

Production of minerals paste from phosphates sludge

Producción de pasta mineral a partir de lamas fosfáticas

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RESUMEN

El método más utilizado para la disposición de los relaves fosfáticos es vía tranque convencional de relaves. La disposición de relaves en forma de pasta mineral presenta ventajas sobre la disposición convencional. El objetivo de este trabajo fue la producción y caracterización de pastas minerales desde lamas fosfáticas utilizando un espesador en escala de laboratorio. Los principales resultados fueron: a) peso específico del material sólido: 2,61 g/cm³; b) lama 100% < 30 µm; c) el análisis químico reveló presencia principal de magnesio, calcio, fósforo y oxígeno; d) el análisis mineralógico indicó predominancia de calcita y dolomita; e) la muestra presentó elevado valor de área superficial específica de 0,976 m²/g; f) en los ensayos de espesamiento el tiempo requerido para el punto máximo de compresión sin floculante fue 7.200 s; g) los ensayos de sedimentación continua con recirculación y floculante aumentaron alrededor de 6% sólidos en la descarga; h) el perfil de concentración del sedimento exhibe una transición de pulpa de alta densidad a pasta mineral entre 55% y 58% sólidos; i) respecto a la generación de flóculos el sistema RGF presentó una descarga más densa de concentración de sólidos en todo el rango operacional; j) el sistema de rastras presentó un aumento alrededor de 7% sólidos en la descarga; k) con las mejores condiciones operacionales fueron obtenidas pastas minerales de 70% sólidos; l) el esfuerzo de cedencia tuvo valores superiores a 80 Pa desde 65% de sólidos característica de una pasta mineral.

Palabras clave: Espesamiento, pasta mineral, reología.

ABSTRACT

The most used method for the disposal of phosphates tailings is through of tailing dams conventional. The tailing disposal in the form of mineral paste has advantages compared with the disposal conventional. The objective of this work is the production and characterization of mineral pastes from phosphates sludges using a thickener in laboratory scale. The main results were: a) the specific gravity of solid material: 2.61 g/cm³; b) sludge 100% < 30 µm; c) the chemical analysis indicated the principals presence of magnesium, calcium, phosphorus and oxygen; d) the mineralogy detected the presence calcite and dolomite as main phases; e) sample showed high value of the superficial specific area through Blaine method was 0.976 m²/g; f) the thickening tests time required for maximum compression point of phosphate mud without the use of flocculant was 7,200 s; g) the tests continuous sedimentation using recirculation and the use of flocculant selected increased by approximately 6% solids in the underflow; h) a transition stage of high

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slurry density to mineral paste in the range between 55% and 58% solids was observed; i) concerning to generations of flocs, the FGR system presented a discharge more dense of solids concentration in all operational range; j) an increase of around 7% solids in the underflow was noted using rake system; k) with the best operational conditions in the equipment were obtained paste minerals of 70% solids; l) the yield stress of phosphate sludge showed values greater than 80 Pa from 65% solids characteristic of mineral paste.

Keywords: Thickening, mineral paste, rheology.

INTRODUCTION

The beneficiation of phosphate ores generates large quantities of tailing and by-products. For example in Florida the phosphate mining and beneficiation produces about 100,000 tons of waste clays are currently produced each day. There are more than 85,000 acres of phosphatic clay ponds and clay-filled mine cuts, with approximately 5,000 acres of additional ponds created each year by ongoing phosphate mining and beneficiation. Waste clay disposal represents one of the most challenging problems for the phosphate industry. Because of their colloidal nature and ultrafine size (91.5% by weight -325 mesh or 44 μm), the phosphatic clays are very stable in water suspensions and settle extremely slowly [1].

Large areas for disposal in conventional dam system are required. The disposal of mineral tailings in the form of mineral paste is an alternative technology that may allow better utilization of the disposal areas, provision of greater stability of disposal, greater reuse of process water, operational optimization and other factors [2].

The consolidation of phosphatic clays to a state suitable for reclamation and reuse of the land may take a decade or more, and suitability for support of constructed facilities may not occur at all. Efforts to solve the problems associated with phosphatic clay disposal and reclamation have included testing of mechanical, electrical, biological, and chemical processes. These efforts have included the addition of coagulants and flocculants, mechanical dewatering, the use of enzymes and bacteria and various other methods of thickening and stabilization [3].

