 . The target metallurgical performances are set to a concentrate of P_2O_5 grade of at least 39% with an overall P_2O_5 recovery above 90%. The beneficiation of the ore is achieved by grinding to the liberation size, magnetic separation to remove the titaniferous magnetite and phosphate flotation. Most of Mine Arnaud's apatite beneficiation flowsheet development testwork was realized by COREM. The most recent work performed, at laboratory and pilot scale, has demonstrated that the target metallurgical performances could be attained using a flotation column only flowsheet composed of one rougher, one scavenger and one cleaner stages (Figure 1). The pilot scale testing was carried out at natural pH, and results show that the collector consumption could be reduced by approximately 50% when temperature was maintained above 20°C throughout the flotation circuit. Additional laboratory flotation tests also showed the potential of further reducing the collector consumption with an optimized frother addition. The presentation will focus on the flowsheet development and the pilot plant modifications in order to reach the client targeted concentrate while maintaining a low collector dosage.

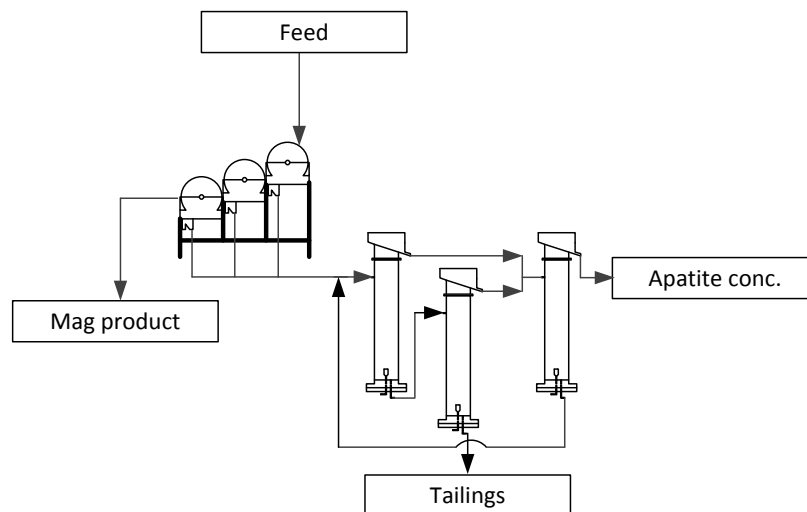


Figure 1. Mine Arnaud ore beneficiation flowsheet

CASE STUDY FOR PROCESSING PHOSPHATE ORES WORLDWIDE

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The characteristics of phosphate ores from different sources can vary significantly. The target specifications for marketable phosphate concentrates can also vary for each end user. The impurities associated with phosphate reserves include clay, silica quartz, silicates, carbonate minerals, iron oxide minerals, organics, etc. Among the technologies for processing phosphate ores, flotation is most commonly used to separate phosphate from those impurities. Examples of processing phosphate ores around the world with ArrMaz CustoFloat® (CF) and CustAmine® (CA) collectors with approaches such as reagent scheme and flowsheet will be presented.

EVALUATION OF THE EFFECT OF Ca^{2+} ION CONCENTRATION ON CALCITE FLOTATION OF SANTA QUITERIA'S ORE USING CARBONIC GAS

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Key Words: process water; ions; calcite, flotation; carbonic gas.

The most widely industrial flotation process for the treatment of phosphate ores containing carbonate as gangue minerals, especially in China, Saudi Arabia, Morocco, use strong acids, such as H_3PO_4 and H_2SO_4 as apatite depressants, and the carbonate minerals (especially calcite and dolomite) are selectively floated with the use of fatty acids as collectors at pH between 5.5 - 6.0. The flotation process currently considered for apatite concentration for the Santa Quitéria ore (Brazil) was developed in the middle of the 1980's at the Center for Development of Nuclear Technology and involves bulk flotation of apatite and calcite with anionic collector (fatty acid soap) at pH=10 followed by calcite flotation at pH=5.5, adjusted with H_3PO_4 . Calcite is collected in the froth phase and apatite concentrate is the sink product. Although this concept is efficient from the point of view of separation between apatite and calcite, the intensive use of strong inorganic acids (>15 kg/t) causes accumulation of ions in the process water, in particular Ca^{2+} and PO_4^{2-} , which leads to problems in the flotation stage and also environmental. Process water containing large amounts of calcium, magnesium, fluoride and phosphate (phosphorus) directly influences the performance of apatite flotation (recovery and grade). Guimarães and Peres (1999) evaluated the effect of ions (dosed in the conditioning stage) on the flotation of barite and apatite. In both cases, the recovery decreased significantly as the ion concentration increased. Based on these results, limited ion concentrations as 20 mg/L ions Ca^{2+} and 30 mg/L Mg^{2+} were proposed to ensure the quality of recirculating water without impairing the flotation process. Santos et al. (2010) presents results of the effect of the ions concentration Ca^{2+} , Mg^{2+} , F^- and PO_4^{2-} on apatite recovery. The recovery of apatite decreases expressively with the increase in the concentration of fluoride, calcium, magnesium and phosphate ions, reaching a maximum reduction of 85% in apatite recovery. According to Hanna and Somasundaran (1976), the collector reacts with the calcium present in the apatite and also the Ca^{2+} addition consumes the available collector for the mineral, resulting in a significantly reduction in apatite recovery. Aquino (1985) carried out studies with the Santa Quitéria ore evaluating the effects of calcium and magnesium ions concentration on the apatite flotation. The results indicated that concentrations of 49 mg/L Ca^{2+} and 30 mg/L Mg^{2+} , the recovery decreased from 94.2% to 53.3% for P_2O_5 and for CaCO_3 from 89.6% to 37.5%. CETEM has been studying a process for the separation of carbonate minerals and apatite that involves the use of carbonic gas injected into the bubble generation system of flotation machines instead of strong inorganic acids. The aim of this work is to evaluate the effect of Ca^{2+} ion concentration on the water just in the calcite flotation stage of Santa Quitéria's ore. For the tests carried out with addition of Ca^{2+} ions, the concentrations above 95 mg/L, a reduction in the selectivity of the process is observed, since a tendency in the increase of the apatite flotation is evidenced, noted by the increase of the content of P_2O_5 in the floated fraction. For tests carried out recirculating the process water, the accumulation of Ca^{2+} ions from 8 mg/L to 285 mg/L increases apatite content in the calcite concentrate around 30%. However, at concentrations above 285 mg/L of Ca^{2+} ions, the P_2O_5 content in the sink fraction and the P_2O_5 losses are not significantly affected. The results obtained in this study indicate that the process based on the application of CO_2 for the separation of calcite and apatite may be a technical alternative for the concentration ores with carbonated gangue that causes less impact in the process residual water, since there is no addition of strong acids as apatite depressant.

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INVESTIGATION OF THE MECHANISM OF APATITE/CARBONATES SEPARATION IN ACIDIC FLOTATION PROCESS USING *IN SITU* RAMAN SPECTROSCOPY

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Key Words: flotation carbonated hydroxyfluorapatite; calcite, phosphoric acid; Raman spectroscopy.