The mineral paste can be defined as a system of fine particles which is presented as a homogenous fluid, in which no occurs particle size segregation

of particles, and which if disposed smoothly on a stable surface, no present a significant water drainage [4-5]. However, can be defined as a non-settling, non-segregation, homogeneous suspension with minimal water release [3].

The characteristics of the behavior of the mineral paste are due to slurry densification in solids from a certain percentage of solids by weight. As a result, higher angles of repose can be achieved for example depend the concentration of the sludge in laboratory scale was obtained values between 4 and 16 degrees [2]. This represents a significant gain in terms of volumes to be arranged in the same area. Among the main properties of a mineral paste include: viscosity, yield stress, consistency and the angle of repose [6-8].

A paste does not obey the rules of flow for a Newtonian fluid. This non-Newtonian character results in a rheology that changes with exposure to different shearing rates and times during thickening, pumping, flowing in a pipeline, and deposition. For many mineral beneficiation tailings pastes, viscosity changes with the shearing rate [3].

The use of thickeners to produce mineral paste is already practiced in several countries such as Australia for disposal of "red mud" [9-10], Canada [11] and the United States for disposal of phosphatic clays [1-3]. In Chile and Peru is an important issue mainly due to lower availability of water in arid regions. Mining of Brazil has also a strong focus because of the need for the application of new technologies that allow for greater reuse of water used in the process and tailings disposal with higher concentrations of solids.

HiDensity and DeepBed paste thickeners were developed to produce higher yield stress underflow solids. The higher the underflow solids, the higher the yield stress produced. Higher density underflow

solids reduce water losses or water usage in many instances. This must be tempered with consideration to the underflow rheology [12].

Yield stress, which is defined as the minimum stress that must be applied in order to induce flow, is a key design parameter in the industrial application of surface disposal of thickened tailings (sometimes termed paste) [13]. Several studies relate to the yield stress with a colloidal stability and surface characteristics of the particles [14-16].

Floc strength is a particularly important operational parameter in solid/liquid separation techniques for the efficient removal of aggregated particles. Smaller particles will generally settle more slowly than larger particles of similar density [17].

The settling rate measurements indicate an inverse relations between floc size and density. The reduced floc density with increasing size is usually attributed to the incorporation of fluid into the floc structure [18-19]. Floc strength is directly related to floc structure and is, therefore, highly dependent upon the floc formation process [17].

The measured apparent yield stress is dependent on the operation procedure and test apparatus due to the materials complex continuous three-dimensional network microstructure. The problems associated with measuring the rheology of structured fluids can be overcome by using a vane.

The vane consists of four or more blades that form a cross shape at the end of a central shaft and can be inserted into the sample with minimal disturbance of the microstructure [20]. The vane is primarily used to determine the yield stress of structured systems and detailed reviews on this and other methods to characterise the yield stress can be found in several articles [7-21-22].

Unlike a slurry, which is basically a liquid, a paste will hold its shape to a degree determined primarily by the solids concentration. This property is a defining characteristic of a paste that promotes increasingly solid-like behavior as the material becomes more concentrated.

Yield stress, which is the pressure required to make the paste flow, arises from a unique property of

non-newtonian fluids. The bond formation between fine particles through electrostatic and structural interactions produces a continuous network that breaks only when a certain pressure is applied, which is the yield stress. The coarser particles are inhibited from settling by the fine particle network acting like a net to hold solids in suspension. This phenomenon is responsible for the nonsettling and non-segregation behavior of pastes. Shearing introduces energy that disrupts the bonding between fine particles, causing a change in viscosity. Yield stress is dependent on many parameters such as particle size, temperature, mineralogy, pH, and solids concentration [3].

The yield stress of tailings increases exponentially with increasing solids concentration so a small change in concentration can result in a large change in the yield stress [13], consequently, control of material consistency in waste disposal operations is critical [23-24]. Various studies make a relationship of yield stress with the solids concentration of tailings [25-29].

The yield stress is characterized principally for: i) the shear stress required to initiate significant flow, ii) below its yield stress a material will behave elastically, iii) crucially important in preparation, transport and deposition of thickened tailings [30]. Figure 1 shows, in qualitative character, the yield stress as a function of percent of solids.