The separation of phosphate from carbonate gangue using flotation is extremely complex due to the similarities in surface properties. The acidic flotation process of carbonates at pH 4.5-5.0 followed by a direct phosphate flotation uses phosphoric acid as depressor of apatite during carbonate flotation. The mechanism of apatite depression is rather hypothetical because of lack of data on the surface speciation in such a complex mineral suspension. A real-time method for *in situ* control of surface compounds on the minerals (carbonated hydroxylapatite and calcite) using a submerged Raman analyzer probe is proposed in this work to collect the data on the nature and kinetics of phase formed. The carbonated hydroxyfluorapatite (C-HFap) and calcite reactivity in phosphoric acid at concentrations between 0 and 1 M) during 15 minutes was studied. The Raman spectra collected *in situ* and *ex situ* showed the peak at 1085 cm^{-1} of calcium carbonate at 10^{-3} and 10^{-1} M on the surface of hydroxyfluorapatite that disappears at high acid concentration. Kinetics studies performed at 4.6 (± 0.1) pH range during 15 minutes showed calcium carbonate formation after 2 minutes of conditioning and that disappears after three minutes (Figure 1). The infrared spectra of C-HFap in presence of phosphoric acid showed at 10^{-3} and 10^{-1} M new peak at 713 cm^{-1} that belongs to calcium carbonate vibrations which seems to be due to C-HFap surface conversion as observed on XRD patterns. In flotation of phosphates from carbonate minerals in acid media, implications of such conversion surface phenomena favours separation of these

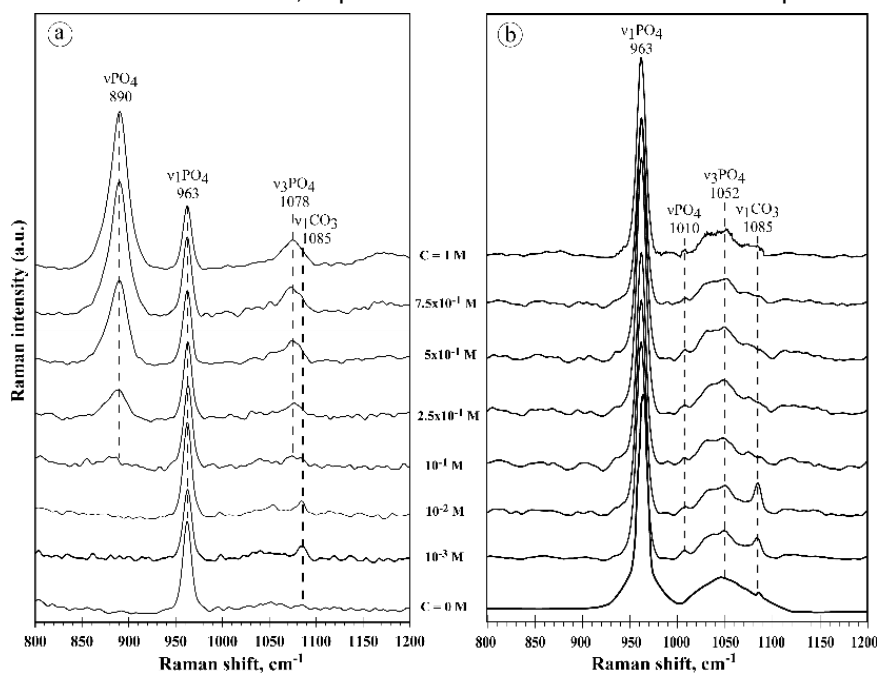


Figure 2 – Raman spectra of C-HFap after 15 min of contact with solutions of phosphoric acid of different concentrations measured (a) *in-situ* and (b) after filtration and drying (800 – 1200 cm^{-1} region).

minerals by preventing C-HFap dissolution and collectors adsorption on its surface. The Raman and infrared spectra show the characteristic carbonate and phosphate peaks on the calcite surface attributed to a new phosphate phase which intensity decreases and increases, respectively with increasing phosphoric acid concentration and reaction time. A similar trend was observed with increasing acid concentration in the XRD analysis of calcite which also revealed that this new phosphate phase is dicalcium phosphate dihydrate or brushite. Fine sheets of calcium phosphate particles were observed by SEM analysis covering the calcite particles at a high phosphoric acid concentration.

The stability of calcium phosphate compounds on the surface of calcite and apatite is dependent on the initial acid concentration and stable pH values. Preferential fatty acid collector adsorption on the carbonates is attributed to the differential kinetics of hydrophilic calcium phosphates formation on the mineral surfaces. The observed mechanisms were used to adjust the reagent regimes of the flotation at various pH values performed on a sedimentary ore samples.

FORMULATION OF A REAGENT USED IN A PROCESS OF WATER RECOVERY THE SLUDGE FROM BENEFICIATION PROCESS OF PHOSPHATE ORE USING MIXTURE DESIGN

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Keywords: Phosphate, sludge, Design of Experiment, formulation, Mixture Design, Screening Mixture Design, Polymers.

Sludge is generated by several industries. Sludge is a problem for both industrial and the environment. In order to treat this sludge and recover water many process was developed. These process use reagents to increase the sedimentation rate. Several reagent kind used. There are mineral and organic compounds. However, the organic ones, particularly polymers, are judged more efficient.

In this work, our goal was to prepare a new polymer-based reagent as a flocculant. This new reagent, which used in a process of water recovery the sludge from beneficiation process of phosphate ore, was prepared using mixture design. In the first, we studied the effect of eight reagents on sludge treatment by a screening mixture design. The screening mixture design gave us some information on the morphology of the polymers that allows to have the best flocculant. Afterward, thanks to the mixing design, we could find several formulas able to give new reagents by mixing the reagents that are selected by the screening mixture design.

In the end, the application of this new reagent that resulted to this study gives an interesting result.

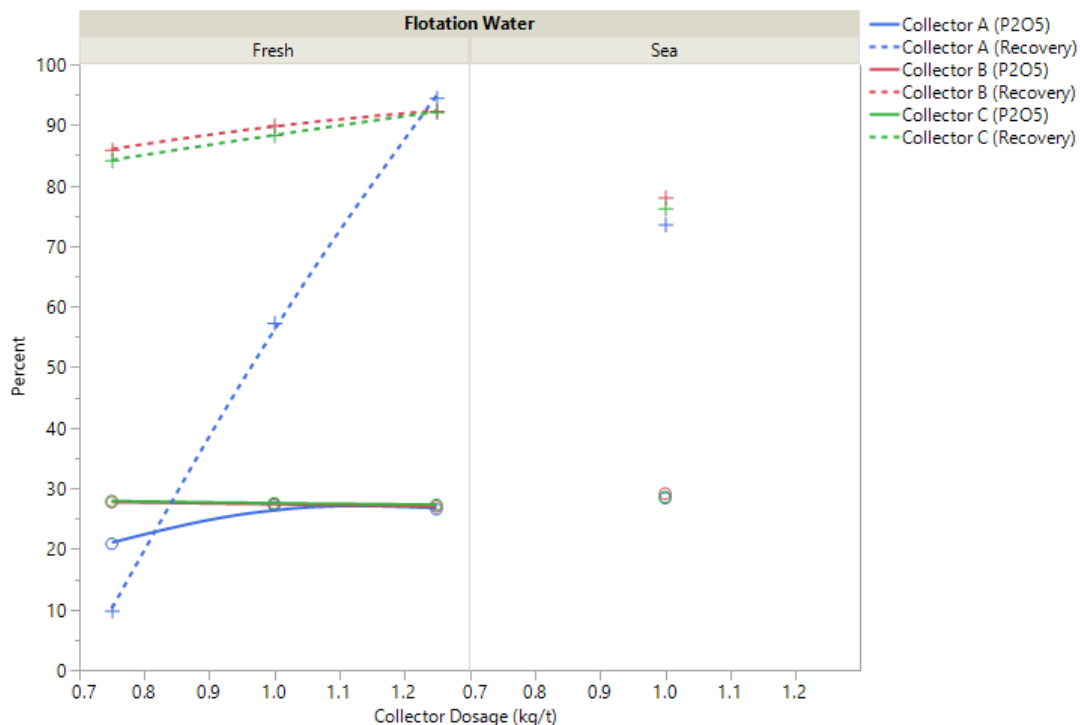
TAILORING COLLECTORS TO ALLOW FOR SEA WATER USAGE IN PHOSPHATE BENEFICIATION

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Key words: phosphate flotation, beneficiation, collector, sea water