The objective of this work is show other alternative of tailings disposal through the production, characterization and analyses rheological of mineral pastes from phosphates sludges using a thickener in laboratory scale considering the possible disposal of these materials because that this technique is few used actually in Latin American mining.

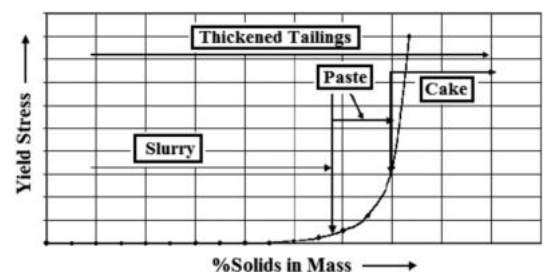


Figure 1. Yield stress as a function of the percentage of solids [30].

METHODOLOGY

Sample Mineral

The sample mineral used represent sludge obtained from the beneficiation plant of phosphate ore of Brazil. The Table 1 presents the different techniques and equipments used to determine the physical and chemical characterization of the mineral sample.

Settling Tests

The settling tests were carried out using graduated cylinders (2 L) at 10% solids in mass for preliminary determination of the minimum residence time necessary to obtain slurry with higher density.

The selection of the best performing flocculants was effected in graduated cylinders (0.5 L) at 10% solids in mass, value corresponding to the industrial plant of beneficiation.

The flocculants evaluated are the types: cationic, anionic and non-ionic prepared at concentrations of 0.1% W/V and diluted to 10% V/V. The flocculation was carried out in a graduated cylinder of 2 L using the manual agitator, in upward and downward movements for mixing the slurry with the polymer for 5 s based in previous works [8-31].

The flocculant which presented the best performance was selected for thickening tests in laboratory scale.

Laboratory Mineral Paste Thickener

This equipment is made of acrylic with three cylindrical modules having total height of 1.8 m with 0.1 m of diameter and maximum slurry volume of 10 L. The rake system, with low speed rotation, can be used. Sampling and recirculation of slurry can be performed by means of a series of lateral nozzles. The underflow discharge of the mineral paste is carried out on the bottom of the equipment

and the overflow exits from the top. The continuous feed operation was performed by using peristaltic pumps. The Figure 2 shows the general view of the assembly of the equipment.

Static Mixer and Floc Generator Reactor

The system of static mixer consists of a tube of carbon steel with 0.0508 m internal diameter and 0.30 m length with a series of baffles positioned to stimulate increased contact between slurry and polymer. Figure 3 shows the system used with the static mixer.

The Figure 4 shows the floc generator reactor system (FGR) used in the mineral paste thickener.

The floc generator reactor consists in a transparent polyurethane tube with an internal diameter of 0.0127 m wrapped on the outside of a fixed column of polyvinyl chloride with an internal radius of 0.1 m and length of 1.2 m.

The purpose of the two devices is to operate in order to improve the interaction polymer/mineral particle allowing the most appropriate flocculation.

Mineral Paste Thickener Tests

All the tests in the mineral paste thickener aimed to obtain the maximum percentage of solids possible in the underflow. The best operational conditions found were the followings: total time in continuous operating of 180 min, slurry feed at 10% solids in mass, dosage of the selected flocculant 80 g/t, total volume of operation 10 L, use of the two types of mixers slurry/flocculant (SM and FGR), recirculation of the slurry at different points of the compaction zone, rake rotation 5 rpm (when used).

The solid concentration profile along the settled layer was determined by use of lateral nozzles.

Table 1. Techniques and equipments used to characterize the mineral sample.

Property	Technique Used	Required Equipment
Specific gravity	Simple pycnometry and gas	Pycnometer and gas
Size distribution	Centrifugal classification and Laser diffraction	Cyclosizer and Sympatec
Specific area	Adsorption of nitrogen and air of permeability	BET adsorption system and Blaine apparatus
Yield stress	Rheometry	Haake rheometer
Chemical composition	X-ray fluorescence	Dispersive X-ray spectrometer
Mineral composition	X-ray diffraction	X-ray spectrometer

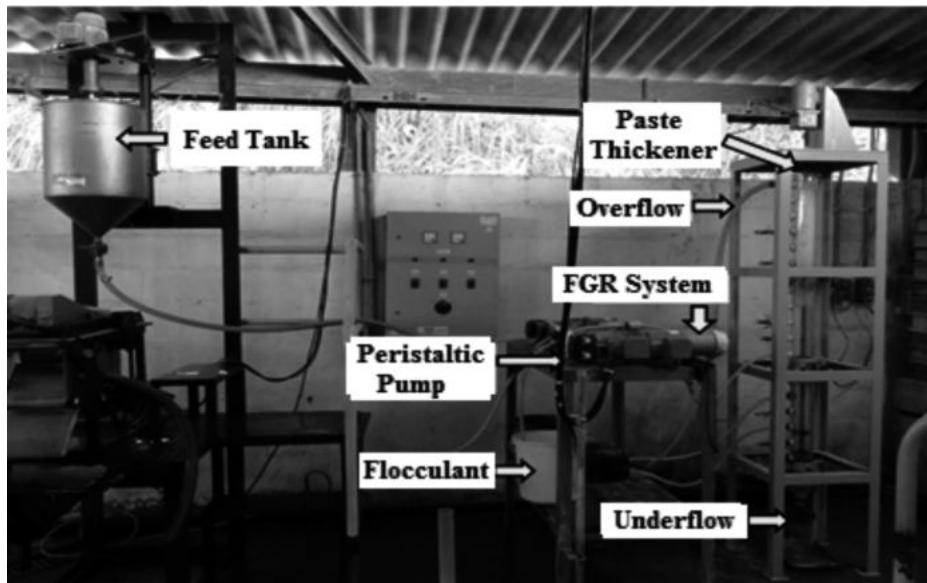


Figure 2. General view of operational assembly of the mineral paste thickener [2].

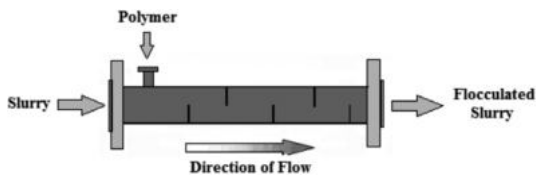


Figure 3. Static Mixer (SM) for feeding slurry with polymer [2].

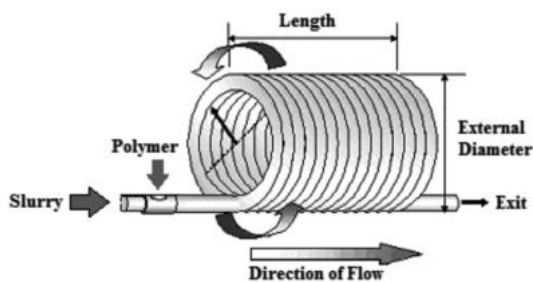


Figure 4. Floc Generator Reactor (FGR) for feeding slurry with polymer [32].

Yield Stress Measurements

Measurements of yield stress were performed on slurries and minerals pastes using a Haake digital rheometer with interface for constant sending data to the computer. Figure 5 shows the rheometer utilized.



Figure 5. Haake rheometer, model VT-550 with interface to the computer.

Two models were used to determine the yield stress of the slurries and minerals pastes: The model of Break-Through and Bingham with a duration of approximately 300 seconds for each solids concentration. In the first model, the sensor helical of the viscometer turned slowly measuring the resulting drag force. The turning force increased until reaching a point maximum decreasing gradually until stop reading.

The point maximum was used as a measure of the yield stress. The second model consists in generating a flow curve by placing the samples in a series of shear stress, which are obtained by applying the slurries and minerals pastes in different speeds of rotation through the helical sensor.

The data generated by the flow curve were interpreted using the Bingham model, which extrapolates the data back to zero shear.

RESULTS

Mineral Sample Characteristics

The specific weight values obtained by simple pycnometry (SP) and gas (GP) are presented in the Table 2. Based in the data historical of the industrial plant for comparative effect the specific gravity obtained by simple pycnometry was considered for calculation as guidelines of the tests [2]. The standard desviation for measurement of the simple pycnometry was 1.23%.