Water supply is critical for ensuring the optimal operation of any beneficiation plant. As ore quality continues to decline, the demand on water for mineral processing will only increase. Traditionally, ground and surface water sources have been extensively utilized for mining. In some regions, these sources are proving to be insufficient to meet the increasing demands of this industry, as well as the municipal requirements. As a result, alternate water supplies must be considered. The purpose of this investigation was to develop a collector that allows for the flotation of phosphate in sea water, while still meeting the grade ($> 27.5\% \text{P}_2\text{O}_5$) and recovery ($> 80\%$) criteria.

The graph below shows a series of collectors that were tailor made for this mineral. The grade could be met, with significantly higher recoveries than was the criteria. However, this graph also shows the negative impact of the sea water on the flotation recoveries. Therefore, this paper will introduce the concept of tailor making collectors not only for the mineral, but also the water.



THE SURFACE CHEMISTRY AND MICROFLOTATION OF XENOTIME AND DEPRESSION OF GANGUE MINERALS

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Key Words: Flotation, xenotime, sodium silicate, ammonium lignosulfonate, octano hydroxamic acid..

A pre-concentrated xenotime sample and four selected gangue minerals, ilmenite, zircon, schorl and staurolite were used in this project. Using Octano-hydroxamic acid as a collector, the surface chemistry was investigated through surface area measurements, zeta potential tests and adsorption density determinations. The results of adsorption studies were then correlated with microflotation tests conducted at room temperature in a Partridge-Smith cell. In this paper, the surface chemistry and microflotation behaviors are then discussed based on both lab observations and a literature review.

Further, because of their selective depressing power, sodium silicate and lignosulfonate have been widely used as depressants in rare-earth mineral flotation to separate minerals from specific types of gangue minerals. In this project, the flotation of a xenotime pre-concentrate and pure samples of the gangue minerals ilmenite, zircon, schorl and staurolite using octano-hydroxamic acid and sodium oleate as collectors was conducted in a Partridge-Smith microflotation cell. The flotation of the mixed samples (weight ratio=1:1) of xenotime and each one of its gangue minerals was also investigated at room temperature and 80°C, using sodium silicate and ammonium lignosulfonate in the presence of octano-hydroxamic acid and sodium oleate respectively. The flotation results are described and compared with those observed by previous researchers. The effects of sodium silicate and ammonium lignosulfonate on weight recoveries and grade of xenotime are also discussed and compared.

HEAVY METAL CONTAMINATION AND HEALTH RISK ASSESSMENT IN WASTE MINE WATER DEWATERING USING PHOSPHATE BENEFICIATION PROCESSES IN JORDAN

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Keywords Phosphate beneficiation. Mine water. Health risk. Heavy metals. Multivariate statistical analysis

The main objective of study was to determine the degree of heavy metal contamination in Washing Mine Water of phosphate bed-A1 (WMW-A1) and Flotation Mine Water of phosphate bed-A3 (FMW-A3), and to assess the possible health risks associated with oral daily intake and dermal absorption skin of mine water effluents from phosphate mining process. Results showed that the average concentrations of Cd, Cr, Li, Mn, Mo, Ni, Pb, U, Tl, V, and Zn were below the required standard. The contamination index indicated that both mine waters showed slightly low contamination at mine sites, this indicates low raw phosphate inputs from phosphate mining activities. The mean concentrations of Mn and Cr were higher in mine water compared to stream water surface water, waste water, and industrial water. Multivariate statistical analyses including factor analysis and cluster analysis results revealed that natural input from phosphate raw materials was the main source of mine water contamination. The health risk assessment showed that hazard quotient (HQ) and Hazard index (HI) values were <1, indicating non-carcinogenic risk through daily intake and dermal exposure pathway in mine water. The daily intake (DDI) were V>Cr>Ni> Zn> U>Mn>Cd>Pb>Tl in WMW-A1, respectively, and Mn>Cd>V>Ni>Zn>Cr>U>Tl>Pb in FMW-A3, respectively. The Carcinogenic risk for Pb was lower than the acceptable risk of 1:10,000 for regulatory purposes. In long term use, the results showed that the mine water of the Phosphate beneficiation waste processing in Eshidiya mine is contaminated with heavy metals that might affect human health as well as the health of the ecosystem.

ALTERNATIVE DEPRESSOR FOR APATITE FLOTATION

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Key Words: Flotation. Depressors. Recovery

The reality of the phosphate mines in the world has been changing. The reserves content has reduced over the years. As a consequence of this reduction, contaminants such as iron, silica and magnesium contained in phosphate rock concentrate are affecting the stability of the chemical plants, which compromises the rate of phosphate utilization and the quality of the products produced. In order to obtain a concentrate of phosphatic rocks of market acceptable quality (35% P_2O_5), the flotation process is the most suitable method for processing this. The present study aims to evaluate the potential of replacing corn starch as a depressant in the flotation step. Corn starch is the most commonly used depressant because of its availability, or even lack of studies on the efficiency of other depressants. Microflotation tests were carried out in modified Hallimond tubes with the concentration of the Clariant Flotigam 5806 collector at 5.0 mg / L and the corn starch depressant used in industries, cassava starch and cassava flour at dosages of 0, 400, 800, 1,600, 3,200, 4,800 and 6,400 g / t at pH 8, the depressors were evaluated in overdoses with the objective of analyzing depression stagnation or continuity of the same according to increase of the dosage. The air flow used for the tests was $40 \text{ cm}^3\text{s}^{-1}$ and a pressure of 10 psi, which provides less hydraulic drag. 1 g of apatite mineral was used in the granulometry of - 100 + 150 #. The characterization of the starches and the sample of the apatite mineral were made by scanning electron microscopy SEM, EDS, fluorescence and X-ray diffraction, as well as their zeta potential. The results of the characterization showed that the sample of the mineral has high purity, showing absence of mixed particles. The results below (Figure 1) indicate that corn starch depressor presents a better recovery compared to cassava starch and cassava flour. Cassava starch requires a dosage two times higher than corn starch to achieve the same depression of the apatite mineral. Flotation tests showed can propitiate a better understanding of the dosages of starches and flour used.