Table 2. Specific weight values obtained by simple pycnometry and gas.

Technique Used	Specific Weight (g/cm ³)
Simple pycnometry	2.61
Gas pycnometry	3.06

The particle size distribution of the mineral sample is shown in Figure 6. The material is very fine: 100% less than 30 μm. The value of the median size (d₅₀) is about 4.76 μm. The difference between the curves reflects the measurement parameters of each equipment: the size of each particle determined by the laser diffractometer is the arithmetic average of the three major dimensions of this particle, and the size measured by the cyclosizer is the hydraulic diameter of the particle.

The chemical composition of the mineral samples were obtained by X-ray fluorescence. The results

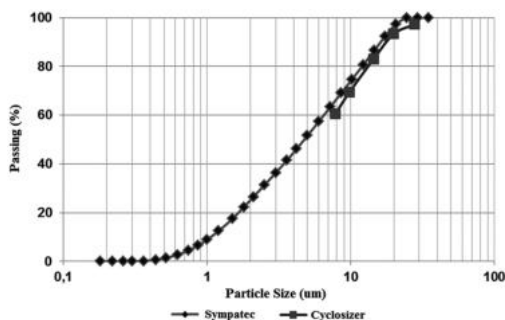


Figure 6. Size distribution of the mineral sample.

are shown in Table 3, in which indicating the predominance of CaO, MgO, SiO₂, P₂O₅ and Fe₂O₃ with the presence of small quantities of Al₂O₃, K₂O, SO₃ and Ti₂O₃. The loss on ignition (LOI) was of 37.5% principally due to the presence of carbonates.

Table 3. Chemical composition of the mineral sample obtained by X-ray fluorescence.

Compound	(%)	Compound	(%)
CaO	48.22	Al ₂ O ₃	0.31
MgO	4.60	K ₂ O	0.11
SiO ₂	3.03	SO ₃	0.87
P ₂ O ₅	3.34	Ti ₂ O ₃	0.11
Fe ₂ O ₃	1.54	LOI	37.5

The analysis X-ray diffraction was by the method of Rietveld that indicated as the main mineralogical phases: CaCO₃ (calcite) and CaMg(CO₃)₂ (dolomite). It was also detected the presence of K₂O·6MgO·Al₂O₃·6SiO₂·2H₂O (phlogopite) and Fe₃O₄ (magnetite). The Table 4 shows the specific superficial area determined by BET and Blaine techniques. It was observed the significant difference between the values, as expected. However, the adsorption isotherm obtained by the BET method showed how type III, does not represent the monolayer formation necessary for adequate evaluation.

Table 4. Specific Superficial Area.

Technique Used	Specific Superficial Area (m ² /g)
BET	1.972
Blaine	0.976

Settling Tests

The Figure 7 shows the settling curve obtained by using graduated cylinder of 2 L for slurry at 10% solids without use of flocculant. The maximum compression point has been reached to values greater than 7,200 s with a settling rate of 0.0119 cm/s, which can be seen as slow when compared metallic ore tailings.

Selection and Dosage of Flocculant Tests

The settling rate of de slurry with different dosages of flocculant was determined in the initial phase of the zone free settling through relationship the

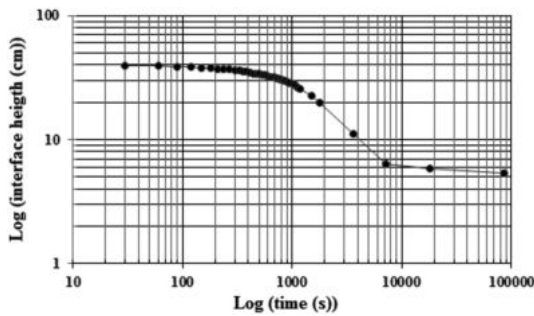


Figure 7. The height of the interface versus time both in logarithmic scale.

height of the interface (cm) and the time (s) of the experimental data from the setting curve. The Table 5 presents the results of the settling rate tests carried out in the presence of cationic, anionic and non-ionic polymers that for comparatives purpose was selected a dosage of 50 g/t.

The flocculant selected was the CF3 for subsequent tests. The settling rate reached was 0.831 cm/s at the dosage of 80 g/t. It represents a significant increase (~70 times) of the settling rate when compared with the test without addition of flocculant. The Figure 8 indicates the settling rates obtained at various dosages of this flocculant.

Thickening with Recirculation of Slurry Tests

The tests with recycle of slurry (10% solids in mass) were performed between zones sedimentation denominated as transition and compression with the addition of 80 g/t of cationic flocculant (CF3) reaching a total volume of 10 L.

The time residence of the samples was of 15 min (900 s) approximately. The combinations of recirculation and thickening obtained are shows in the Figures 9 and 10.

Table 5. Settling rate tests for cationic (CF), anionic (AF), and non-ionic flocculants (NIF).

Settling Rate CF (cm/s)	Settling Rate AF (cm/s)	Settling Rate NIF (cm/s)
CF1/0.187	AF1/0.241	NIF1/0.153
CF2/0.263	AF2/0.185	NIF2/0.126
CF3/0.480	AF3/0.125	NIF3/0.087
CF4/0.354	AF4/0.095	NIF4/0.195
CF5/0.194	AF5/0.106	NIF5/0.163

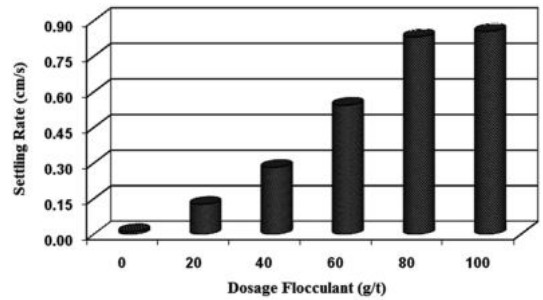


Figure 8. Settling rate of the slurry as a function of dosage of cationic flocculant (CF3).

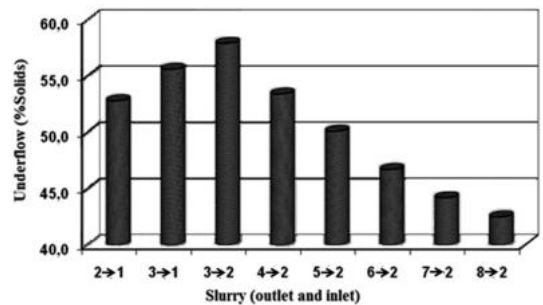


Figure 9. Percentage of solids in weight of the underflow of thickener as a function of the combinations of nozzles (exit and entry).

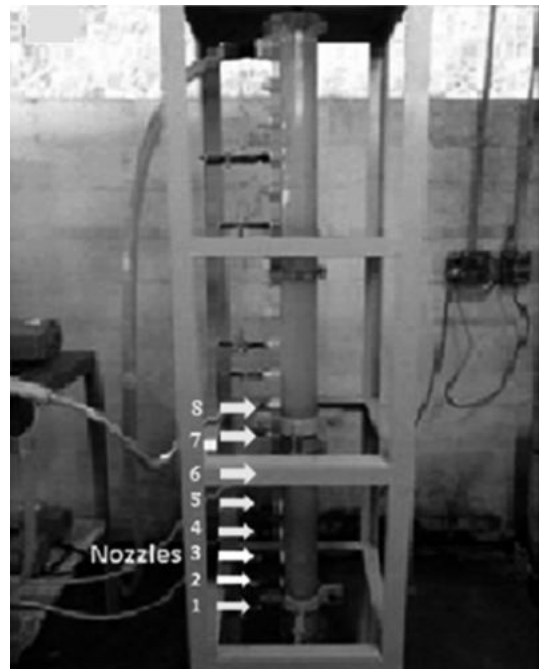


Figure 10. View of the positions of the nozzles in the thickener.

It was observed that the best conditions of operation with continuous recirculation of the slurry was from the nozzle 3 to the nozzle 2 reaching a value of 57.9% of solids in mass in the underflow. It was evidenced that the recirculation of the slurry affects the concentration of solids in the discharge of the thickener. In the compression zone from the diluted region to concentrated region improve the concentration of solids in the discharge.

Static Mixer and the Flocs Generator Reactor System Tests

The Figure 11 shows the percentage of solids in mass obtained in the underflow as a function of operating time for static mixer (SM) and floc generator reactor system (FGR) in presence of 80g/t of flocculant.