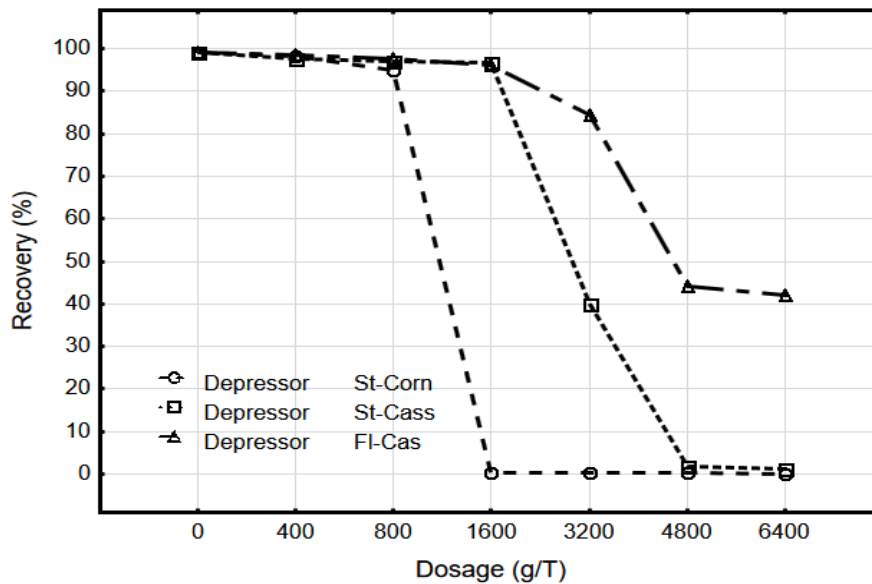


Figure 1 - Apatite recovery for different dosages of depressants

SEPARATION OF DOLOMITE FROM PHOSPHATE AT LOW COST AND REDUCED ENVIRONMENTAL IMPACT

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Key Words: Phosphate, packed flotation column, dolomite separation

Magnesium (MgO) in phosphate rock causes numerous problems in the phosphoric acid manufacturing process, including reducing filtration capacity, increasing sulfuric acid consumption, and making it difficult to meet product grade. MgO-containing minerals are fully dissolved and usually coexist in phosphoric acid by acid hydrolysis reaction to form $Mg(H_2PO_4)_2$. This makes it extremely difficult to separate the dissolved Mg from phosphoric acid. Therefore, it makes both economic and technological sense to remove dolomite from phosphate prior to acidulation. Except for limited use of heavy media gravity separation, flotation is the main technology currently practiced for separation of dolomite from phosphate. Because fine grinding is required to liberate dolomite for flotation separation, use of mechanical flotation cells is very expensive due to energy cost, long flotation time and high reagent use.

Recent development efforts have resulted in industry acceptance of an innovative flotation device, the packed flotation column (PFC), thus realizing its six (6) main advantages:

1. low energy use
2. long separation zone
3. small footprint
4. reduced water use
5. high throughput
6. effective for very fine particles

An industrial PFC, 12 meters tall and 3 meters in diameter, was installed at a phosphate mine in Hubei, China in early 2017 for separating dolomite from phosphate by flotation. Prior to switching the plant from mechanical flotation machines to PFC, long-term parallel testing was conducted, and the average results are shown in Table 1 for comparison.

Table 1. Parallel Plant Testing Comparison of PFC with Mechanical Cells.

Item	Mechanical machine	PFC
Capacity, dry tons/day	2000	2000
Machine volume, cubic meters	264	51
Power consumption, kilowatts	650	88
Fresh water use, cubic meter/hour	20-30	1-2
Flotation time, minutes	58	15
Operation mode	Manual	Computer control
Product grade, %P ₂ O ₅	30-31	30.7
Product grade, %MgO	0.60	0.50
%P ₂ O ₅ Recovery	~80	~80

USING TWO ALTERNATIVE VEGETABLE OIL'S AS COLLECTOR IN APATITE FROTH FLOTATION

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Key Words: Froth Flotation. Collectors. Apatite.

The Brazilian phosphate deposits are of igneous origin exhibiting a complex mineralogy where fluorapatite is the predominant mineralogical variety from the apatite group, this deposits still contain carbonateapatite and hydroxyapatite associated with secondary phosphates, iron oxides (magnetite, goethite and limonite), micas and minerals bearing niobium, titanium and barium. The flotation process as a separation and enrichment of the acceptable levels by the P_2O_5 market (35%) is the most efficient. Brazilian mineral industries use synthetic collectors from international companies. Many of these oils were initially produced to sort out the demand for biodiesel production, with their market being diminished and farmers having their request reduced, impacting the income of many families, besides what using natural oils is more ecofriendly and sustainable. In the present study were used collectors obtained by the cold saponification of vegetable oil of macaúba (*Acrocomia aculeata* (Jacq.) Lodd. ex Mart., purchased from a regional producer), pinhão-manso (*Jatropha curcas* L., donated by EMBRAPA Agroenergia), or the mixture of both in the proportion of 50% (m/m). The saponification process was carried out with the addition of 5 g of each oil or their mixture in 20 g of distilled water and stirred for 5 min. Subsequently, 10 mL water solution of NaOH (10% w/v) was added while stirring was continued for 5 minutes. After this period, the solution was doused with water until 100 g of total mass was reached and homogenized for another 10 min. Microflotation tests were performed on modified Hallimond tubes with a collector concentration of 2.5 mg.L^{-1} at pH 8, and 1 g of apatite less than 100#. The conditioning time was 7 min and 1 min for flotation. An application rate for tests of $40 \text{ cm}^3\text{s}^{-1}$ and a pressure of 10 psi. The results below (figure 1) point to the best recovery of the macaúba oil (1) reaching 90% recovery of the apatite, while the pinhão-manso (2) obtained only about 15%. The influence of the mixture of oils can be observed, reducing the recovery of the mixture of oils (3) to 10% when compared to macauba (1). The means are different by the Tukey test at 95% confidence. As each vegetable oil has characteristic lipid profiles, this differentiated composition seems to directly influence the ability to collect the mineral. It is concluded that the oils present employment potential as collectors, requiring more tests to optimize the mixture of them.

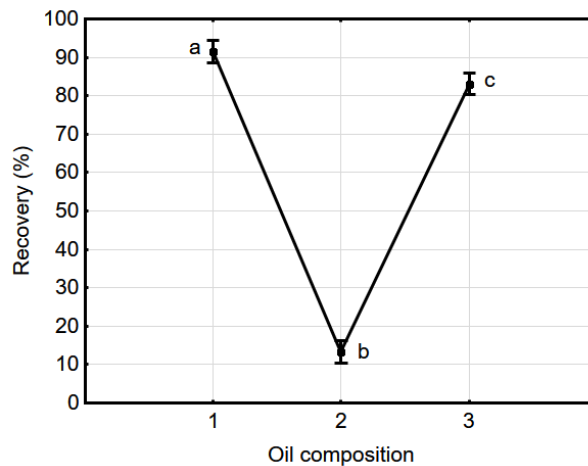


Figure 1– Recovery of apatite for oils composition: (1) 100% macaúba; (2) - 100% pinhão-manso and (3) 50% of each oil. Different letters are statistically different by the Tukey test ($p < 0.05$).

LATEST TREND FOR FLUORINE IN THE PHOSPHORIC INDUSTRY: ABSORPTION EFFICIENCY IMPROVEMENT, CONVERSION INTO RAW MATERIAL

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Almost all the phosphate apatite used in the world to produce phosphoric acid through the wet process contains fluorine. When not properly controlled, this element creates process, health and environmental issues that can have dramatic impact. In recent years several technologies have been established or improved to tackle these challenges.

F absorption has been improved by simple changes that ensure a high efficiency while minimizing the CAPEX. F usage has also been largely investigated to produce valuable products as AlF_3 or HF but also easy to transport raw material as CaF_2 . This paper will describe some recent developments related to these topics.

CAN YOUR P₂O₅ BE COMMERCIALY EXPLOITED?

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Key Words: Phosphate, Beneficiation, Phosphoric Acid, Fertilizers

World reserves of high-grade, high-quality phosphate rock are steadily decreasing. As a result, lower quality rock is being increasingly evaluated to determine its potential for commercial exploitation. It is well known that the chemical and mineralogical composition of phosphate rock varies widely from deposit to deposit, and therefore, the conditions for processing different phosphate rock types also varies.

Unfortunately, most early stage phosphate feasibility studies tend to focus on the P₂O₅ content and neglect the other chemical constituents or impurities in the rock. It is the remainder of the chemical constituents that often define the quality of the P₂O₅ concentrate and its potential for conversion to phosphate fertilizers. Typically, when evaluating phosphate ore and subsequent concentrate as a raw material, the first step is to analyze the composition (P₂O₅ and impurities) to gain a preliminary understanding of how the rock may behave in downstream chemical processing or as a direct application fertilizer. In addition to %P₂O₅, the following chemical constituents, at a minimum, should be evaluated when assessing the suitability of any phosphate rock:

- Calcium (%CaO)
- Magnesium (%MgO)
- Iron (%Fe₂O₃)
- Aluminum (%Al₂O₃)
- Silica (%SiO₂)
- Chlorine (%Cl)
- Fluorine (%F)
- Carbon Dioxide (CO₂) and Organics
- Sulfides
- Sodium (Na₂O) and Potassium (K₂O)
- Heavy metals, rare earths, and other elements.

Once the above chemical analyses of a representative phosphate rock product are known, it is often possible to predict the suitability of the rock as feed for conventional wet process phosphoric acid production. However, given the vast range in which these chemical constituents can be present, acidulation pilot plant testing must be carried out during the feasibility study project phase in order to properly assess the deportment of these constituents into the acid and/or gypsum.

This paper is presented to give a general understanding of the conventional wet acid process for conversion (acidulation) of phosphate rock as it relates to the presence and concentration of the major chemical constituents usually associated with phosphate rocks. Five case studies are presented.

HOW PHOSPHATE ROCK QUALITY IMPACTS THE PHOSPHORIC ACID PLANT AND GRANULATION PLANT OPERATIONS

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Key Words: Impurities, Phosphoric Acid, Beneficiation, Granulation, Phosphate Rock

The phosphate rock quality from region-to-region can vary significantly. Phosphate rock from Morocco the United States and Peru are significantly different with a wide range of impurity levels as shown in figure 1.

	USA Central Florida	Morocco	Peru
% P ₂ O ₅	31.20	31.50	30.05
% CaO	46.60	51.20	47.10
% Fe ₂ O ₃	1.50	0.24	0.70
% Al ₂ O ₃	1.20	0.35	0.63
% MgO	0.60	0.47	0.69
% F	3.70	3.86	3.02

Figure 1 – Typical Phosphate Rock Analyses

The beneficiation process is typically used to remove silica, clays, dolomite and calcite. Other impurities such as iron, aluminum, magnesium, and chlorides are not removed during the beneficiation process. The impurities that are not removed during the beneficiation process remain in the phosphate rock and impact the operation of the phosphoric acid and granulation plants. Additional parameters such, P₂O₅ and calcium (CaO) also have a significant impact on the operation of the phosphoric acid and granulation plants. The impact of variations in phosphate rock quality and impurities on plant operations will be reviewed. The impact of varying phosphate rock quality including P₂O₅, CaO, chlorides and aluminum content will be discussed regarding their effect on the phosphoric acid plant. The impact of iron, aluminum, magnesium, fluorides and P₂O₅ content will be discussed regarding their effect on the granulation plants.

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RECOVERY OF RARE EARTHS AND P FROM A PHOSPHATE FLOTATION TAILS

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Key Words: Rare earths, phosphate, gravity separation, flotation, leaching

Phosphate ore is one of the most significant secondary resources for rare earth elements (REEs), and Florida phosphate is of particular interest due to its much higher ratio of heavy REEs, especially yttrium. Analyses of numerous samples from different phosphate processing streams in Florida showed that heavy REEs account for about 50% of the total REEs in Florida phosphate with yttrium accounting for approximately 30%. It was estimated that the amount of yttrium from US phosphate processing could meet the world demand for this critical element.

This research aimed at recovering both REEs and phosphorus from a phosphate flotation tails. This tails is composed of mainly silica and phosphate with a small fraction of heavy minerals including monazite and xenotime. Since the specific gravity of silica is 2.6 versus 3.2 for phosphate and 4.5-5 for rare earth minerals, pre-concentration of the minerals of interest by gravity separation was investigated first. A pilot scale shaking table was used in this study. One pass gravity concentration achieved a shaking table concentrate analyzing over 600 ppm REEs at about 40% recovery. Flotation of the shaking table concentrate resulted in a product containing 1809 ppm REEs and 26.37% P_2O_5 with corresponding recoveries of 73% and 82%. Sulfuric acid leaching at 75°C could recover over 60% REE and nearly all the P_2O_5 . Higher REEs (>90%) recovery was achieved by conducting leaching at higher temperature with concentrated sulfuric acid. The phosphoric acid from leaching of the flotation concentrate is of superior quality compared with that generated from regular phosphate rock.

REVISITING THE MERSEBURG PROCESS: ECONOMIC OPPORTUNITY AND ENVIRONMENTAL BENEFIT?

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Key Words: Phosphogypsum, Ammonium sulfate, Calcium carbonate, Ocean acidification

The process for manufacture of ammonium sulfate from gypsum, known as the Merseburg Process, named for the town in Germany where it was developed, has been commercially produced in plants in Austria, India, Pakistan and the United Kingdom in the past. It is based on reacting ammonia and carbon dioxide to produce ammonium carbonate, which is then reacted with gypsum to produce ammonium sulfate and calcium carbonate. Ammonium sulfate is mainly used as direct fertilizer or in producing compound fertilizers. The global ammonium sulfate market is projected to grow 4% annually through 2025, from \$4.6 billion to \$6.8 billion. As global efforts to utilize more of the phosphogypsum produced in the production of phosphate fertilizers grow, the prospect of re-examining the economic viability would be timely. In addition to the potential for economic gain, the ability to capture and store carbon dioxide that is inherent in the process could result in a concomitant environmental benefit. Another potential environmental benefit can occur in locations where offshore disposal is or can be practiced. Disposal of the calcium carbonate produced, rather than phosphogypsum, could have the potential to offset the growing problem of ocean acidification and its resulting impacts on marine organisms.

RECOVERY OF RARE EARTH ELEMENTS FROM PHOSPHATE ROCK BY HYDROMETALLURGICAL PROCESSES-A CRITICAL REVIEW

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Key Words: Rare earth elements; Phosphate rock; Phosphoric acid; Phosphogypsum; Recovery technologies.