The best condition was obtained using FGR system for 60 minutes of continuous operation. It was reached 64.2% of solids in mass that can be considered attractive for industrial use.

Solid Concentration Profile

The Figure 12 shows the solids concentration profile considering the use of static mixer and floc generator reactor system, with addition of flocculant CF3 (80 g/t).

The best result was 65.4% solids in mass by using the FGR system, probably because the formation of structure flocs of this system is less aggressive than the static mixer so it generates a more compact floc with a less porous network favorable to the higher concentration of solids in the discharge due to the lower water retention. For the value of 65% solids

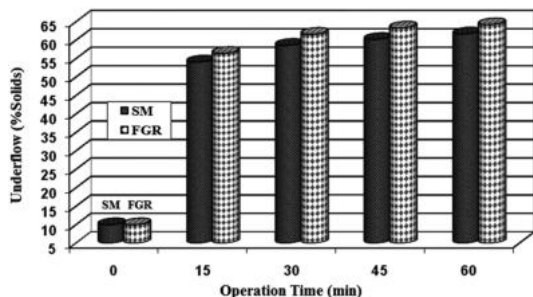


Figure 11. Percentage of solids in mass obtained in the underflow as a function of operating time for SM and FGR with 80g/t of flocculant.

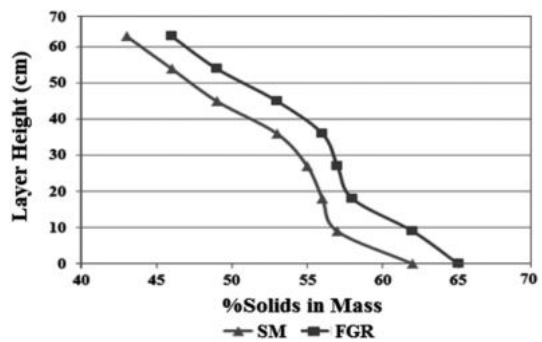


Figure 12. Profile of concentration on solids in mass, considering the use of SM and FGR.

in mass it is appreciated paste formation because the higher consistency of the mixture solid/liquid. It was observed a transition region in the range comprised between 55% and 58% solids in mass, where the solid concentration increasing takes place more slow because, probably, to formation of the flocs network more homogenous, stable and dense. It can be also noted that for percentages larger than 55% solids in mass, approximately, occurs the formation of high density slurry for subsequent production of mineral paste.

Rake Tests

The main operational parameters for the rake tests are indicated in the Table 6.

The percentages of solids in mass obtained in the underflow in function operational time for the best conditions found in the production mineral paste with and not use of the rakes system are shows in the Figure 13.

According to the Figure 13 the use of rake system influenced positively in the increasing of solids concentration in the underflow. This fact is probably due to decrease of empty spaces and or filled by the water that are present in the structure. Values

Table 6. Main parameters in the rake tests.

%Solids in feed	Dosage CF3 (g/t)	Recirculation slurry
10	80	3 to 2
Type mixer	Operation time (min)	RPM rake
FGR	180	5

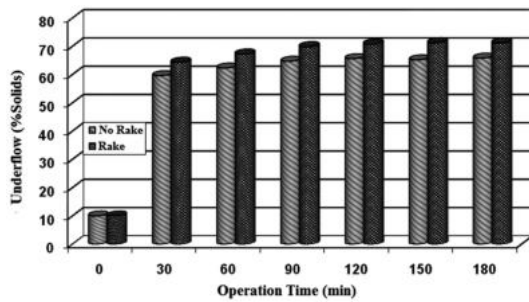


Figure 13. Percentage of solids in mass of the underflow in function of operation time with and not use the of rakes system.

achieved in the best operation condition were around 70% solids in mass after 120 min of continuous operation.

Yield Stress of the Slurry and Mineral Pastes Produced by Thickener

For the meditions was used a rotational rheometer with a sensor of immersion trough vane method. Figure 14 shows the values of the yield stress obtained by the models of Break-Through and Bingham from different slurries and minerals pastes generated through the thickener developed.