Generally, phosphate rock contains about 0.05wt% rare earth elements (REEs) on average. And the world commercial phosphate rock production is estimated to be 250 million tons per year, which makes phosphate rock a potential new REEs resource. However, low content of REEs in phosphate rock leads to the technical challenges and cost overages that hindered the commercial recovery of REEs. In this paper, an overview of achievements aiming at solving the challenges is given. Based on the decomposition processes of phosphate rock by H_2SO_4 , HNO_3 , HCl , H_3PO_4 , various REEs recovery processes via crystallization, precipitation, solvent extraction and ion exchange methods are systematically reviewed. In H_2SO_4 processes, REEs are recovered based on the removal of impurities from phosphoric acid and phosphogypsum since the main challenge is the diluted content of REEs in these products. In the case of HCl , HNO_3 and H_3PO_4 processes, REEs entirely transfers into leaching solution and the recovery research are mainly focused on REEs extraction from leaching solutions. For REE extraction from high phosphorus content leaching solutions, crystallization, precipitation, and ion exchange methods are currently inconsiderable due to the high energy consumption, impurity involvement and low efficiency, respectively. Solvent extraction seems to be the potential promising method in terms of its good overall performance. Finally, recommendations to promote the development of REEs recovery technologies from phosphate rock are provided.

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PHYSICAL SEPARATION OF HIGH GRADE AND LOW GRADE AFRICAN PHOSPHATES

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Key Words: physical separation, African phosphate, low grade

Africa is a source of many phosphate deposits of various resource grades and a significant global contributor of phosphate for fertilizer or phosphoric acid applications. Many of these deposits in Africa exist as sedimentary and alluvial deposits. The main phosphate bearing mineral is usually carbonate fluorapatite with quartz occurring as the main gangue mineral. It is common for phosphate deposits to have clay constituents (varying amounts in each deposit) and Mintek has found through various studies that African phosphates can easily be upgraded by removal of barren fines/clays after some initial upfront wet scrubbing. The scrubbed material is then screened wet to remove the barren fines, which normally occur in the <600µm fractions. Coarser fractions, usually >13mm are also scalped off as they also tend to contain barren coarse waste consisting mainly of quartz, calcite and dolomite. In some cases, energy input is required to remove the contaminants from the phosphate mineral surface, hence attritioning would be carried out in these instances. Much of the testwork conducted at Mintek was to determine if the resource can be upgraded to >30% P₂O₅ target specification.

Mintek has undertaken many projects dealing with the physical beneficiation of African phosphates over the past years and had recently conducted testwork on high grade and low grade Angolan phosphate with the results appearing in Table 1 below. The sample had undergone scrubbing followed by screening at 0.5mm. Additionally, the +12mm material was scalped.

Table 1: Performance of a high grade and low grade Angolan phosphate

	High Grade									Low Grade								
	Grade					Recovery				Grade					Recovery			
	Mass	P ₂ O ₅	SiO ₂	Al ₂ O ₃ +Fe ₂ O ₃ MgO	Cd	P ₂ O ₅	SiO ₂	Al ₂ O ₃ +Fe ₂ O ₃ MgO	Cd	Mass	P ₂ O ₅	SiO ₂	Al ₂ O ₃ +Fe ₂ O ₃ MgO	Cd	P ₂ O ₅	SiO ₂	Al ₂ O ₃ +Fe ₂ O ₃ MgO	Cd
[%]	[%]	[%]	[%]	[ppm]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[ppm]	[%]	[%]	[%]	[%]	
+12mm	2.0	10.5	67.5	3.6	69.7	1.1	3.5	1.1	1.4	0.5	8.7	65.6	26.4	56.1	1.1	0.6	0.8	1.2
-12.5+0.5mm	52.8	30.1	10.1	4.4	153.5	85.5	13.9	36.8	80.8	9.04	13.3	53.6	6.7	78.2	29.3	8.2	3.5	27.7
-0.5mm	45.2	5.5	70.3	8.6	39.6	13.4	82.6	62.1	17.8	90.43	3.15	59.8	18.2	20.1	69.5	91.2	95.7	71.2
Total Calc.	100.0	18.6	38.5	6.3	100.3	100.0	100.0	100.0	100.0	100.0	4.1	59.3	17.2	25.5	100.0	100.0	100.0	100.0

The high grade and low grade samples reported head grades of ~19% P₂O₅ and ~4% P₂O₅ respectively. The high grade sample was upgraded to product specification of 30% P₂O₅ at a yield and recovery of 53% and 86% respectively; whereas the low grade sample only upgraded to 13%P₂O₅ at a yield and recovery of 9% and 29% respectively. More stages of processing is recommended for the low grade sample to achieve the target specification. The marked difference between the samples is the significantly high clay content in the low grade sample as compared to the high grade sample, as well as the higher silica content. The high clay content also caused processing difficulties. Deleterious heavy elements such as cadmium (Cd) need to be minimal in the upgraded phosphate rock in order to be used as fertilizer that grow crops for human consumption. The Cd content in the high grade sample was much higher (153ppm) than that of the low grade sample (78ppm). Calcination tests may be conducted to attempt to reduce the Cd content keeping in mind the detrimental effect of calcination on the phosphate fertilizer applications. African phosphate ores generally concentrate to the coarser size fractions, but in some cases the actual phosphate minerals are not 'clean phosphate' and have inclusions containing Fe, Al and Mg which are unfavorable as it renders phosphoric acid production uneconomical. In cases such as these, magnetic separation would be employed to attempt to remove the magnetic minerals containing Fe, Al and Mg. A specific case investigated on a Mali phosphate sample with a high Fe and Al content did not improve its situation via magnetic separation as mineralogical analysis indicated that the elements were present as tiny inclusions (<10µm in diameter) in the phosphate minerals which then indicated that further liberation of these minerals would not be economically viable. Methods other than physical beneficiation (for example selective leaching) is recommended for these cases. This paper will however primarily focus on testwork on Angolan ore bodies in particular the challenges with the high and low grade material treated.

EVALUATION OF AN AGUIA RESOURCES – TRES ESTRADAS BRAZILIAN PHOSPHATE ORE USING COLUMN FLOTATION

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Key Words: Flotation, Column Flotation, Mineral Processing.

To satisfy increasing domestic fertilizer demand, development of low-grade, finely liberated, phosphate deposits within South America has increased. In response to this demand, and decreasing phosphate prices, efforts are being made to maximize global phosphate recovery and grade within existing and prospective phosphate flotation circuits. Such improvements are beneficial to all phosphate operations, but are most advantageous when treating a lower grade ore where processing costs are highest, operating margins are lowest, and a near zero-waste approach is necessary. Specifically, column flotation technology has proven to greatly increase phosphate flotation selectivity and recovery during treatment of fine, minus 212-micron, and ultra-fine, minus 53-micron, igneous phosphate ores from Aguiá Resources' Três Estradas project. Using column flotation, a deep froth is maintained and washed by means of a counter-current flow of clean wash water to minimize entrainment of gangue (i.e. calcite, silica, mica, etc.). Additionally, cavitation tube spargers are utilized to produce a fine bubble dispersion to maximize total available bubble surface area and improve fine phosphate particle recovery. In reverse flotation applications, where fine calcite or silica are rendered hydrophobic, such features also improve gangue rejection and phosphate recovery. This paper details the improved flotation response of both fresh carbonatite and saprolite of carbonatite phosphate ores from the Três Estradas deposit using CavTube column flotation.

INFLUENCE OF THE IMPELLER SPEED ON PHOSPHATE ROCK FLOTATION

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Key Words: Phosphate rock; cell flotation; impeller speed.