It was observed that the material becomes as mineral paste from 65% of solids in mass with initial values of the yield stress around 80 Pa, this values confirmed the presence of paste mentioned in the Figure 12. Figure 15 presents a preliminary hypothetical comparison for the use of conventional dam and disposal in the form of mineral paste considering a flow of about 30 t/h dry basis for a period of one month.

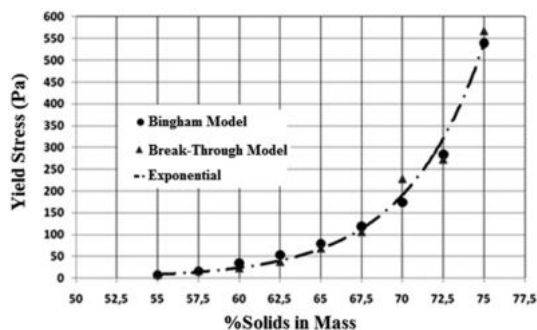


Figure 14. Yield stress as a function of the thickening of phosphate sludge.

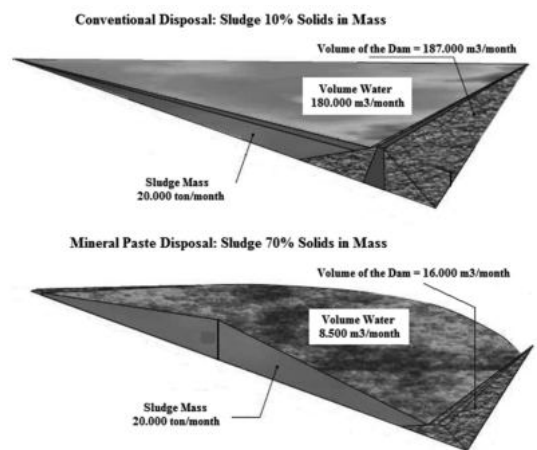


Figure 15. Comparison of areas for conventional disposal and mineral paste of phosphate sludge [Adaptation, 33].

Considering as base to the determination of disposal volume it was the density and mass of the phosphate sludge in state of slurry and paste respectively. It can be seen from the Figure 15 that conventional dam would require a volume of approximately 187,000 m³/month for disposal of phosphate sludge “untreated”. In the case of disposal of the phosphate sludge in form of mineral paste are required around 16,000 m³/month of volume of the dam, in other words, is required a total volume for dam of approximately 12 times smaller, decreasing thereby significantly the area required for the disposal, thus increasing the life’s dam around one year. The use of mineral paste enables the reuse of approximately 170,000 m³/month of water coming directly from the overflow of the equipment producer of mineral paste. On the other hand, the conventional disposal pumping systems are usually required to improve water recovery and recirculation.

CONCLUSIONS

The phosphate slurry had particle size quite thin with an average size of 4.76 μm and high specific superficial area 0.976 m²/g. The predominant mineralogical species in the sample are calcite and dolomite.

The minimum time required to reach the point of maximum compression of the slurry was greater than 7,200 s. The flocculant selected is of cationic type in dosage fixed of 80 g/t. The best operating

condition in the tests of recirculation of the slurry in the compression zone of the sludge was from the nozzle 3 to the nozzle 2 obtaining high density values around 58% solids in mass.

The use of floc generator reactor system in the feed system of slurry with flocculant presented a better efficiency than the static mixer probably because that static mixer presents higher energy of dissipation what generate flocs networks more porous and unstable that entails yet higher entrapment of water in the network. It was observed in the concentration profile that there is a transition region in the range between 55% and 58% solids in mass, where the increase of the percentage of solid in mass is increased more slowly.

The use of rake system influenced favorably the concentration of solids in the underflow maybe for the liberation of trapped water in the flocs network. In the best operational conditions values of 70% solids in mass were obtained for 180 min of continuous operation.

The phosphate sludge appears as mineral paste from of 65% solids in mass, approximately, with initial values of yield stress about 80 Pa, which can be considered acceptable for the transport by pumping of these tailings.

The disposal of phosphate sludge in the form of mineral paste would require a volume of dam approximately of 12 times lower when compared with the conventional dam disposal, thus increasing the life operational of the dam. The use of the equipment producer of mineral paste increases operational flexibility to reuse water of process directly since the unit mineral processing, with a capacity of approximately 170,000 m³/ month.

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