Flotation represents, at present, the technology capable of efficiently concentrate the minerals of interest, separating them from other gangue minerals. The flotation process is based on interactions between minerals (solid phase), water (liquid phase), and air (gas phase). However, it depends on physical forces driving the system. If mineral particles do not have enough momentum during the collision with the air bubble no attachment between them occurs, and consequently no flotation. Impeller rotation speed is undoubtedly a factor that changes the hydrodynamic conditions of the flotation system playing a vital role on it. It supplies the flotation system with air by suction, produces bubbles on the base of the cell, and stirs the pulp. By keeping the mineral particles in movement, the impeller rotation avoid fast sedimentation of the particles and gives the energy required for the particles attach themselves to the bubble. Although the impeller rotation has very high importance to the flotation, it also has a negative action on the system since it is one of the greater producer of turbulence in the flotation cell. The better understand the influence of the impeller rotation speed in the phosphate rock flotation experiments using a Denver cell with 3.0L cell were carried out using phosphate rock from Copebras/CMOC mine, a magmatic deposit situated in Ouidor/GO/Brazil. Cornstarch (600 g/t) supplied by Cargill was used as depressant and Lioflot 502-A (500 g/t), a mix of fatty acids, supplied by Miracema-Nuodex was used as collector. The conditioning time was three minutes for the depressant and two minutes for the collector and flotation time was five minutes. The pH was kept at 9 with hydrochloric acid and sodium hydroxide, both at 1%. Five different impeller speeds were tested (1000, 1150, 1300, 1450, and 1600 RPM). All tests were performed in triplicate at room temperature and using tap water. Samples of the feed, concentrate and tailings were analyzed at Copebras S/A laboratory using an X-ray Fluorescence spectrometer AXIOX MAX series DY 5001 from PANalytical. Results show that an increasing in the impeller speed until 1450 RPM produces a reduction in the P_2O_5 content in the concentrate. Above this value the P_2O_5 content raises with the impeller speed. The higher grade obtained was around 34% of P_2O_5 at 1600 RPM.

SUCCESSFUL APPLICATIONS OF VERTICAL-ROLLER-MILLS IN PHOSPHATE PROCESSING

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Key Words: Comminution, compressive grinding, VRM technology, energy efficiency, operation experience.

To keep up with increasingly challenging tasks in the processing of ores and industrial minerals, the design of the process as well as the choice of the equipment to be installed will become more complex. This accounts for the process step of grinding in particular, as it has significant impact onto the performance of the downstream sorting process and represents a major share in the overall operating expenditures of a mining operation. Especially with increased requirements to the grinding product, alternative technologies such as compressive comminution in Vertical-Roller-Mills (VRM) can offer efficient solutions and replace conventional grinding technology.



Figure 1 – LM 50.4 with elongated classifier at Phalaborwa, Foskor

VRM technology has several intrinsic benefits. The system for combined crushing, grinding and drying offers a very high reduction ratio at low energy consumption and low specific wear rates. The internal circuit of compressive grinding and immediate classifying leads to improved mineral liberation and a narrow particle size distribution. Online adjustable process parameters allow the system to respond quickly to fluctuations in feed mass flow and mineralogical composition. With several thousands of VRM's installed in various industries, including the grinding of phosphate rock and phosphate concentrates, VRM technology proved to be more than suitable for the different grinding tasks in phosphate processing.

Next to various installations for phosphate concentrate grinding, especially hard rock operations like Foskor Ltd. (Phalaborwa, RSA) and EuroChem (Zhanatas, Kazakhstan) clearly benefit from VRM technology. Since 19 years Foskor operates a Loesche VRM for the comminution of pyroxenite hard rock (BW_i ~ 25.3 kWh/t). Due to a close cooperation between Foskor and Loesche, the mill is operated with good performance data and positive impact on the production. In January 2016 another Loesche VRM for the processing of pyroxenite phosphate rock (BW_i = 18.5 kWh/t) went into operation at EuroChem in the south of Kazakhstan. Despite strong fluctuations in the feed material, the mill is operating smoothly at very positive performance data.

The paper summarizes the key features of Loesche VRM technology and its benefits for the comminution of phosphate materials. The status in successful hard rock applications of the VRM technology, such as Foskor (Phalaborwa) and EuroChem (Zhanatas), is presented. Selected performance data is reported and compared to results of conventional ball mill circuits.

A COMPARITIVE STUDY OF DIFFERENT COLUMNS SIZES FOR ULTRAFINE APATITE FLOTATION

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Key Words: apatite; flotation; slimes; column flotation.

The Brazilian phosphate industry has a long-term tradition in beneficiation of weathering igneous phosphate ores. The typical flowsheet applied involves grinding; classification in coarse and fines fractions; magnetic separation (low and high intensity); desliming to remove particles < 10 µm; barite flotation and apatite flotation. Desliming operation to discharge the ultrafine particles less than 20 µm prior to concentration by flotation is a common practice on the phosphate ores beneficiations plants, including those that treats sedimentary and igneous ores. Guimarães and Peres (2002, 2003) resume the Brazilian industrial experience in beneficiation of the material with particle size < 44 µm. The first industrial application was in Araxá concentrator in the beginning of 80's in a circuit consisting of desliming in 40 mm hydrocyclones and apatite flotation in column machines. Corn starch and fatty acids being utilized as reagents. The concept of this process was expanded to other Brazilian plants located in Catalão (Goias state), Tapira (Minas Gerais state) and Cajati (São Paulo state) are still in the 90's. It is estimated that the apatite concentrate from slimes represents 11% to 13% of the overall production. Matiolo et al (2017) showed results of a flowsheet developed to apatite concentration from a slime sample provided by the industrial phosphate ore plant of the Copebras (China Molybdenum – CMOC International), located at Catalão (Goias state, Brazil). The flowsheet involves desliming in hydrocyclones (40 mm) in two stages, followed by apatite flotation in columns. The authors compare the flotation results obtained in a rougher flotation in a 3" diameter column flotation (2.0 m in height) in comparison with the results obtained in a circuit with rougher/cleaner configuration applying a 4" diameter column flotation (6.0 m in height) to rougher and a 2" diameter column flotation (6.0 in height) to cleaner. This work shows flotation results considering a circuit with rougher/cleaner configuration with different sizes column flotation, as follow: Circuit 1 (rougher – 4" diameter column flotation; cleaner – 2" diameter column flotation), and circuit 2 (rougher – 6" diameter column flotation; cleaner 4" column flotation). The solids feed rate in each circuit was 35 kg/h and 46 kg/h dry basis respectively. The rougher flotation results show that 6" diameter column flotation achieved higher apatite recovery when compare with the results obtained in the 4" diameter column flotation, for similar P₂O₅ grades. By the other hand, the results obtained in cleaner flotation are very similar in both columns (4" and 2" diameter column flotation). The best results obtained in the 6" diameter column flotation can be explained by the fact that in this column, the bubble generation system is a cavitation tube and the system applied in the 4" diameter column flotation is a porous tube. The bubble size distribution obtained on the cavitation tube is in general smaller than the obtained on the porous tubes. Fine bubbles improved the collision efficiency between bubbles and particles which leads to higher recoveries, in special for ultrafine particles flotation.

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THE FUNDAMENTALS, UNIQUE FEATURES AND PERFORMANCE OF A NEWLY IMPROVED PACKED COLUMN JIG FOR PHOSPHATE PROCESSING

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Key Words: Phosphate, gravity separation, dolomite.

The packed column jig (PCJ) is an innovative gravity separation device. PCJ was patented 20 years ago, but its commercial application only began recently after three significant improvements were implemented: 1) new design of packing and feeding system to overcome the plugging problem, 2) computer control to optimize operating parameters, and 3) semi-industrial testing to obtain the optimal design parameters. This paper discusses the fundamentals behind the superior performance of PCJ, demonstrates its unique features that result in energy and operating costs savings, and presents two industrial applications, one for separating silica from phosphate and another for separating dolomite from phosphate. The results shown in the table below are perhaps the best gravity separation results on phosphate.

Table 1. Semi-industrial Scale Testing Results Using PCJ for Silica Removal from Phosphate.

Size range, mesh	Feed grade, % P ₂ O ₅	Concentrate grade, % P ₂ O ₅	% P ₂ O ₅ recovery
-20+40	23.10	28.88	88.46
-40+100	22.18	26.73	87.72
-100+200	21.99	28.67	84.94

MILLING OF THE PHOSPHATE ROCK FLOTATION CIRCUIT CIRCULATING LOAD AIMING PRODUCTION INCREASE AND IRON CONTENT REDUCTION IN THE FINAL CONCENTRATE

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Key Words: Phosphate rock; froth flotation; circulating load; milling.

Phosphorus has its main application in the production of phosphoric acid, being the apatite series its main ore. The Brazilian main phosphorus reserves are igneous, as is the case of Catalão alkaline dome, where Copebras/CMOC International has operations. The content of P_2O_5 in the ROM has been reduced over the years and, as a consequence, the content of the contaminants in the concentrate, produced by flotation, which enhances the difficulty in the phosphoric acid production. Only traces of arsenic, iron, and heavy metals can be present in high-purity phosphoric acid. The circulating load of the Copebras' phosphate rock flotation circuit was submitted to bench scale flotation tests with addition of a milling step were carried out in order to increase the recovery of P_2O_5 and the reduction of the Fe_2O_3 content to values below 5%. Samples were collected from the flow composed by the scavenger concentrate and cleaner tail, with $d_{95} < 208 \mu m$ and average content of 26.2% of P_2O_5 and 13% of Fe_2O_3 (6% hematite, 5% goethite, 1% magnetite, and 1% ilmenite) and comminuted in a pilot pant rod mill. Three samples with different d_{95} (< 150 , < 105 , and $< 74 \mu m$) were produced. Exploratory flotation tests were performed to optimize collector and depressant dosages. The best results were obtained for dosages of 500 g/t of depressant and 20 g/t of surfactant. For the collector dosage the best results were obtained for 235 g/t ($d_{95} < 150 \mu m$), 220 g/t ($d_{95} < 105 \mu m$) and 200 g/t ($d_{95} < 74 \mu m$). The results indicated that the milling of the flotation circulating load increased the mass recovery (from 27.3% to 37.5%) and metallurgical recovery (from 45.8 to 62.9%) of phosphate rock, as well as a reduction from 2.32 to 2.04% of Fe_2O_3 content (for the sample with $d_{95} < 150 \mu m$). The industrial implementation of a milling stage of the flotation circulation load and a subsequent flotation of this material has the potential to increase the overall process efficiency by approximately 5.5%, resulting in a production increase of 62,000 tons/year of phosphate rock concentrate, with higher quality than the one currently produced. The phosphate rock flotation tailings feeds a niobium processing plant, a co-product of extreme importance due to its economic value. The proposed circuit increased the niobium liberation degree and its content in the produced tailings, which will probably lead to a higher recovery of the same.

GEOSCAN-M USE AT A MIDDLE EASTERN PHOSPHATE PLANT

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Key Words: Online analysis, real-time, Geoscan, process control.

Access to real-time data is critical for process control. Scantech’s GEOSCAN-M elemental analyser has been utilized in the minerals industry for the past two decades in a range of different commodities. Already established for use in the coal and cement industries, commodities such as iron ore, copper, lead zinc, manganese, bauxite and lithium have benefited from the real-time access to elemental composition data on conveyed flows provided by the GEOSCAN. The phosphate industry has also recognized the benefits of the equipment and a number of operators and companies have installed GEOSCANs to provide real-time analysis back to the plant.

A Middle Eastern phosphate company, one of the biggest phosphate producers in the region, entered discussions with Scantech regarding the use of a GEOSCAN for analyzing phosphate rock, and underwent a process that included thorough vetting and due diligence, and included test work conducted on samples sent from site to Scantech in Australia. The GEOSCAN-M was installed and commissioned at the Middle Eastern mine in 2017. Initially, the GEOSCAN was intended to be used for grade control, and is currently being used for this purpose, as well as to control downstream processes. Analysis data for a wide range of elements is available in real time allowing for accurate and timely control of ore processing.

This paper provides an introduction to the GEOSCAN-M and how it works in the phosphate industry, while treating the installation with the Middle Eastern site as a case study. The benefits achieved by site as a result of their GEOSCAN are summarized, as well as a discussion of the GEOSCAN performance in phosphate. Recent calibration data comparing GEOSCAN results to laboratory results are shown below in figures 1 and 2 for phosphate and silica, with a summary of root mean square deviation (RMSD) performance between laboratory and GEOSCAN for these and other elements provided in table 1.

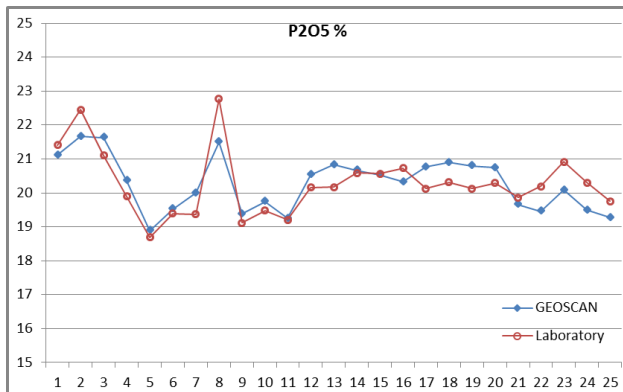


Figure 1 – Comparison of GEOSCAN and laboratory data for phosphate

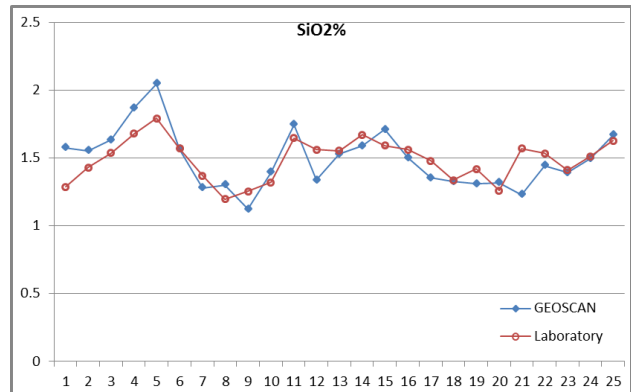


Figure 2 – Comparison of GEOSCAN and laboratory data for silica

Oxide	P ₂ O ₅ %	SiO ₂ %	CaO %	MgO %	Cl ppm
RMSD	0.55	0.14	0.51	0.15	71

Table 1 – RMSD between GEOSCAN and laboratory

As can be seen, excellent agreement between GEOSCAN and laboratory results is observed on dynamic data, allowing operators to have confidence in the data being used for control. Scantech enjoys an ongoing relationship with site in providing further utilization of GEOSCANs across their sites in phosphate.